IUPAC–NIST Solubility Data Series #.
Transition and 12 to 14 Main Group Metals, Lanthanide, Actinide and Ammonium Halates

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Abstract

This paper is the fourth and final volume in the halate solubility series. The solubility data for halates of transition metals, lanthanides, actinides, ammonium and metallic elements of the main groups 12 to 14 are reviewed. Where appropriate binary-, ternary- and multi-component systems are critically evaluated. Most of the solubility results were obtained in water or aqueous solutions of electrolytes. The solubilities in organic solvents and aqueous–organic solvent mixtures are also collected in this volume. All these data were critically examined for their reliability. The best values were selected on the basis of critical evaluations and presented in tabular form. Fitting equations and graphical plots are also provided. When numerical data were not reported in an original publication, they were read out from figures and digitized by the compilers. The quantities, units and symbols used in this

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volume are in accord with the IUPAC recommendations. We always reported the original data and, if necessary, transferred them into the IUPAC recommended units and symbols. The literature on the solubility data was researched through 2002. The compilations and critical evaluations have been presented in IUPAC–NIST Solubility Data Series format. The halates of the mentioned metals play a role in industrial processes. For example, some halates are essential as catalysts, heat-stabilizers, and blanching reagents for manufacturing polymer products such as textiles and resins. Some iodates are used in pyrotechnic compounds for weather modification and colored smoke generation. The nonlinear halate crystals are important in construction of optical devices.

Key words: transition metals; lanthanides; actinides; elements of main groups 12–14; aqueous solutions; solubility; chlorates; bromates; iodates.

Contents

List of Figures

List of Tables

TABLES

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FIGURES

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I. PREFACE

The present volume is the final publication in a series of four volumes on inorganic metal halates: that is, chlorates, bromates and iodates. The first volume on *Alkaline Earth Metal Halates* was published in 1983 (1), the second volume on *Alkali Metal Halates, Ammonium Iodate and Iodic Acid* was published in 1987 (2), and the third volume on *Copper and Silver Halates* was published in 1990 (3).

This volume includes compilations and critical evaluations for the solubility of transition metal, lanthanide and actinide halates. The solubility data for the halates of metal element in the groups 12 to 14 are also included. The solubility of ammonium iodate was compiled previously in (2). Ammonium chlorate and bromate could not be compiled together in (2) because the respective solubility studies had not yet been reported. For completeness, the data of ammonium chlorate are therefore included to this volume. Compilations for the solubilities of the title halates in water and organic solvents (such as ethanol and acetone), and those in aqueous–organic solvent mixtures, aqueous electrolyte and buffer solutions are included in this volume and presented in the IUPAC–NIST Solubility Data Series format.

The critical evaluations and compilations of the halate solubilities deal only with the simple salts of a type of MX_m (M = metal; X = halate) and do not treat complex compounds such as hexaamminecobalt(III) halates; $[Co(NH_3)_6]X_3$. Many solubility data concerning the complex compounds of cobalt and other transition metal elements were reported in a book edited by Linke (4).

The notation of the word "transition elements" was described in Nomenclature of Inorganic Chemistry, IUPAC Recommendations 1990 (The Red Book) edited by Leigh (5). According to The Red Book, the transition elements may be defined strictly as elements that have partly filled d or f shells only. Cotton and Wilkinson (6) stated in their famous textbook that we shall adopt, however, a slightly broader definition and also include elements that have partly filled d or f shells in any of their commonly occurring oxidation states. With our broad definition in mind, we find that there are now some 56 transition elements, counting the heaviest elements through atomic number 104. This large number of transition elements is subdivided into three main groups: (a) the main transition elements or d-block elements, (b) the lanthanide elements and (c) the actinide elements. These authors adopted the broad definition in order to classify the transition elements. This volume contains the evaluation and compilation on the solubility data for the halates of seven elements (Ti, Mn, Fe, Co, Ni, Zr and Hf) in the first, second and third transition series in the first two chapters. Information concerning the halates of the other main transition elements was not found during our literature survey. In the two succeeding chapters, the solubilities of bromates and iodates of lanthanide with those of scandium and yttrium were compiled and critically evaluated, but those of chlorates are missing because of the absence of chlorate solubility information which could not be found in the Chemical Abstracts. Because of the similarity of the chemical properties between lanthanide, scandium and yttrium compounds, the solubility studies of scandium and yttrium halates were treated with those of lanthanides. The element yttrium, which belongs to group 3, has a similar $+3$ ion with noble gas core, and has an ionic radius close to that of terbium($+3$) or dysprosium($+3$). Scandium also belongs to group 3. The radius of scandium($+3$) is appreciably smaller than the radii of the rare earth elements and its chemical behavior is intermediate between that of aluminum and the lanthanides. Therefore, scandium($+3$) is not classified a rare earth element as discussed in (6). The studies of the two actinide elements, thorium and uranium (as uranyl ions) are described in a separate chapter. Papers on the solubility of chlorates and bromates of the actinide elements could not be found in Chemical Abstracts. The solubility data for the halates of some elements (Al, Zn, Ga, Cd, In, Hg, Tl, and Pb) in the main groups 12 to 14 are discussed separately. The solubility of ammonium chlorate was also described therein.

All data were critically examined for their reliability and best value estimates were selected on the basis of critical evaluation. Reported data and best selected data are presented in the form of tables, fitting equations and graphical illustrations. When numerical data were not given in a publication, the data have been often read out from figures and digitized by the compilers. The quantities, units and symbols used throughout this volume are those recommended by IUPAC. We always report data in units originally used by the authors. If IUPAC recommended units and symbols, that are SI units, were not used in the original paper, the reported solubility values were converted to IUPAC recommended symbols and SI units by the compilers. The authors' units were frequently expressed in terms such as mol dm^{-3} instead of mol l^{-1} or mol L^{-1}. If the original data are given in mass percent or mass fraction, the compilers converted the solubility values to mol percent units in concentrated solutions or to molality units in diluted solutions.

The halates play a role in many industrial processes. In order to learn details about applications of transition metal and lanthanide halates, we surveyed the patent documents described in Chemical Abstracts published in the last 30 years. Some halates are used as catalysts, heat-stabilizers, and blanching reagents for manufacturing polymer products such as textiles and resins. Systems containing some chlorates are ideal for low-temperature refrigeration and energy storage. Some iodates are used as pyrotechnic compounds for weather modification and colored smoke generation. The nonlinear halate crystals are important optical materials. Some secondary nonaqueous electrolyte batteries have lithium intercalating anodes and metal iodate cathodes.

The literature survey of all halates was covered through 1897 to 2002, and we believe that the survey is complete. Chemical Abstracts were mainly used as the reference source for solubility data. However, the keyword "solubility" under a compound name was not sufficient to find complete solubility information from corresponding literature. Particularly, important information on mutual solubility data was obtained from the keyword "system" under a compound name. The survey of the corresponding literature published before 1907 was based on the solubility data books edited by Linke (4) and Stephen and Stephen (8). In a few instances, relevant papers were not available for the compilers. As some published solubility data of the halates may have been overlooked, the editors would appreciate being informed about any omitted sources of solubility data on the title compounds.

On the fitting equation

Mole fraction, molality (mol kg^{-1}), molarity (mol dm^{-3}) and mass % units were usually used to report the solubility data. Use of three types of fitting equations for the former three kinds of units are found in IUPAC Solubility Data Series (SDS) Vol. 30 edited by Miyamoto and Salomon (2). In the present volume, the mole fraction units were mainly used according to advices of Cohen-Adad (9). Solubility values reported in other units were transfered to mole fraction units to formulate a smoothing equation. The following forms of equations were used (10–12).

$$Y_x = \frac{A}{(T/\mathrm{K})} + B\,\ln(T/\mathrm{K}) + C + D\,(T/\mathrm{K}), \qquad (1)$$

where

$$Y_x = \ln\left(\frac{x^v(1-x)^r(v+r)^{v+r}}{r^r(1+x)^{v+r}}\right). \tag{2}$$

In eq. (1), A, B, C and D are adjustable parameters which are determined by the regression analysis described below. In eq. (2), x is the mole fraction solubility, r is the solvation number in the equilibrium solid phase, and v is the number of ions produced upon dissolution. T is the temperature in the Kelvin scale. For example, for $\mathrm{Co(ClO_3)_2 \cdot 6H_2O}$, $r = 6$, $v = 3$. For the ice polytherm, $v = 0$ and $r = 1$. When the number of the observed data is larger than four, the least-square fitting of Y_x versus T can be performed to estimate four adjustable parameters in eq. (1). In general, when applying the observed data to the above equations, some of the data were rejected when the difference in calculated and observed solubilities ($|x_{\mathrm{obsd}} - x_{\mathrm{calcd}}|$) exceeded $2\sigma_x$. σ_x is the standard error, and two kinds of standard deviations shown below were calculated in the usual manner for estimating fitting quality:

$$\sigma_x = \sqrt{\sum \frac{(x_{\mathrm{obsd}} - x_{\mathrm{calcd}})^2}{N-4}}, \tag{3}$$

$$\sigma_Y = \sqrt{\sum \frac{(Y_{\mathrm{obsd}} - Y_{\mathrm{calcd}})^2}{N-4}}, \tag{4}$$

where N is the number of data points, and note that there are four parameters in eq. (1). Further information concerning the fitting equation can be found in the INTRODUCTION TO THE SOLUBILITY OF SOLIDS IN LIQUIDS in SDS Vol. 30 (2).

There are some cases that we cannot use the solubility in mole fraction units to fit the smoothing equations, eqs. (1, 2). When the original solubility values were only given in molarity unit, the data could not be converted to mole fraction units. In this case, the following equation was used:

$$\ln\left(\frac{c_i}{\mathrm{mol\,dm^{-3}}}\right) = \frac{A}{(T/\mathrm{K})} + B\,\ln(T/\mathrm{K}) + C + D\,(T/\mathrm{K}), \tag{5}$$

where c_i is the concentration of a species i based on the molarity unit. Furthermore, the Y_x values given by eq. (2) are undefined, when (i) the equilibrium solid phase is an anhydrous salt, where $r = 0$, and (ii) the data of the freezing point of the pure water is included, where $x = 0$ at $T = 273.15$ K. In these cases, the following equation was used:

$$x = \frac{A}{(T/\mathrm{K})} + B\,\ln(T/\mathrm{K}) + C + D\,(T/\mathrm{K}). \tag{6}$$

The least-square fitting to eqs. (1, 2) was performed by the original software, which was made by one of the editors (RM); the procedure done by the program named “soly_calc_rmiya” is as follows. First, experimental data of temperature (T), solubility (mole fraction, molality, mass %, or molarity), and weight of this data point (or accuracy) were read, and then the units of solubility are converted to mole fraction. Using these data with eq. (2), Y_{obs} values were obtaind to construct the Jacobian matrix. Then the least-square fitting was performed by DGELS routine in LAPACK 3.0 (13). After obtaining the coefficients A, B, C and D, calculated solubilities were estimated from eq. (2) with Newton-Raphson method. Furthermore, some statistics properties such as the standard errors (eqs. (3, 4)) were estimated. When $|x_{\mathrm{obs}} - x_{\mathrm{calc}}| > 2\sigma_x$, such data are candidate of rejection. Finally, the recommended value of solubility at selected temperature is calculated. If the standard error is not sufficiently small or there are some rejection-candidate data, the accuracy of the experimental data can be reduced to zero and the smoothing equation can be re-calculated.

When the units of the originally reported solubility data were mass fraction (w) (or mass % ($100w$)), molality (m), and other units which can be converted to w or m, the w or m values were used as the input data for fitting procedure. These given w or m values were converted to mole fraction (x) in our fitting program; then x values were fitted to eqs. (1, 2). The recommended solubilities were calculated in the mole fraction units from the obtained smoothing equation and tabulated with the values in molality, for convenience of the reader, in Evaluation Sheet of each salts in this volume. The phase diagrams or the temperature dependent solubilties of the salts were drawn in the mole fraction unit. If a reader wants the solubility values in units other than the mole fraction units, the conversion can be proceeded by usual way.

Use of the three types of fitting equations are presented in SDS Vol. 30 (2). The programs "CALC_X" and "CALC_M" were prepared by Salomon (2), in which either recommended or tentative solubility values can be obtained as shown in PREFACE of (2).

Advantages of our new program "soly_calc_rmiya" are as follows; (i) all fitting calculations are performed in the mole fraction unit, if possible. This avoids an ambiguities in the units used, when original paper gave solubilities in more than two kinds of units; the calculated solubilities are different upon the used fitting equation. (ii) A fitting to the smoothing eq. (1) is a simple task of the linear least-square problem. However, the condition of the Jacobian must be critical, because the values in the first column ($1/T$) is *ca.* 10^5 times smaller than the value in the fourth column (T). Then the Jacobian has to be treated carefully, and the variables in calculating program must be of double precision. In the view point of robustness and reliability, we used the DGELS routine in the LAPACK version 3.0 library (13). (iii) Table of recommended values of solubilities is automatically obtained.

The source list of "soly_calc_rmiya" is available on Web site (14).

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II. INTRODUCTION TO THE SOLUBILITY OF SOLIDS IN LIQUIDS: SOLUBILITY OF SOLIDS IN LIQUIDS

A. The Nature of the Project

The Solubility Data project (SDP) has as its aim a comprehensive review of published data for solubilities of gases, liquids and solids in liquids or solids. Data of suitable precision are compiled for each publication on data sheets in a uniform format. The data for each system are evaluated and, where data from independent sources agree sufficiently, recommended values are proposed. The evaluation sheets, recommended values, and compiled data sheets are published on consecutive pages.

B. Compilations and Evaluations

The formats for the compilations and critical evaluations have been standardized for all volumes. A description of these formats follows.

Compilations

The format used for the compilations is, for the most part, self-explanatory. Normally, a compilation sheet is divided into boxes, with detailed contents described below.

Components: Each component is listed according to IUPAC name, formula, and Chemical Abstracts (CA) Registry Number. The Chemical Abstracts name is also included if this differs from the IUPAC name, as are trivial names if appropriate. IUPAC and common names are cross-referenced to Chemical Abstracts names in the System Index.

The formula is given either in terms of the IUPAC or Hill (1) system and the choice of formula is governed by what is usual for most current users: *i.e.*, IUPAC for inorganic compounds, and Hill system for organic compounds. Components are ordered on a given compilation sheet according to:

(a) saturating components:
(b) non-saturating components;
(c) solvents.

In each of (a), (b) or (c), the components are arranged in order according to the IUPAC 18-column periodic table with two additional rows:

Columns 1 and 2:	H, alkali elements, ammonium, alkaline earth elements
Columns 3 to 12:	transition elements
Columns 13 to 17:	boron, carbon, nitrogen groups; chalcogenides, halogens
Column 18:	noble gases
Row 1:	Ce to Lu
Row 2:	Th to the end of the known elements, in order of atomic number.

The same order is followed in arranging the compilation sheets within a given volume.

Original Measurements: References are abbreviated in the forms given by Chemical Abstracts Service Source Index (CASSI). Names originally in other than Roman alphabets are given as transliterated by Chemical Abstracts. In the case of multiple entries (for example, translations) an asterisk indicates the publication used for compilation of the data.

Variables: Ranges of temperature, pressure, etc. are indicated here.

Prepared by: The names of all compilers are given here.

Experimental Data: Components are described as (1), (2), etc., as defined in the "Components" box. Data are reported in the units used in the original publication, with the exception that modern names for units and quantities are used; *e.g.*, mass per cent for weight per cent; mol dm^{-3} for molar; etc. Usually, only one type of value (*e.g.*, mass per cent) is found in the original paper, and the compiler has added the other type of value (*e.g.*, mole per cent) from computer calculations based on 1989 atomic weights (2). Temperatures are expressed as $t/^{\circ}C$, $t/^{\circ}F$ or T/K as in the original; if necessary, conversions to T/K are made, sometimes in the compilations and always in the critical evaluation. However, the author's units are expressed according to IUPAC recommendations (3) as far as possible.

Errors in calculations, fitting equations, etc. are noted, and where possible corrected. Material inserted by the compiler is identified by the word "compiler" or by the compiler's name in parentheses or in a footnote. In addition, compiler-calculated values of mole or mass fractions are included if the original data do not use these units. If densities are reported in the original paper, conversions from concentrations to mole fractions are included, but otherwise this is done in the evaluation, with the values and sources of the densities being quoted and referenced.

Details of smoothing equations (with limits) are included if they are present in the original publication and if the temperature or pressure ranges are wide enough to justify this procedure and if the compiler finds that the equations are consistent with the data.

The precision of the original data is preserved when derived quantities are calculated, if necessary by the inclusion of one additional significant figure. In some cases, compilers note that numerical data have been obtained from published graphs using digitizing techniques. In these cases, the precision of the data can be determined by the quality of the original graph and the limitations of the digitizing technique. In some cases graphs have been included, either to illustrate data more clearly, or if this is the only information in the original. Full grids are not usually inserted as it is not intended that users should read data from the graphs.

Method: The apparatus and procedure are mentioned briefly. Abbreviations used in Chemical Abstracts are often used here to save space, reference being made to sources of further detail if these are cited in the original paper.

Source and Purity of Materials: For each component, referred to as (1), (2), etc., the following information (in this order and in abbreviated form) is provided if available in the original paper: source and specified method of preparation; properties; degree of purity.

Estimated Error: If estimated errors were omitted by the original authors, and if relevant information is available, the compilers have attempted to estimate errors (identified by "compiler" or the compiler's name in parentheses or in a footnote) from the internal consistency of data and type of apparatus used. Methods used by the compilers for estimating and reporting errors are based on Ku and Eisenhart (4).

Comments and/or Additional Data: Many compilations include this section which provides short comments relevant to the general nature of the work or additional experimental and thermodynamic data which are judged by the compiler to be of value to the reader.

References: The format for these follows the format for the Original Measurements box, except that final page numbers are omitted. References (usually cited in the original paper) are given where relevant to interpretation of the compiled data, or where cross-reference can be made to other compilations.

Evaluations

The evaluator's task is to assess the reliability and quality of the data, to estimate errors where necessary, and to recommend "best" values. The evaluation takes the form of a summary in which all the data supplied by the compiler have been critically reviewed. There are only three boxes on a typical evaluation sheet, and these are described below.

Components: The format is the same as on the Compilation sheets.

Evaluator: The name and affiliation of the evaluator(s) and date up to which the literature was checked.

Critical Evaluation: **Critical text.** The evaluator checks that the compiled data are correct, assesses their reliability and quality, estimates errors where necessary, and recommends numerical values based on all the published data (including theses, patents and reports) for each given system. Thus, the evaluator reviews the merits or shortcomings of the various data. Only published data are considered. Documented rejection of some published data may occur at this stage, and the corresponding compilations may be removed.

The solubility of comparatively few systems is known with sufficient accuracy to enable a set of recommended values to be presented. Although many systems have been studied by at least two workers, the range of temperatures is often sufficiently different to make meaningful comparison impossible.

Occasionally, it is not clear why two groups of workers obtained very different but internally consistent sets of results at the same temperature, although both sets of results were obtained by reliable methods. In such cases, a definitive assessment may not be possible. In some cases, two or more sets of data have been classified as tentative even though the sets are mutually inconsistent.

Fitting equations. If the use of a smoothing equation is justifiable the evaluator may provide an equation representing the solubility as a function of the variables reported on all the compilation sheets, stating the limits within which it should be used.

Graphical summary. In addition to the fitting equation, graphical summaries are often given.

Recommended values. Data are recommended if the results of at least two independent groups are available and they are in good agreement, and if the evaluator has no doubt as to the adequacy and reliability of the applied experimental and computational procedures. Data are reported as tentative if only one set of measurements is available, or if the evaluator considers some aspect of the computational or experimental method as mildly undesirable but estimates that it should cause only minor errors. Data are considered as doubtful if the evaluator considers some aspect of the computational or experimental method as undesirable but still considers the data to have some value where the order of magnitude of the solubility is needed. Data determined by an inadequate method or under ill-defined conditions are rejected. However, references to these data are included in the evaluation together with a comment by the evaluator as to the reason for their rejection.

References. All pertinent references are given here, including all those publications appearing in the accompanying compilation sheets and those which, by virtue of their poor precision, have been rejected and not compiled.

Units. While the original data may be reported in the units used by the investigators, the final recommended values are reported in SI units (3) when the data can be accurately converted.

C. Quantities and Units Used in Compilation and Evaluation of Solubility Data

Mixtures, Solutions and Solubilities

A *mixture* (5) describes a gaseous, liquid or solid phase containing more than one substance, where the substances are all treated in the same way.

A *solution* (5) describes a liquid or solid phase containing more than one substance, when for convenience one of the substances, which is called the *solvent*, and may itself be a mixture, is treated differently than the other substances, which are called *solutes*. If the sum of the mole fractions of the solutes is small compared to unity, the solution is called a *dilute solution*.

The *solubility* of a solute 1 (solid, liquid or gas) is the analytical composition of a saturated solution, expressed in terms of the proportion of the designated solute in a designated solvent (6).

"Saturated" implies equilibrium with respect to the processes of dissolution and precipitation; the equilibrium may be stable or metastable. The solubility of a substance in metastable equilibrium is usually greater than that of the same substance in stable equilibrium. (Strictly speaking, it is the activity of the substance in metastable equilibrium that is greater.) Care must be taken to distinguish true metastability from supersaturation, where equilibrium does not exist.

Either point of view, mixture or solution, may be taken in describing solubility. The two points of view find their expression in the reference states used for definition of activities, activity coefficients and osmotic coefficients.

Note that the composition of a saturated mixture (or solution) can be described in terms of any suitable set of thermodynamic components. Thus, the solubility of a salt hydrate in water is usually given as the relative proportions of anhydrous salt in solution, rather then the relative proportions of hydrated salt and water.

Solubilities of solids have been the subject of research for a long time, and have been expressed in a great many ways, as described below. In each case, specification of the temperature and either partial or total pressure of the saturating gaseous component is necessary. The nomenclature and units follow, where possible, the IUPAC *Green Book* (3). A few quantities follow the ISO standards (7) or the German standard (8); see a review by Cvitaš (9) for details.

A note on nomenclature. The nomenclature of the IUPAC *Green Book* (3) calls the solute component B and the solvent component A. In compilations and evaluations, the first-named component (component 1) is the solute, and the second (component 2 for a two-component system) is the solvent. The reader should bear these distinctions in nomenclature in mind when comparing equations given here with those in the *Green Book.*

Mole fraction of substance 1, x_1 or $x(1)$ (condensed phases), y_1 (gases):

$$x_1 = \frac{n_1}{\displaystyle\sum_{s=1}^{c} n_s} \tag{7}$$

where n_s is the amount of substance of s, and c is the number of distinct substances present (often the number of thermodynamic components in the system). *Mole per cent* of substance 1 is $100x_1$.

Ionic mole fractions of salt i, x_{+i}, x_{-i}: For a mixture of s binary salts i, each of which ionizes completely into ν_{+i} cations and ν_{-i} anions, with $\nu_i = \nu_{+i} + \nu_{-i}$ and a mixture of p non-electrolytes k, of which some may be considered as solvent components, a generalization of the definition in (10) gives:

$$x_{+i} = \frac{\nu_{+i} x_i}{1 + \displaystyle\sum_{j=1}^{s} (\nu_j - 1) x_j}, \quad x_{-i} = \frac{\nu_{-i} x_{+i}}{\nu_{+i}}, \qquad i = 1, \cdots, s, \tag{8}$$

$$x_{0k} = \frac{x_k}{1 + \displaystyle\sum_{j=1}^{s} (\nu_j - 1) x_j}, \qquad k = (s+1), \cdots, c. \tag{9}$$

The sum of these mole fractions is unity, so that, with $c = s + p$,

$$\sum_{i=1}^{s} (x_{+i} + x_{-i}) + \sum_{k=s+1}^{c} x_{0k} = 1. \tag{10}$$

General conversions to other units in multicomponent systems are complicated. For a three-component system containing non-electrolyte 1, electrolyte 2 and solvent 3,

$$x_1 = \frac{\nu_{+2} x_{01}}{\nu_{+2} - (\nu_2 - 1) x_{+2}}, \quad x_2 = \frac{x_{+2}}{\nu_{+2} - (\nu_2 - 1) x_{+2}}. \tag{11}$$

These relations are used in solubility equations for salts, and for tabulation of salt effects on solubilities of gases.

Mass fraction of substance 1, w_1 or $w(1)$:

$$w_1 = \frac{g_1}{\sum_{s=1}^{c} g_s} \tag{12}$$

where g_s is the mass of substance s. *Mass per cent* of substance 1 is $100w_1$. The equivalent terms *weight fraction*, *weight per cent* and *g(1)/100 g solution* are no longer used.

Solute mole fraction of substance 1, $x_{\nu,1}$:

$$x_{s,1} = \frac{m_1}{\sum_{s=1}^{c'} m_s} = \frac{x_1}{\sum_{s=1}^{c'} x_s} \tag{13}$$

where c' is the number of solutes in the mixture. These quantities are sometimes called Jänecke mole (mass) fractions (11, 12). *Solute mass fraction* of substance 1, $w_{s,1}$, is defined analogously.

Solvent mole fraction of substance 1, $x_{\nu,1}$:

$$x_{\nu,1} = \frac{x_1}{\sum_{s=1}^{p} x_s} \tag{14}$$

Here, p is the number of solvent components in the mixture. Solvent mass fraction of substance 1, $w_{\nu,1}$, is defined analogously.

Molality of solute 1 in a solvent 2, m_1:

$$m_1 = \frac{n_1}{n_2 M_2} \tag{15}$$

SI base units: mol kg^{-1}. Here, M_2 is the molar mass of the solvent.

Aquamolality, Solvomolality of substance 1 in a mixed solvent with components 2, 3 (13), $m_1^{(3)}$:

$$m_1^{(3)} = \frac{m_1 \bar{M}}{M_3} \tag{16}$$

SI base units: mol kg^{-1}. Here, the average molar mass of the solvent is

$$\bar{M} = x_{\nu,2} M_2 + (1 - x_{\nu,2}) M_3 \tag{17}$$

and $x_{\nu,2}$ is the solvent mole fraction of component 2. This term is used most frequently in discussing comparative solubilities in water (component 2) and heavy water (component 3) and in their mixtures.

Amount concentration of solute 1 in a solution of volume V, c_1:

$$c_1 = \text{[formula of solute]} = \frac{n_1}{V} \tag{18}$$

SI base units: mol m^{-3}. The symbol c_1 is preferred to [formula of solute], but both are used. The old terms *molarity*, *molar* and *moles per unit volume* are no longer used.

Mass concentration of solute 1 in a solution of volume V, ρ_1:

$$\rho_1 = \frac{g_1}{V} = \frac{c_1 M_1}{V} \tag{19}$$

SI base units: kg m^{-3}.

Mole ratio, $r_{A,B}$ (dimensionless) (9):

$$r_{n,12} = \frac{n_1}{n_2} \tag{20}$$

Mass ratio, symbol $\zeta_{A,B}$, may be defined analogously (9).

Ionic strength, I_m (molality basis), or I_c (concentration basis):

$$I_m = \frac{1}{2}\sum_i m_i z_i^2, \quad I_c = \frac{1}{2}\sum_i c_i z_i^2, \tag{21}$$

where z_i is the charge number of ion i. While these quantities are not used generally to express solubilities, they are used to express the compositions of non-saturating components. For a single salt i with ions of charge numbers z_+ and z_-,

$$I_m = |z_+ z_-|\, \nu m_i, \quad I_c = |z_+ z_-|\, \nu c_i. \tag{22}$$

Mole and mass fractions and mole ratios are appropriate to either the mixture or the solution point of view. The other quantities are appropriate to the solution point of view only. Conversions between pairs of these quantities can be carried out using the equations given in Table 1 at the end of this Introduction. Other useful quantities will be defined in the prefaces to individual volumes or on specific data sheets.

Salt hydrates are generally not considered to be saturating components since most solubilities are expressed in terms of the anhydrous salt. The existence of hydrates or solvates is noted carefully in the critical evaluation.

Mineralogical names are also quoted, along with their CA Registry Numbers, again usually in the text and CA Registry Numbers (where available) are given usually in the critical evaluation.

In addition to the quantities defined above, the following are useful in conversions between concentrations and other quantities.

* *Density*, ρ:

$$\rho = \frac{g}{V} = \sum_{s=1}^{c} \rho_s \tag{23}$$

SI base units: kg m^{-3}. Here g is the total mass of the system.

Relative density, $d = \rho/\rho^\circ$: the ratio of the density of a mixture at temperature t, pressure p to the density of a reference substance at temperature t', pressure p'. For liquid solutions, the reference substance is often water at 4 °C, 1 bar. (In some cases 1 atm is used instead of 1 bar.) The term *specific gravity* is no longer used.

D. Thermodynamics of Solubility

Thermodynamic analysis of solubility phenomena provides a rational basis for the construction of functions to represent solubility data, and thus aids in evaluation, and sometimes enables thermodynamic quantities to be extracted. Both these aims are often difficult to achieve because of a lack of experimental or theoretical activity coefficients. Where thermodynamic quantities can be found, they are not evaluated critically, since this task would involve examination of a large body of data that is not directly relevant to solubility. Where possible, procedures for evaluation are based on established thermodynamic methods. Specific procedures used in a particular volume will be described in the Preface to that volume.

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This section was written by:

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J. W. Lorimer,	London, Ont., Canada
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M.-T. Saugier-Cohen Adad	Villeurbanne, France

December, 1995

TABLE 1. Interconversions between Quantities Used as Measures of Solubilities c-component Systems Containing $c-1$ Solutes i and Single Solvent c.

	x_i	w_i	m_i	c_i
$x_i =$	x_i	$\dfrac{1}{1+\dfrac{M_i}{M_c}\left\{\dfrac{1}{w_i}-1+\sum_{j\neq 1}^{c-1}\left(\dfrac{M_c}{M_j}-1\right)\dfrac{w_j}{w_i}\right\}}$	$\dfrac{1}{1+\dfrac{1}{m_iM_c}+\sum_{j\neq i}^{c-1}\dfrac{m_j}{m_i}}$	$\dfrac{1}{1+\dfrac{1}{M_c}\left(\dfrac{\rho}{c_i}-M_i\right)+\sum}$
$w_i =$	$\dfrac{1}{1+\dfrac{M_c}{M_i}\left\{\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	w_i	$\dfrac{1}{1+\dfrac{1}{m_iM_i}\left(1+\sum_{j\neq i}^{c-1}m_jM_j\right)}$	$\dfrac{c_iM_i}{\rho}$
$m_i =$	$\dfrac{1}{M_c\left(\dfrac{1}{x_i}-1-\sum_{j\neq i}^{c-1}\dfrac{x_j}{x_i}\right)}$	$\dfrac{1}{M_i\left(\dfrac{1}{w_i}-1-\sum_{j\neq i}^{c-1}\dfrac{w_j}{w_i}\right)}$	m_i	$\dfrac{1}{\dfrac{1}{c_i}\left(\rho-\sum_{j\neq i}^{c-1}c_jM\right.}$
$c_i =$	$\dfrac{\rho}{M_i+M_c\left\{\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	$\dfrac{\rho w_i}{M_i}$	$\dfrac{\rho}{\dfrac{1}{m_i}\left(1+\sum_{j\neq i}^{c-1}m_jM_j\right)+M_j}$	c_i

ρ – density of solution; M_i – molar masses of i. For relations for 2-component systems, set summations to 0.

PART 1. COMPILATION AND EVALUATION OF SOLUBILITY DATA OF TRANSITION METAL HALATES

III. TRANSITION METAL CHLORATES

A. Cobalt Chlorate

Components:
(1) Cobalt chlorate; $\mathrm{Co(ClO_3)_2}$; [80546-49-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
A. Meusser, Ber. Dtsch. Chem. Ges. **35**, 1414–24 (1902).

Variables:
Temperature: 251–334 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 2. Experimental solubility of $\mathrm{Co(ClO_3)_2}$ in water

t / °C	T / K	Molar ratio[a]	$100w_1$	$100x_1$[b]
Solid phase: Ice				
0[c]	273.15	—	0[c]	0
−12	261	3.41	29.97	3.30
−22	251	4.76	37.40	4.55
Solid phase: $\mathrm{Co(ClO_3)_2 \cdot 6H_2O}$				
−30[d]	243	8.57[d]	51.80[b]	7.90
−21	252	9.08	53.30	8.34
−20[d]	253	9.11[d]	53.31[b]	8.35
−19	254	9.20	53.61	8.44
−10[d]	263	9.71[d]	54.91[b]	8.85
0	273	10.75	57.45	9.72
10[d]	283	12.14[d]	60.35[b]	10.83
10.5	283.7	12.90	61.83	11.44
15[d]	288	13.29[d]	62.48[b]	11.73
17.2[d]	290.4	14.00[d]	63.70[b]	12.28
18.3[d]	291.5	15.00[d]	65.28[b]	13.04
19.3[d]	292.5	16.64[d]	67.55[b]	14.24
Solid phase: $\mathrm{Co(ClO_3)_2 \cdot 4H_2O}$				
18	291	14.28	64.19	12.51
20[d]	293	14.46[d]	64.45[b]	12.63
21	294	14.51	64.39	12.61
30[d]	303	15.45[d]	65.95[b]	13.38
35	308	16.10	67.09	13.69
40[d]	313	16.82[d]	67.83[b]	14.40

47	320	18.29	69.66	15.48
50[d]	323	18.80[d]	70.21[b]	15.83
60[d]	333	23.41[d]	74.59[b]	18.97
60.7[d]	333.9	24.00[d]	75.05[b]	19.35
61	334	25.39	76.12	20.27

[a] Molar ratio is amount of $\mathrm{Co(ClO_3)_2}$ mol per 100 mol of water.

[b] Calculated by the compilers

[c] Pure water

[d] Read out by the compilers from Fig. 1 of the original paper

The author reported the specific density of the saturated solution of 1.861 g cm^{-3} at $18\ ^\circ\text{C}$. During thermal analysis of the solid phases, two phase transition temperatures were observed:

$$\mathrm{Co(ClO_3)_2 \cdot 6H_2O} \longrightarrow \mathrm{Co(ClO_3)_2 \cdot 4H_2O} \text{ at } 18\ ^\circ\text{C},$$
$$\mathrm{Co(ClO_3)_2 \cdot 4H_2O} \longrightarrow \text{Cl, O, and } \mathrm{Co_2O_3} \text{ at } \sim 61\ ^\circ\text{C}$$

$\mathrm{Co(ClO_3)_2 \cdot 2H_2O}$ was obtained by dehydration of $\mathrm{Co(ClO_3)_2 \cdot 4H_2O}$ with $\mathrm{P_2O_5}$. The anhydrous salt was not observed over the temperature range investigated (251–334 K).

The mole fraction solubility values obtained from the Meusser's results are used to fit to the smoothing equation given in the Preface to this volume. The detailed information of the calculation procedure for the smoothing equation has been described therein.

(1) Smoothing equation for $\mathrm{Co(ClO_3)_2 \cdot 6H_2O}$ as the solid phase (243–293 K):

$$Y_x = \frac{-77982.8132}{(T/\mathrm{K})} - 613.347442 \ln{(T/\mathrm{K})} + 3394.83967 + 1.21589042(T/\mathrm{K}), \tag{24}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^6(3+6)^{3+6}}{6^6(1+x)^{3+6}}\right). \tag{25}$$

$\sigma_Y = 0.031$ and $\sigma_x = 0.0035$ were obtained.

(2) Smoothing equation for $\mathrm{Co(ClO_3)_2 \cdot 4H_2O}$ as the solid phase (289–334 K):
The reported solubility of 76.12 mass % (20.27 mol %) was doubtful value because $\mathrm{Co(ClO_3)_2 \cdot 4H_2O}$ was decomposed at $\sim 61\ ^\circ\text{C}$.

$$Y_x = \frac{-165674.486}{(T/\mathrm{K})} - 1096.65274 \ln{(T/\mathrm{K})} + 6260.60574 + 1.82426017(T/\mathrm{K}), \tag{26}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^4(3+4)^{3+4}}{4^4(1+x)^{3+4}}\right). \tag{27}$$

$\sigma_Y = 0.014$ and $\sigma_x = 0.0023$ were obtained.

(3) Smoothing equation for ice as the solid phase (251–273 K):

$$x = -6.63268251 \times 10^{-5}(T/\mathrm{K})^2 + 0.0327114337(T/\mathrm{K}) - 3.98642251 \tag{28}$$

Two reported solubilities and melting point of ice gave this quadratic equation.

The calculated smoothing curves are plotted in the Fig. 1. During analysis of the solubility curve, peritectic point was obtained at 289 K. The descrepancy between the result of thermal analysis and that of solubility analysis was found to be small. The solubilities calculated from the smoothing equations at various temperatures are given in Table 3; these values are designated as tentative.

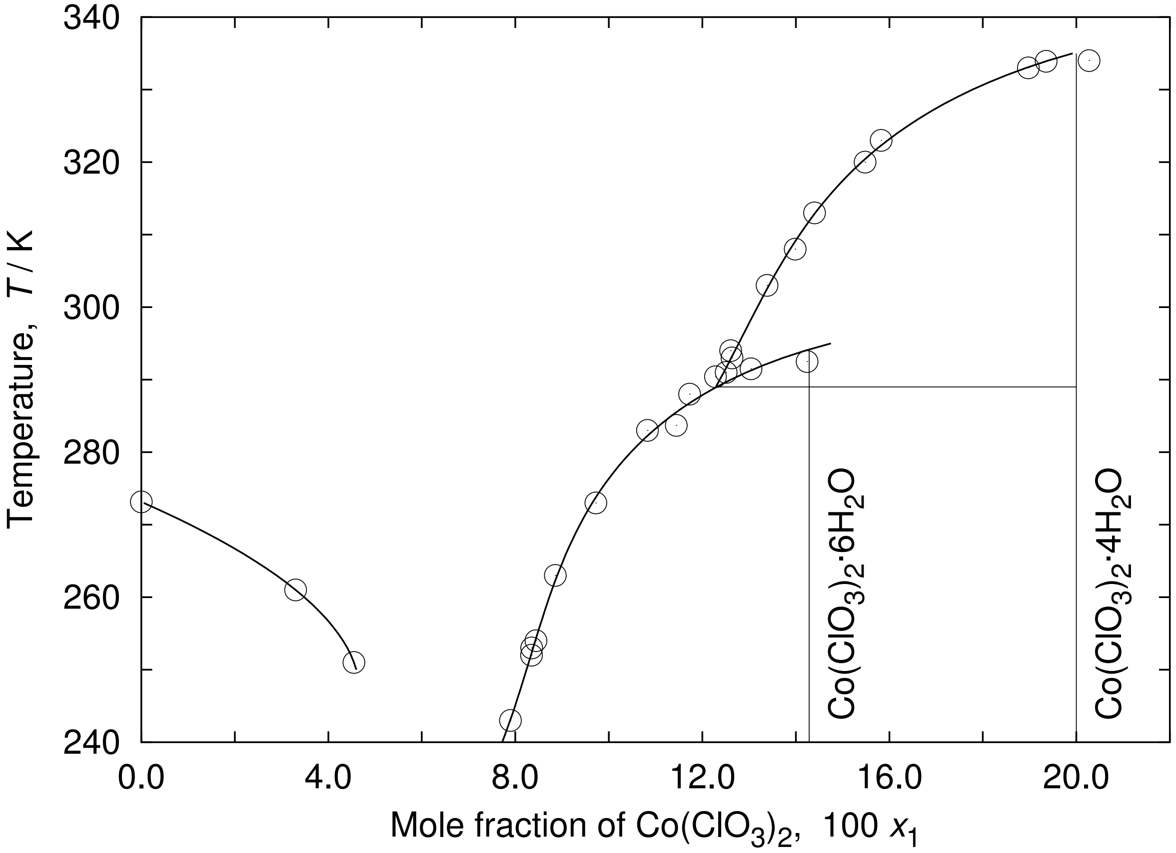

FIG. 1. Partial phase diagram of the $Co(ClO_3)_2$–H_2O system

TABLE 3. Tentative solubility of $Co(ClO_3)_2$ in water

T / K	$100x_1$
Solid phase: Ice	
270	1.04
265	2.4
260	3.5
255	4.2
Solid phase: $Co(ClO_3)_2 \cdot 6H_2O$	
248	8.1
253	8.4
258	8.6
263	8.9
268	9.2
273	9.7
278	10.2
283	11.0
288	12.0
Solid phase: $Co(ClO_3)_2 \cdot 4H_2O$	
289	12.3

293	12.6
298	13.0
303	13.4
308	13.9
313	14.4
318	15.1
323	16.0
328	17.2
333	18.9

Auxiliary Information

Method/Apparatus/Procedure:

Cobalt chlorate hydrate and water were placed in a test tube and shaken for 1 h. Aliquots of saturated solution were withdrawn with a pipet, and the cobalt content was determined gravimetrically as $CoSO_4$. Temperature was probably controlled by a thermostat. Analysis of the solid phase gave the following results: $Co(ClO_3)_2 \cdot 6H_2O$—66.50 mass % $Co(ClO_3)_2$ and 33.50 mass % H_2O, this gives a mole ratio of H_2O to $Co(ClO_3)_2$ of 6.30; $Co(ClO_3)_2 \cdot 4H_2O$—53.74 mass % $Co(ClO_3)_2$ and 24.46 mass % H_2O, this gives a mole ratio of H_2O to $Co(ClO_3)_2$ of 4.05. Thermal analysis of the solid was performed with the use of a thermometer.

Source and Purity of Materials:

Cobalt chlorate was prepared from cobalt sulfate and barium chlorate. Water was evaporated and the crystals recrystallized, if needed. “Very pure” salts from Kahlbaum were used.

Estimated Error:

Solubility: the difference between the theoretical and practically determined stoichiometry coefficient is up to 5 %. It seems that the solubility determination precision is not better than ± 1 % (compilers).

Temperature: no better than ± 0.5 K (compilers).

B. Nickel Chlorate

Components:

(1) Nickel chlorate; $Ni(ClO_3)_2$; [67952-43-6]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

A. Meusser, Ber. Dtsch. Chem. Ges. **35**, 1414–24 (1902).

Variables:

Temperature: 255–353 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 4. Experimental solubility of $Ni(ClO_3)_2$ in water

t / °C	T / K	Molar ratio[a]	$100w_1$	$100x_1$[b]
		Solid phase: Ice		
0[c]	273.15	—	0[c]	0
−9	264	2.90	26.62	2.81
−13.5	259.7	3.73	31.85	3.59
		Solid phase: $Ni(ClO_3)_2 \cdot 6H_2O$		
−30[d]	243	7.00[d]	46.71[b]	6.54
−20[d]	253	7.68[d]	49.08[b]	7.15
−18	255	7.84	49.55	7.27
−10[d]	263	8.29[d]	50.92[b]	7.65
−8	265	8.49	51.52	7.82
0	273	8.88	52.66	8.16
10[d]	283	9.75[d]	54.97[b]	8.88
18	291	10.47	56.74	9.48
20[d]	293	10.75[d]	57.38[b]	9.70
30[d]	303	12.21[d]	60.47[b]	10.89
40	313	15.35	64.47	12.66
40[d]	313	16.64[d]	67.58[b]	14.27
		Solid phase: $Ni(ClO_3)_2 \cdot 4H_2O$		
45[d]	318	16.21[d]	67.00[b]	13.95
48.5	321.7	16.65	67.60	14.28
50[d]	323	16.86[d]	67.88[b]	14.44
55	328	17.59	68.78	14.96
60[d]	333	18.43[d]	69.77[b]	15.56
65	338	18.01	69.05	15.12
70[d]	343	20.64[d]	72.11[b]	17.11
79.5	352.7	24.68	75.50	19.75

[a] Molar ratio is amount of $Ni(ClO_3)_2$ mol per 100 mol of water.

[b] Calculated by the compilers

[c] Pure water
[d] Read out by the compilers from Fig. 1 of the original paper

Transition of $\mathrm{Ni(ClO_3)_2 \cdot 6H_2O}$ to $\mathrm{Ni(ClO_3)_2 \cdot 4H_2O}$ was observed at 39 °C. $\mathrm{Ni(ClO_3)_2 \cdot 4H_2O}$ is too unstable to enable the existence of the dihydrate salt to be established. The tetrahydrated salt melts at about 80 °C and lose water, decomposing into Cl_2, O_2 and $\mathrm{Ni_2O_3}$. Specific densities of the saturated solution at 18 °C was found to be 1.661 g $\mathrm{cm^{-3}}$. The anhydrous salt was not observed over the temperature range investigated (255–353 K).

The observed solubility data based on mole fraction obtained from the Meusser's results are used to fit to the smoothing equation given in the Preface in this volume. The detailed information of the calculation procedure for the smoothing equation has been described therein. The calculated smoothing equations are given below.

(1) Smoothing equation for $\mathrm{Ni(ClO_3)_2 \cdot 6H_2O}$ as the solid phase (243–313 K):

$$Y_x = \frac{-53941.5373}{(T/\mathrm{K})} - 399.967266 \ln{(T/\mathrm{K})} + 2235.83245 + 0.753336917(T/\mathrm{K}), \tag{29}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^6(3+6)^{3+6}}{6^6(1+x)^{3+6}}\right). \tag{30}$$

$\sigma_Y = 0.031$ and $\sigma_x = 0.0041$ were obtained.

(2) Smoothing equation for $\mathrm{Ni(ClO_3)_2 \cdot 4H_2O}$ as the solid phase (313–353 K):

$$Y_x = \frac{-440243.452}{(T/\mathrm{K})} - 2674.19415 \ln{(T/\mathrm{K})} + 15500.1466 + 4.06843443(T/\mathrm{K}), \tag{31}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^4(3+4)^{3+4}}{4^4(1+x)^{3+4}}\right). \tag{32}$$

$\sigma_Y = 0.037$ and $\sigma_x = 0.0046$ were obtained.

(3) Smoothing equation for ice as the solid phase (259–273 K):

$$x = -9.34788224 \times 10^{-5}(T/\mathrm{K})^2 + 0.0471352254(T/\mathrm{K}) - 5.90044565 \tag{33}$$

Two reported solubilities and melting point of ice gave this quadratic equation.

The calculated smoothing curves are plotted in the Fig. 2. The solubilities calculated from the smoothing equations at various temperatures are given in Table 5; these values are designated as tentative.

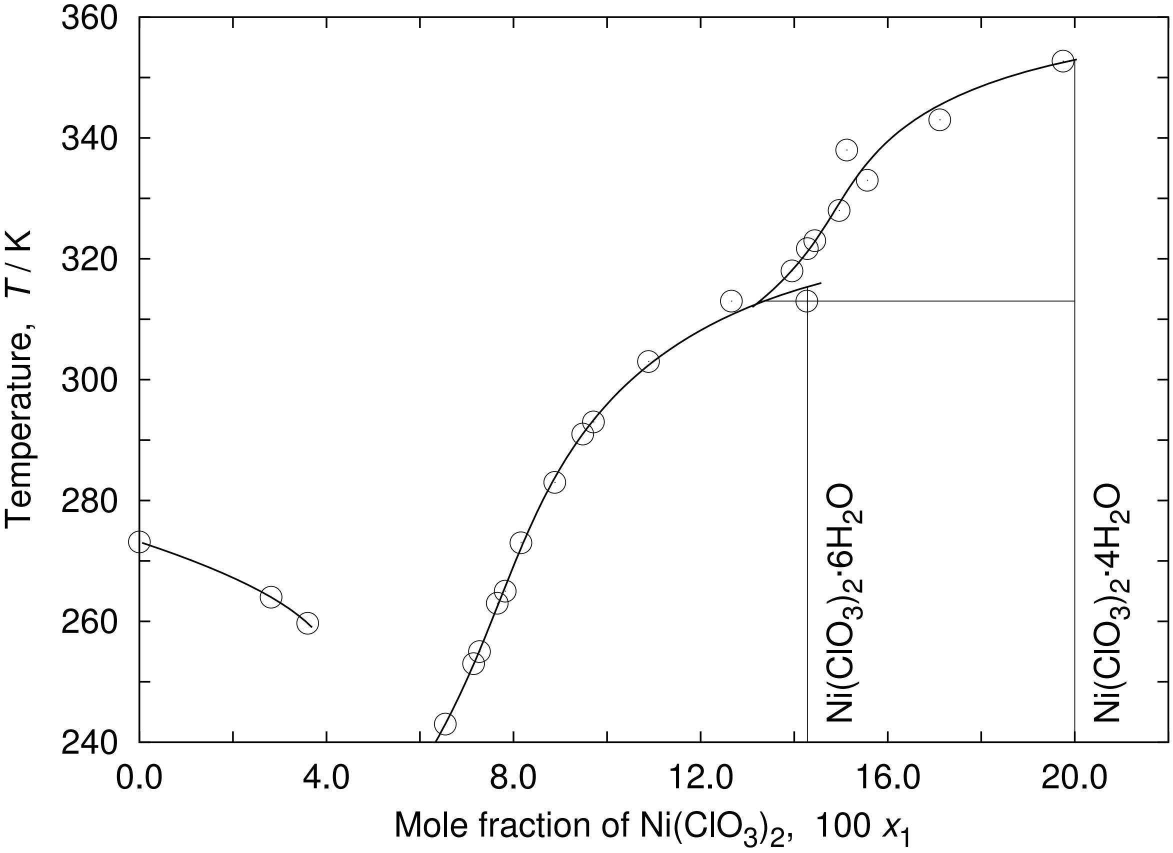

FIG. 2. Partial phase diagram of the $Ni(ClO_3)_2$–H_2O system

TABLE 5. Tentative solubility of $Ni(ClO_3)_2$ in water

T / K	$100x_1$
Solid phase: Ice	
270	1.15
265	2.6
260	3.6
Solid phase: $Ni(ClO_3)_2 \cdot 6H_2O$	
253	7.2
258	7.4
263	7.7
268	7.9
273	8.2
278	8.5
283	8.8
288	9.2
293	9.7
298	10.3
303	11.0
308	12.0

313	13.4
Solid phase: $Ni(ClO_3)_2 \cdot 4H_2O$	
313	13.3
318	13.9
323	14.5
328	14.9
333	15.3
338	15.8
343	16.6
348	17.8
353	20.0

Auxiliary Information

Method/Apparatus/Procedure:

Nickel chlorate hydrate and water were placed in a test tube and shaken for 1 h. Temperature was probably controlled by a thermostat. Aliquots of saturated solution were withdrawn with a pipet, and the nickel content was determined as $NiSO_4$. Analysis of the solid phase gave the following results: $Ni(ClO_3)_2 \cdot 6H_2O$—65.54 mass % $Ni(ClO_3)_2$ and 34.46 mass % H_2O, this gives a mole ratio of H_2O to $Ni(ClO_3)_2$ of 6.58; $Ni(ClO_3)_2 \cdot 4H_2O$—75.81 mass % $Ni(ClO_3)_2$ and 24.19 mass % H_2O, this gives a mole ratio of H_2O to $Ni(ClO_3)_2$ of 4.00. Thermal analysis of the solid was performed with the use of a thermometer.

Source and Purity of Materials:

Nickel chlorate was prepared from nickel sulfate and barium chlorate. The crystals were recrystallized in a case of need. "Very pure" salts from Kahalbaum were used.

Estimated Error:

Solubility: the difference between the theoretical and determined stoichiometry coefficient is up to 10 %. It seems that the determination precision is not better than ± 1 % (compilers).
Temperature: no better than ± 0.5 K (compilers).

IV. TRANSITION METAL IODATES

A. Titanium Iodate

Components:

(1) Titanium iodate; $Ti(IO_3)_4$; [73621-77-9]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

The solubility of titanium iodate in pure water at 298 K have been reported in two publications (1, 2). These studies were performed by Shklovskaya et al. (1, 2) with ternary systems and the binary system was given as one point on a corresponding phase diagram. The stable solid in equilibrium with the saturated solution was $Ti(IO_3)_4 \cdot 2H_2O$. The values reported in 2 publications are identical, and the solubility of 6.3×10^{-5} mol kg^{-1} at 298 K is designated as a tentative value.

The Ternary System

Solubility data for the ternary systems have been reported in 2 publications (1, 2). The $Ti(IO_3)_4$–$LiIO_3$–H_2O (1) and $Ti(IO_3)_4$–$CsIO_3$–H_2O (2) systems were studied by group of Shklovskaya (1, 2). In both systems, the solid solutions are the equilibrium solid phases. The reader finds the detailed compilation of the ternary $Ti(IO_3)_4$–$LiIO_3$–H_2O system in (3).

References:

1. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A Kuzina, and T. E. Vdovkina, Zh. Neorg. Khim. **27**, 513 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 292 (1982).

2. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, T. V. Vdovkina, and V. A Kuzina, Zh. Neorg. Khim. **30**, 1338 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 763 (1985).

3. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 305.

Components:

(1) Titanium iodate; $\mathrm{Ti(IO_3)_4}$; [73621-77-9]
(2) Cesium iodate; $CsIO_3$; [13454-81-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, T. V. Vdovkina, and V. A. Kuzina, Zh. Neorg. Khim. **30**, 1338–9 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 763–4 (1985).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 6. Composition of saturated solutions in the ternary $\mathrm{Ti(IO_3)_4}$–$CsIO_3$–H_2O system at 25 °C

$\mathrm{Ti(IO_3)_4}$		$CsIO_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.0047[c]	1.1×10^{-4}	—	—	A
0.0038	9.2×10^{-5}	0.13	0.0076	B
0.0016	3.9×10^{-5}	0.27	0.016	B
0.0012	2.9×10^{-5}	0.51	0.030	B
0.00098	2.4×10^{-5}	0.68	0.040	B
0.00077	1.9×10^{-5}	0.87	0.051	B
0.00070	1.7×10^{-5}	1.09	0.065	C
0.00065	1.6×10^{-5}	1.41	0.084	C
0.00058	1.4×10^{-5}	1.76	0.105	C
0.00051	1.3×10^{-5}	1.99	0.119	C
0.00048	1.2×10^{-5}	2.11	0.126	C
0.00054[d]	1.3×10^{-5}	2.46[d]	0.147	C+D
—	—	2.61[c]	0.157	D

[a] Calculated by the compiler.

[b] The solid phases are: A = $\mathrm{Ti(IO_3)_4 \cdot 2H_2O}$; B = solid solution based on $\mathrm{Ti(IO_3)_4 \cdot 2H_2O}$; C = solid solution based on $\mathrm{CsIO_3 \cdot Ti(IO_3)_4 \cdot 2H_2O}$; D = $CsIO_3$.

[c] For binary systems the compiler computes the following values:

Solubility of $\mathrm{Ti(IO_3)_4}$ = 6.3×10^{-5} mol kg^{-1}
Solubility of $CsIO_3$ = 0.0871 mol kg^{-1}.

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

Isothermal method was used. Equilibrium was reached in 25–30 d. The cesium content in the coexisteing phases was determined by emission spectrometry; it was found that iodates and titanium had no influence on the cesium determination. The liquid phases were analyzed for titanium content by colorimetry with chromotropic acid after preliminary reduction of the iodate ion with hydroxylammonium sulfate in an acidic medium followed by removal of iodine by evaporation of the solutions (1), and the "residues" were analyzed for iodate ion by iodometric titration. The solid phases were identified by the "residues" method, and checked by X-ray diffraction.

Source and Purity of Materials:

"Highly pure" cesium iodate was used. Titanium iodate was synthesized by the reaction of freshly precipitated titanium hydroxide and iodic acid.

Estimated Error:

Solubility: precision 2–8 %.

Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A. Kuzina, and T. E. Vdovkina, Zh. Neorg. Khim. **27**, 513 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 292 (1982).

B. Manganese Iodate

Components:
(1) Manganese iodate; $\mathrm{Mn(IO_3)_2}$; [25659-29-4]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. March 2002.

Critical Evaluation:

The Binary System

Data for the solubility of manganese(II) iodate in water have been reported in 5 publications (1–5). Hayes and Martin (1) measured the solubility over a wide temperature range of 276–363 K. The result of Serebrennikova's group (5) at 298 K is in a good agreement with that of (1). The solubility of $\mathrm{Mn(IO_3)_2}$ in water was reported in the studies (2, 3) concerning the solubility in mixed aqueous-organic solvents. The solubility study of (4) deals with a ternary system, and the solubility in the binary system was treated as one point in this ternary system. The solubility values of (1) and (5) at 298 K are in good agreement, however, the scatter of all data is significant (up to 10 %); see the Figure. The result of (4) is 6 % lower than that of (1) and (5). A summary of solubility studies for the binary $\mathrm{Mn(IO_3)_2}$–H_2O system is given in Table 7.

TABLE 7. Summary for solubility studies of $\mathrm{Mn(IO_3)_2}$ in water at various temperatures

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method	Reference
276	5.68^a	Iodometry	(1)
293	5.77	Iodometry	(3)
298	6.20	Iodometry	(2, 3)
298	6.58^a	Iodometry	(1)
298	6.58	Radiometry	(5)
303	6.33	Iodometry	(3)
318	7.13^a	Iodometry	(1)
323	6.59	Complexometry ($\mathrm{Mn^{2+}}$); Iodometry ($\mathrm{IO_3^-}$)	(4)
363	8.54^a	Iodometry	(1)

^{a}in mol kg^{-1}.

The solubility data given in Table 7 were fitted to the smoothing equation given in the Preface in this volume. The detailed information of the calculation procedure for the smoothing equation has been described therein. The experimental solubilities based on mol dm^{-3} units were fitted to give the smoothing equation:

$$\ln\left(\frac{c_{\mathrm{i}}}{\text{mol dm}^{-3}}\right) = \frac{-17771.263}{(T/\mathrm{K})} - 113.94286\,\ln(T/\mathrm{K}) + 648.2121 + 0.186238926\,(T/\mathrm{K}), \quad (34)$$

where $\sigma_c = 0.00034$. The calculated smoothing curves are plotted in the Fig. 3. The solubilities calculated from the smoothing equation are also given in Table 8; these values are designated as tentative solubilities. The redetermination over a wide temperature range are required.

TABLE 8. Doubtful solubility of $\mathrm{Mn(IO_3)_2}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}
273	5.5
283	5.9
293	6.2
303	6.4
313	6.7
323	6.9
333	7.2
343	7.5
353	8.0
363	8.5

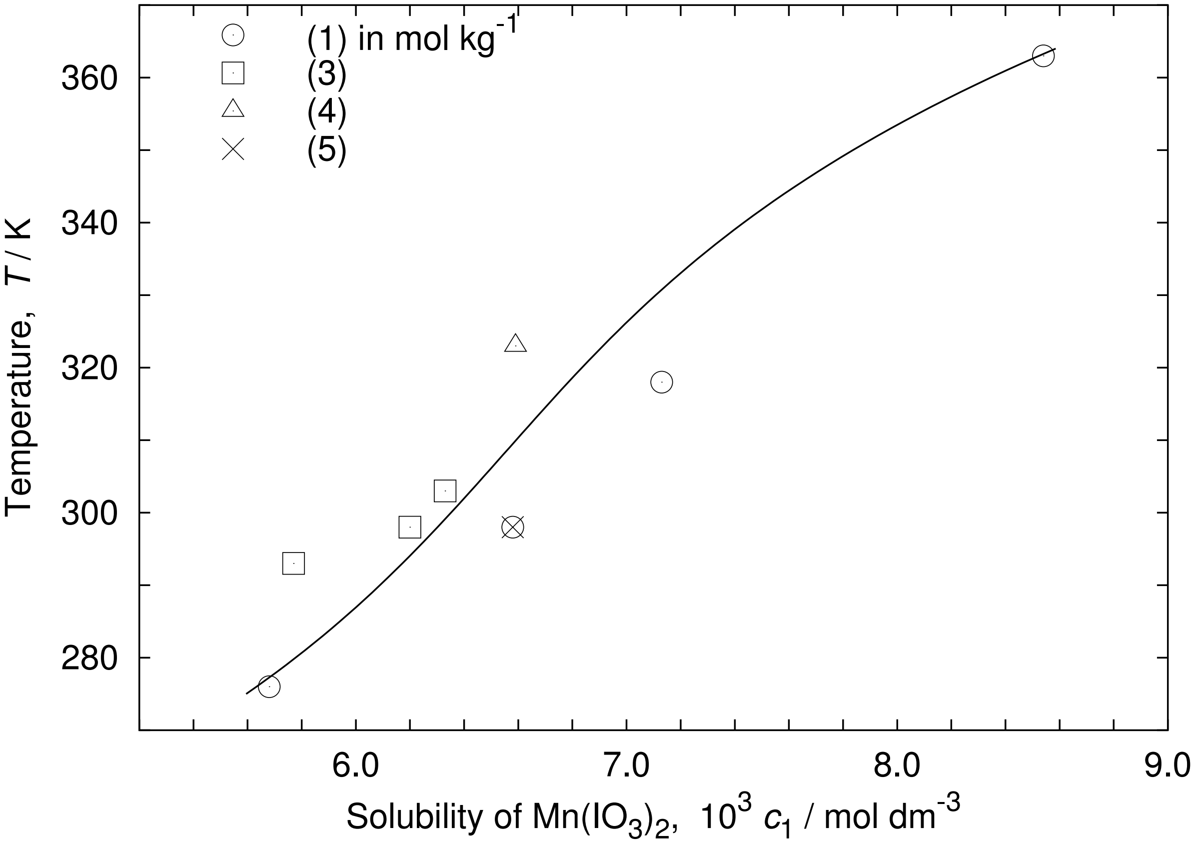

FIG. 3. Temperature dependence of solubility of $\mathrm{Mn(IO_3)_2}$ in water

The Ternary System

Hayes and Martin (1) measured the solubilities of $\mathrm{Mn(IO_3)_2}$ in presence of added salt at 276 and 298 K. The solubility in sodium chloride solution increases with an increase of the NaCl concentration at both temperatures. In the case of potassium iodate added the solubility of manganese iodate decreased. Serebrennikova et al. (5) studied the solubility in aqueous solutions of alkali metal iodates. The solubility decreases with increasing concentration of the added salts in such system.

Miyamoto's group (2, 3) measured the solubilities of $\mathrm{Mn(IO_3)_2}$ in tetrahydrofuran- and *N*,*N*-dimethylformamide–water mixtures. In both systems, the logarithm of the solubility of $\mathrm{Mn(IO_3)_2}$ decreased almost linearly with the reciprocal of dielectric constant of the solvent, as expected from the Born equation.

The ternary $\mathrm{Mn(IO_3)_2}$–KIO_3–H_2O system at 323 K has been studied by Vinogradov et al. (4). The solubility of manganese iodate considerably decreased in the presence of potassium iodate. The dominant feature in the system is the existence of complex compound, $\mathrm{K_2[Mn(IO_3)_4(H_2O)_2]}$ (dipotassium diaquotetraiodatomanganate(II)). Composition of solid phases in the binary $\mathrm{Mn(IO_3)_2}$–H_2O and KIO_3–H_2O systems were the anhydride salts. The detailed compilation of the paper reported by Vinogradov et al. (4) was given in (6).

The solubilities of $\mathrm{Mn(IO_3)_2}$ in aqueous solution of manganese(II) perchlorate at 298 K were measured by Hayes and Martin (1). The manganese(II) perchlorate solution contained a small excess of perchloric acid. The solubility in this solution decreased with an increase of the manganese(II) perchlorate concentration.

References:

1. A. M. Hayes and D. S. Martin, Jr., J. Am. Chem. Soc. **73**, 4853 (1951).
2. H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659.
3. H. Miyamoto, M. Yamamoto, and Y. Maruyama, Nippon Kagaku Kaishi **1979**, 546.
4. E. E. Vinogradov, I. M. Karataeva, and I. N. Lepeshkov, Zh. Neorg. Khim. **24**, 223 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 124 (1979).
5. G. M. Serebrennikova, L. V. Vasil'eva, V. K. Malashenkova, and V. I. Morozov, Zh. Neorg. Khim. **24**, 1378 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 764 (1979).
6. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 399.

Components:
(1) Manganese iodate; $\mathrm{Mn(IO_3)_2}$; [25659-29-4]
(2) Sodium chloride; NaCl; [7647-14-5]
or (2) Potassium iodate; KIO_3; [7758-05-6]
or (2) Manganese perchlorate; $\mathrm{Mn(ClO_4)_2}$; [13770-16-6]
(3) Perchloric acid; $HClO_4$; [7601-90-3]
(4) Water; H_2O; [7732-18-5]

Original Measurements:
A. M. Hayes and D. S. Martin, Jr., J. Am. Chem. Soc. **73**, 4853–5 (1951).

Variables:
Concentration of salts
Temperature: 276–363 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 9. Solubility of $\mathrm{Mn(IO_3)_2}$ in aqueous solution of NaCl and KIO_3 at various temperatures

Additive salt	t / °C	$10^2 m_2$ / mol kg^{-1}	$10^3 m_1$ / mol kg^{-1}	$10^6 K_\mathrm{s}$ / mol^3 kg^{-9}
None	3	—	5.68^a	0.352 (0.733)^b
	25	—	6.58^a	0.479 (1.14)^b
	45	—	7.13^a	(1.45)^b
	90	—	8.54^a	(2.49)^b
NaCl	3	1.007	6.21	
		2.462	6.65	
		5.54	7.51	
		11.08	7.86	
		40.7	9.02	
	25	1.007	6.94	
		2.462	7.61	
		5.54	8.36	
		11.08	8.96	
		40.7	10.41	
KIO_3	25	0.688	4.74	
		2.213	2.11	
		6.22	0.685	
		10.91	0.301	

[a] Estimated by extrapolation to zero ionic strength.
[b] Calculated by the compilers for concentrations using $K_\mathrm{s} = 4m_1^3$, where m_1 is the solubility of $\mathrm{Mn(IO_3)_2}$ in water at zero ionic strength.

TABLE 10. Solubility of $\mathrm{Mn(IO_3)_2}$ in aqueous solutions of $\mathrm{Mn(ClO_4)_2}$ containing $HClO_4$ at 25 °C

pH	$10^2 m_2$ / mol kg^{-1}	$10^3 m_1$ / mol kg^{-1}
4.03	0	6.68
2.63	0.743	5.60
3.00	1.030	5.31

Auxiliary Information

Method/Apparatus/Procedure:

Freshly prepared aqueous solutions in contact with solid manganese(II) iodate were shaken in glass-stoppered flasks for 24 h in a water thermostat regulated at the selected temperature except 90 °C. A manually controlled water-bath was used at 90 °C. After withdrawing and analyzing aliquots from these samples, the flasks were placed in the other temperature bath and shaken for 6 h. Aliquots were again withdrawn and analyses performed. The density determination was also done. The aqueous manganese iodate solutions containing manganese perchlorate and sodium chloride were analyzed by adding HCl and KI and titrating the liberated iodine with sodium thiosulfate. Aliquots from the solutions containing KIO_3 were oxidized to permanganate with periodic acid in the presence of sulfuric acid and compared colorimetrically in a spectrophotometer with a standard prepared by identical treatment from a standard solution of manganese(II) perchlorate. The pH of each aliquot was measured by a pH meter.

Source and Purity of Materials:

Manganese(II) iodate was prepared by precipitation from aqueous solutions of reagent grade iodic acid and manganese(II) chloride. The precipitate was filtered on a glass sinter and washed with water several times. The vacuum-dried salt (anhydrous) was analyzed for manganese and iodate. Calcd. Mn content in $\mathrm{Mn(IO_3)_2}$ was 13.57% and found 13.5%. Manganese perchlorate solution was prepared by dissolving very pure manganese metal in perchloric acid. The solutions contained a small excess of perchloric acid which could be determined from the pH measurements. Reagent grade sodium chloride and potassium iodate were used. Redistilled water from alkali permanganate was used.

Estimated Error:

Solubility: precision within ± 0.2 %.

Temperature: stability ± 0.02 K.

Components:

(1) Manganese iodate; $\mathrm{Mn(IO_3)_2}$; [25659-29-4]

(2) Tetrahydrofuran; $\mathrm{C_4H_8O}$; [109-99-9]

(3) Water; $\mathrm{H_2O}$; [7732-18-5]

Original Measurements:

H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659–61.

Variables:

Concentration of tetrahydorofuran: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 11. Solubility of $\mathrm{Mn(IO_3)_2}$ in tetrahydrofuran–water mixtures at 25.00 °C

$100w_2$	$100x_2$[a]	10^3c_1 / mol dm^{-3}
0	0	6.20
5	1.3	4.50
10	2.7	3.29
15	4.2	2.50
20	5.9	1.89
25	7.7	1.42
30	9.7	1.07
40	14.3	0.65

[a] Calculated by the compiler.

Auxiliary Information

Method/Apparatus/Procedure:

An excess of manganese iodate crystals and the solvent mixtures were placed into glass-stoppered bottles. The bottles were rotated in a thermostat at the selected temperature for 48 h. After the saturated solutions were allowed to settle, the solutions were withdrawn through a siphon tube equipped with a sintered-glass filter. The iodate content in the saturated solution was determined iodometrically.

Source and Purity of Materials:

Anhydrous manganese iodate was prepared by dropping solutions of equivalent quantities of manganese nitrate (Wako guaranteed reagent) and potassium iodate solution. The precipitate was filtered, washed and then dried under reduced pressure. Tetrahydrofuran was distilled over sodium hydroxide and then redistilled over sodium metal.

Estimated Error:

Solubility: probable error up to $\pm 0.6 \times 10^{-4}$ mol dm^{-3} (compiler)

Temperature: precision ± 0.05 K.

Components:

(1) Manganese iodate; $\mathrm{Mn(IO_3)_2}$; [25659-29-4]

(2) *N*,*N*-Dimethylformamide (DMF); $\mathrm{C_3H_7NO}$; [68-12-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, M. Yamamoto, and Y. Maruyama, Nippon Kagaku Kaishi **1979**, 546–8.

Variables:

Concentration of DMF: 0–41 mass %

Temperature: 293.15–303.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 12. Solubility of $\mathrm{Mn(IO_3)_2}$ in *N*,*N*-dimethylformamide–water mixtures at various temperatures

t / °C	$100w_2$	$100x_2$[a]	10^3c_1 / mol dm^{-3}
20.00	0	0	5.77
	4.93	1.26	4.57
	10.02	2.671	3.58
	14.97	4.159	2.75
	20.06	5.825	2.10
	24.99	7.588	1.59
	30.24	9.653	1.17
	41.08	14.66	0.62
25.00	0	0	6.20
	5.25	1.35	4.81
	9.52	2.53	3.83
	16.36	4.599	2.74
	19.84	5.749	2.25
	24.38	7.361	1.75
	29.88	9.504	1.26
	40.00	14.11	0.68
30.00	0	0	6.33
	4.80	1.23	5.17
	9.89	2.63	3.95
	15.09	4.196	3.03
	20.01	5.807	2.37
	24.91	7.558	1.81
	29.89	9.508	1.34
	40.39	14.35	0.71

[a] Calculated by the compiler.

The equilibrium solid phase was not stated but the authors specified it as $\mathrm{Mn(IO_3)_2}$.

Auxiliary Information

Method/Apparatus/Procedure:

An excess of manganese iodate crystals and the solvent mixtures were placed into glass-stoppered bottles. The bottles were placed into a thermostat at a given temperature and rotated for 72 h. After saturation, the solutions were separated from the solid phase using a glass-sintered filter. After the saturated solutions were diluted with water, the concentration of the iodate was determined iodometrically.

Source and Purity of Materials:

Anhydrous manganese iodate was prepared by adding dilute solutions of manganese nitrate and potassium iodate to boiled aqueous potassium nitrate solution. The precipitate was washed and dried at room temperature. Dimethyl formamide (from Mitsubishi Gas Chem. Co) was distilled under reduced pressure. After the product was dried over $\mathrm{Na_2CO_3}$, the distillation of the solvent was repeated 3 times.

Estimated Error:

Solubility: probable error between $\pm 0.03 \times 10^{-4}$ and $\pm 0.06 \times 10^{-4}$ mol dm^{-3}.
Temperature: precision ± 0.05 K.

Components:

(1) Manganese iodate; $\mathrm{Mn(IO_3)_2}$; [25659-29-4]
(2) Lithium iodate; $LiIO_3$; [13765-03-2]
or (2) Sodium iodate; $NaIO_3$; [7681-55-2]
or (2) Potassium iodate; KIO_3; [7758-05-6]
or (2) Rubidium iodate; $RbIO_3$; [13446-76-9]
or (2) Cesium iodate; $CsIO_3$; [13454-81-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

G. M. Serebrennikova, L. V. Vasil'eva, V. K. Malashenkova, and V. I. Morozov, Zh. Neorg. Khim. **24**, 1378–81 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 764–6 (1979).

Variables:

Concentration of the added salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 13. Solubility of $\mathrm{Mn(IO_3)_2}$ in aqueous alkali metal iodate solutions at 25 °C

Added salt	I / mol dm^{-3}	10^3c_1 / mol dm^{-3}
None	—	6.58
$LiIO_3$	0.16	0.12
	0.32	0.098
	0.69	0.094
	1.6	0.092
	2.2	0.075
	4.4	0.0012
$NaIO_3$	0.11	0.30
	0.22	0.17
	0.46	0.072
KIO_3	0.10	0.86
	0.20	0.49
	0.41	0.038
$RbIO_3$	0.09	0.52
$CsIO_3$	0.07	0.57

The solid equilibrium phases contained solid solutions of the salts.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility in the systems was studied isothermally at the selected temperature. Equilibrium was reached in 5–7 d. An analysis for the manganese iodate content in the saturated solutions was not reported in details but the authors used the method described in (1). Content of manganese in equilibrium solid phase was found by measurement of ^{54}Mn radiation.

Source and Purity of Materials:

Nothing specified.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: nothing specified.

References:

[1] M. Hayes and D. S. Martin, Jr., J. Am. Chem. Soc. **73**, 4853 (1951).

Components:

(1) Iron(III) iodate; $Fe(IO_3)_3$; [29515-61-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

A. von Endredy, Z. Anorg. Allg. Chem. **217**, 53–61 (1934).

Variables:

Polymorphic form of $Fe(IO_3)_3$
One temperature: 293 K

Prepared By:

Cezary Guminski and Hiroshi Miyamoto

Experimental Data and Critical Evaluation

TABLE 14. Solubility of iron(III) iodate modifications in water at 20 °C

Solid phase	$10^4 c_1$ / mol dm^{-3}
α-$Fe(IO_3)_3$	6.2
β-$Fe(IO_3)_3$	5.7

Anhydrous forms of the solute are in equilibrium in the saturated solutions. Iron iodate is stable up to 130 °C. Above this temperature, the identified decomposition products are: iodine vapor and iron(III) oxide.

Iron iodate is slightly soluble in diluted H_2SO_4 and decomposes in concentrated H_2SO_4 or HNO_3, and slowly in HCl solution. Iron iodate is not dissolved to a measurable extent in diluted HNO_3 and concentrated HIO_3.

The reported solubility results seem to be tentative.

Auxiliary Information

Method/Apparatus/Procedure:

The equilibrium between the salt and the saturate solution was reached in a thermostat. The iodate content was determined iodometrically in a CO_2 atmosphere. The KI addition made reduction of iron(III) and iodate, therefore the subsequent titration of the solution with standard thiosulfate should take into account a correction for iron(III) content. Iron(III) was determined gravimetrically as Fe_2O_3 after decomposition of iodate with either H_2SO_4–SO_2 solution and H_2O_2 reoxidation or treatment with concentrated HCl.

Source and Purity of Materials:

α-$Fe(IO_3)_3$ was prepared as follows: $Fe(NO_3)_3$ was dissolved in 4 mol dm^{-3} HNO_3 and HIO_3 was dissolved in water. The $Fe(NO_3)_3$ solution was added dropwise while stirring to the HIO_3 solution. The precipitate was centrifuged and dried in a desiccator over P_2O_5. The greenish-yellow powder of α-$Fe(IO_3)_3$ was obtained.

β-$Fe(IO_3)_3$ was prepared as follows: $Fe(NO_3)_3$ was dissolved in 4 mol dm^{-3} HNO_3 and HIO_3 was dissolved in 1 mol dm^{-3} HNO_3. $Fe(IO_3)_3$ was prepared by dropwise addition of the solution of $Fe(NO_3)_3$ to the HIO_3 while boiling. Yellowish precipitate was obtained. It was dried using the same method as this described above. Analyses of the product: 9.40–9.73 % Fe and 89.83–90.61 % IO_3; the calculated composition: 9.62 % Fe and 90.38 % IO_3. All chemicals used were from Merck or Kahlbaum. They were recrystallized several times.

Estimated Error:

Nothing specified

D. Cobalt Iodate

Components:

(1) Cobalt iodate; $\mathrm{Co(IO_3)_2}$; [13455-28-2]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. March 2002.

Critical Evaluation:

The Binary System

The earliest studies on the preparation and the qualitative solubility values of cobalt iodate in water have been summarized by Mellor (1). There was reported on the existence of cobalt iodate salts with 0, 1, 1.5, 2, 3, 4, 5 or 6 water molecules for the one $\mathrm{Co(IO_3)_2}$ salt molecule. Because these papers are very old the evaluators could get only the Meusser's paper (2). Data for the solubility of cobalt iodate in water were reported in 7 publications (2–8). Meusser reported the solubility in water in mass % over a wide range of temperatures (273–373 K). This paper has been the most comprehensive source of knowledge on cobalt iodate in the earlier years. Cordfunke (3) studied the solubility for the binary $\mathrm{Co(IO_3)_2}$–H_2O system as a function of temperature (293–334 K). Any numerical data have not been reported in his article except the solubility values of the tetrahydrate and dihydrate at 293 K. The measured solubility values of the anhydride and the hydrates as a function of temperature were shown in a figure. Therefore, the corresponding data were read out from the figure by the evaluators. Other investigators (4–8) have reported individual results of the solubility in vicinity of room temperature. Trimble (4) reported the molarity solubilities of cobalt iodate in various salt solutions including the data in pure water. Fedorov's group (5, 6) obtained the solubility product of $\mathrm{Co(IO_3)_2}$ from the solubility data in solutions at various ionic strength at 298 K. Miyamoto (7) have reported the solubility of cobalt iodate in tetrahydrofuran–water mixture which included that in pure water. Paper of Lepeshkov et al. (8) was concerned to isothermal studies of a ternary system at 323 K which included data for the binary $\mathrm{Co(IO_3)_2}$–H_2O system. All experimental solubilities data including the doubtful data are collected in Table 15.

TABLE 15. Summary for solubility studies of $\mathrm{Co(IO_3)_2}$ in water

T / K	$10^2 c_1$ / mol dm^{-3}	$10^2 m_1$ / mol kg^{-1} ^{a}	$100 x_1$ ^{a}	Reference
		Solid phase: $\mathrm{Co(IO_3)_2 \cdot 4H_2O}$ (metastable)		
273	—	1.33	0.024	(2)
291	—	2.05	0.037	(2)
293	—	2.0	0.036	(3)
303	—	2.4^{b}	0.044	(3)
303	—	2.55	0.046	(2)
305	—	2.5^{b}	0.045	(3)
306	—	2.6^{b}	0.046	(3)
316	—	3.1^{b}	0.055	(3)

323	—	3.62	0.065	(2)
333	—	4.64	0.083	(2)
338	—	5.43	0.098	(2)
Solute: $Co(IO_3)_2 \cdot 2H_2O$ (crystalline)				
273	—	0.79	0.014	(2)
291	—	1.11	0.019	(2)
293	—	1.0	0.018	(3)
298	1.13	—	—	(6)
298	1.159	—	—	(7)
298	—	1.1^b	0.019	(3)
303	1.165	—	—	(4)
303	—	1.28	0.023	(2)
306	—	1.2^b	0.021	(3)
315	—	1.3^b	0.024	(3)
320	—	1.4^b	0.026	(3)
323	—	1.65^d	0.030	(2)
323	—	1.9^d	0.034	(8)
328	—	1.5^b	0.027	(3)
330	—	1.6^b	0.029	(3)
331	—	1.6^b	0.028	(3)
335	—	1.7^b	0.031	(3)
337	—	1.6^b	0.029	(3)
340	—	1.7^b	0.030	(3)
344	—	1.8^b	0.032	(3)
346	—	1.7^b	0.030	(3)
347	—	1.8^b	0.030	(3)
348	—	2.07^d	0.037	(2)
355	—	1.9^b	0.035	(3)
358	—	2.0^b	0.036	(3)
362	—	2.1^b	0.037	(3)
373	—	2.2^b	0.040	(3)
373	—	2.52	0.045	(2)
375	—	1.2^{bd}	0.022	(3)
377	—	1.6^{bd}	0.029	(3)
380	—	1.2^{bd}	0.022	(3)
382	—	1.2^{bd}	0.021	(3)
385	—	1.2^{bd}	0.022	(3)
Solid phase: $Co(IO_3)_2$ (metastable)				
291	—	2.55	0.046	(2)
303	—	2.20	0.040	(2)
323	—	2.10	0.038	(2)
333	—	1.4^b	0.025	(3)
342	—	1.4^b	0.025	(3)
343	—	1.3^b	0.024	(3)
348	—	1.85	0.033	(2)

350	—	1.4[b]	0.025	(3)
354	—	1.3[b]	0.024	(3)
363	—	1.3[c]	0.024	(3)
369	—	1.3[b]	0.023	(3)
373	—	1.70	0.031	(2)
376	—	1.3[c]	0.023	(3)
392	—	1.2[b]	0.021	(3)
399	—	1.1[b]	0.020	(3)

[a] Calculated by the evaluators from the experimental values (see compilation).

[b] Read out from the figure and calculated by the evaluators.

[c] Calculated from solubility product at $I = 0$ by the evaluators.

[d] Rejected for calculating the smoothing equation, because $|x_{\mathrm{calc}} - x_{\mathrm{obs}}| > 2\sigma_x$.

The following equilibrium solid phases have been reported in these solubility studies.

$Co(IO_3)_2 \cdot 4H_2O$	[42183-36-8]	(2), (3).
$Co(IO_3)_2 \cdot 2H_2O$	[42183-35-7]	(2), (3), (4), (6), (7), (8).
$Co(IO_3)_2$	[13455-28-2]	(1), (3).

Meusser (2) studied the binary $Co(IO_3)_3$–H_2O system and, based on the solubilities, found the tetrahydrate to be unstable in solution at all temperatures. The dihydrate was stable below 63 °C and the anhydride stable above this temperature. Meusser' solubilities which were unreliable because of the tetrahydrate metastability were discussed in terms of activity coefficient by Rosenberg (9). The solubility data reported by Meusser were also cited in the book of Linke and Seidell (10).

Cordfunke (3) examined the preparation of the hydrated cobalt iodates, redetermined the solubilities in water and the composition of equilibrium solid phases. Cordfunke showed that the solubility of $Co(IO_3)_2 \cdot 4H_2O$ is difficult to measure because of its instability in respect to the dihydrate. It seems possible to determine the solubilities after correction for the small amount of dihydrate formed during the measurements. These results appear to be in reasonable agreement with the Meusser's data. The correction method was not specified. The tetrahydrate is a metastable form transforming into dihydrate in a slow process. Anhydrous salt was formed from the dihydrate upon heating over 375–377 K but the reverse formation of dihydrate from the anhydride was not observed upon cooling.

Nassau's group (11, 12) has reviewed a study on cobalt iodate anhydride and hydrates, and performed detail studies by differential thermal and thermogravimetric analyses as well as optical absorption spectroscopy, although the solubility study was not performed. Abrahams et al. (12) have reported the results of crystallographic, magnetic and nonlinear optical survey. Elemans and Verschoor (13) also studied structures for the anhydride and the hydrates by X-ray diffraction. The existence of $Co(IO_3)_2 \cdot 4H_2O$ (beige), $Co(IO_3)_2 \cdot 2H_2O$ (pink), and $Co(IO_3)_2$ (purple) has been reported. They stated that the tetrahydrate precipitates from acid or neutral solution at (or below) room temperature. During the standing in the presence of water for a number of days the tetrahydrate converts to the dihydrate. In agreement with (3), near the boiling point of water the anhydride was the only equilibrium product. When dry, all three cobalt compounds are stable at room temperature, but in the presence of water both the tetrahydrate and the metastable anhydride can be formed.

Fig 4 shows the solubility of various forms of cobalt iodate from (3) as a function of temperature. Several data of other investigators were also added to the figure by the evaluators. The values of Trimble (4), Fedorov et al. (5, 6) and Miyamoto (7) are placed very near to the mean solubility line of (3). The reported solubilities of Meusser (2) are about 10 % larger than those of Cordfunke (3) in the whole temperature range.

The evaluators fitted the observed solubility data based on mole fraction concentration to the smoothing equation presented in the Preface in this Volume. The detailed information of the calculation procedure for the smoothing equation has been described therein. The resulting smoothing equation for $Co(IO_3)_2 \cdot 2H_2O$ (crystalline) of Meusser's (2) data, Cordfunke's (3) data and Trimble's (4) data at 273–373 K region, are given below.

$$Y_x = \frac{-64241.0366}{(T/\mathrm{K})} - 386.637977 \ln(T/\mathrm{K}) + 2218.14276 + 0.608420107(T/\mathrm{K}), \qquad (35)$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^2(3+2)^{3+2}}{2^2(1+x)^{3+2}}\right). \tag{36}$$

$\sigma_Y = 0.13$ and $\sigma_x = 1.33 \times 10^{-5}$ were obtained.

The solubilities calculated from the smoothing equations at round temperatures are designated as recommended solubilities and are given in Table 16. The solubility curve, calculated with the equation (35), is plotted in Fig. 4.

TABLE 16. Recommended solubility of $\mathrm{Co(IO_3)_2}$ (solid phase: dihydrate, crystalline) in water

T / K	$100x_1$
273	0.014
283	0.017
293	0.019
303	0.022
313	0.024
323	0.026
333	0.029
343	0.031
353	0.034
363	0.038
373	0.042

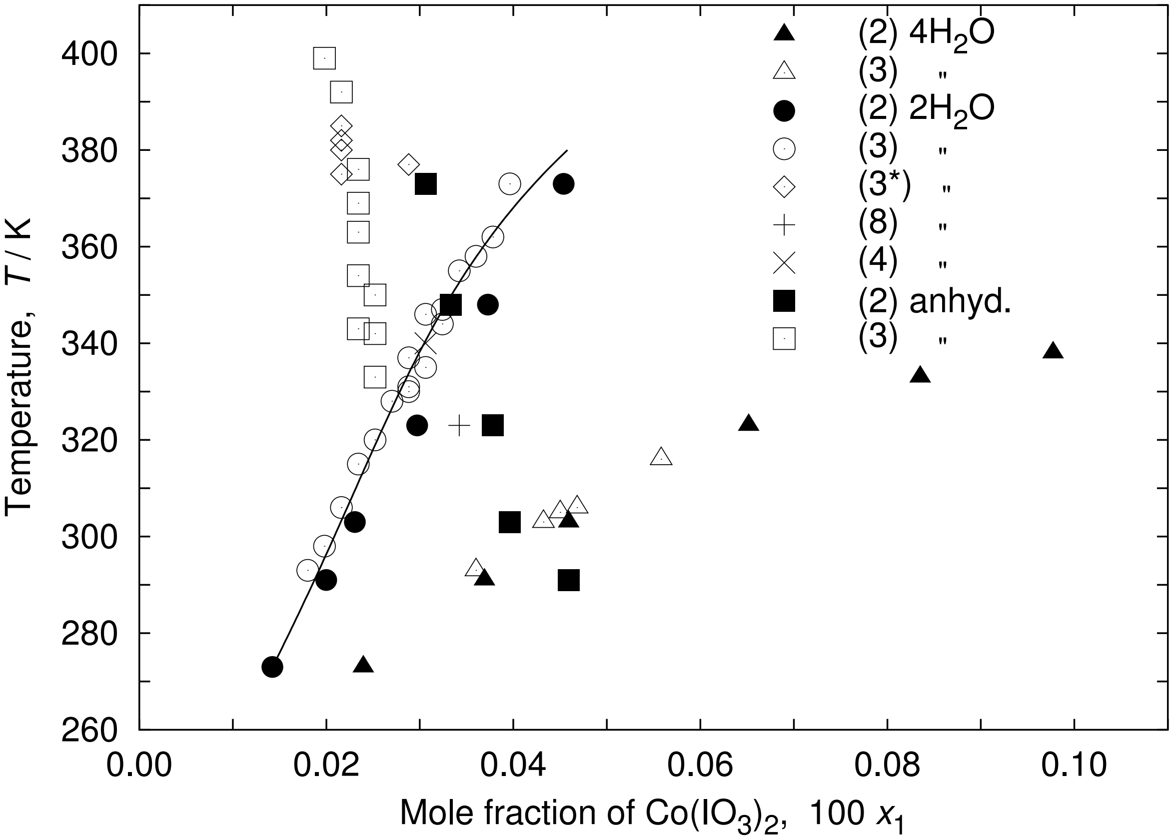

FIG. 4. Solubility of cobalt(II) iodate hydrates in water (The dihydrate is a cryltalline form, and the tetrahydrate and the anhydrate are metastable. *: rejected data, see footnote of Table 15)

The Multicomponent System

Trimble (4) measured the solubilities of cobalt iodate in the presence of $CoSO_4$, $NaIO_3$ or NaCl at 303 K. In the case of $CoSO_4$ or $NaIO_3$, the solubility decreased monotonically with an increase of the concentration of added salt and it increased with an increase of the NaCl concentration.

The solubility of cobalt iodate in lithium nitrate and perchlorate solutions has been reported by Fedorov et al. (5). Lithium nitrate and perchlorate were used to adjust the ionic strength. The results on the solubility have been used to determine the composition and stability constants of the ionic complexes $\mathrm{Co(NO_3)_n^{2-n}}$ when n is 1 or 2.

The solubility of $\mathrm{Co(IO_3)_2}$ in tetrahydrofuran–water mixtures at 298 K has been measured by Miyamoto (7). The solubility decreased with an increase of the tetrahydrofuran concentration. Maneva and Koleva (14) determined the solubility of $\mathrm{Co(IO_3)_2}$ in methanol, ethanol and acetone with 0.5 % water content at 293 K. The solubility values measured in these lyophilic organic solvents are about one-thousandth of that in water.

Lepeshkov et al. (8) have reported the solubility data for the ternary $\mathrm{Co(IO_3)_2}$–$\mathrm{KIO_3}$–$\mathrm{H_2O}$ system. An incongruently soluble compound, $\mathrm{2KIO_3 \cdot Co(IO_3)_2 \cdot 2H_2O}$ was formed. The detailed compilation was presented previously in (15).

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15. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 400.

Components:
(1) Cobalt iodate; $\mathrm{Co(IO_3)_2}$; [13455-28-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
A. Meusser, Ber. Dtsch. Chem. Ges. **34**, 2432–42 (1901).

Variables:
Temperature: 273–373 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 17. Experimental solubility of various forms of $\mathrm{Co(IO_3)_2}$ in water at various temperatures

Equilibration time / h	t / °C	$100w_1$	$100x_1$ [a]	10^2m_1 / mol kg^{-1} [a]
		Solid phase: $\mathrm{Co(IO_3)_2 \cdot 4H_2O}$		
42	0	0.54	0.024	1.33
42	18	0.83	0.037	2.05
2	30	1.03	0.046	2.55
1	50	1.46	0.065	3.62
0.167	60	1.86	0.083	4.64
0.1	65	2.17	0.098	5.43
		Solid phase: $\mathrm{Co(IO_3)_2 \cdot 2H_2O}$		
42	0	0.32	0.014	0.79
42	18	0.45	0.019	1.10
28	30	0.52	0.023	1.28
28	50	0.67	0.030	1.65
5	75	0.84	0.037	2.07
5	100	1.02	0.045	2.52
		Solid phase: $\mathrm{Co(IO_3)_2}$		
48	18	1.03	0.046	2.55
48	30	0.89	0.040	2.20
5	50	0.85	0.038	2.10
5	75	0.75	0.033	1.85
5	100	0.69	0.031	1.70

[a] Calculated by the compilers.

Auxiliary Information

Method/Apparatus/Procedure:

The saturated solution was equilibrated with cobalt iodate for a long time shaking. The compiler assumed that the solubility of cobalt iodate was determined iodometrically.

Source and Purity of Materials:

Three methods were applied to prepared cobalt iodate tetrahydrate: (1) Freshly prepared cobalt carbonate was dissolved in iodic acid solution, (2) Solutions of sodium iodate and cobalt nitrate were mixed. (3) Equimolar amounts of 5 % iodic acid and cobalt nitrate solutions were mixed. Crystallization was performed at 0–10 °C.

The theoretical contents of the elements calculated from $\mathrm{Co(IO_3)_2 \cdot 4H_2O}$ formula were in good agreement with the results found. When crystallization was performed at 30 °C, the dihydrate salt was obtained. When crystallization was carried out at 120 °C, the anhydrous salt was prepared.

Estimated Error:

Nothing specified.

Components:
(1) Cobalt iodate; $\mathrm{Co(IO_3)_2}$; [13455-28-2]
(2) Cobalt sulfate; $CoSO_4$; [10124-43-3]
or (2) Sodium chloride; NaCl; [7647-14-5]
or (2) Sodium iodate; $NaIO_3$; [7681-55-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. M. Trimble, J. Am. Chem. Soc. **58**, 1868–9 (1936).

Variables:
Concentration of added salts
One temperature: 303 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 18. Solubility of $\mathrm{Co(IO_3)_2}$ in aqueous $CoSO_4$, NaCl and $NaIO_3$ solutions at 30 °C

Added salt	m_2 / mol kg^{-1}	c_1 / mol kg^{-1}	ρ / g cm^{-3}
None	—	0.01165	1.0006
NaCl	0.0029	0.0119	1.0007
	0.0052	0.0120	1.0008
	0.0069	0.0121	1.0009
	0.0111	0.0124	1.0010
	0.0140	0.0125	1.0011
	0.0267	0.0131	1.0016
	0.0355	0.0134	1.0020
	0.0532	0.0139	1.0029
	0.0941	0.0149	1.0054
	0.1449	0.0158	1.0085
$NaIO_3$	0.0044	0.0102	1.0006
	0.0076	0.0093	1.0007
	0.0087	0.0090	1.0008
	0.0127	0.0080	1.0011
	0.0174	0.0069	1.0014
	0.0202	0.0063	1.0017
	0.0280	0.0050	1.0026
	0.0360	0.0040	1.0037
	0.0483	0.0030	1.0056
	0.0999	0.0010	1.0135
$CoSO_4$	0.0026	0.0113	1.0008
	0.0052	0.0111	1.0011
	0.0061	0.0111	1.0012
	0.0123	0.0107	1.0019
	0.0129	0.0106	1.0020

0.0235	0.0102	1.0034
0.0258	0.0101	1.0037
0.0516	0.0095	1.0076
0.0774	0.0094	1.0117
0.1032	0.0093	1.0155

The equilibrium solid phase was not identified but the author specified it as $Co(IO_3)_2 \cdot 2H_2O$.

Auxiliary Information

Method/Apparatus/Procedure:

The undersaturation method was used. Solutions of sodium chloride, sodium iodate, and cobalt(II) sulfate were prepared in suitable concentrations and analyzed. Their densities were also determined. Chloride concentration in the sodium chloride solution were determined by the Volhard method. Sulfate concentration of the cobalt sulfate solutions was determined by the usual gravimetric method. The cobalt sulfate solution was agitated with crystals of cobalt iodate dihydrate. Equilibrium was established for 75–120 h at the selected temperature. After saturation, excess of cobalt iodate was filtered off, using a filter paper designated for the filtration of very fine precipitates. Cobalt was precipitated from the solutions as hydroxide with sodium hydroxide. After filtering and washing this precipitate, it was dissolved in HCl and the cobalt was determined by the perborate method. The cobalt iodate concentration in the saturated solution was calculated from this determination. The total iodate content in the saturated solutions of cobalt iodate containing sodium iodate was determined by iodometric titration with a standard thiosulfate solution. The sodium iodate content was found by difference. Other samples of the solution were treated with an excess of hydrochloric acid and boiled to destroy the iodate present and to remove the chlorine liberated by the reaction. Carefully calibrated apparatus was used. The preparation of the solutions and their analyses were carried out in duplicate or triplicate. The densities of the saturated solutions were determined.

Source and Purity of Materials:

Cobalt iodate dihydrate was prepared by the reaction of “the best obtainable” cobalt nitrate and sodium iodate. Upon agitating the mother liquor for some days, conversion to dihydrate was complete. The product was washed thoroughly and dried to constant weight at 50 °C. Repeated analyses established its exact composition and its purity. The commercial “chemically pure” grade cobalt sulfate was recrystallized before use. Sodium chloride was a special product marked “for analysis” and was not further purified. Sodium iodate was prepared in the author’s laboratory and several times recrystallized, but the starting materials for preparation were not reported. Redistilled water was used in this study.

Estimated Error:

Nothing specified.

Components:

(1) Cobalt iodate; $\mathrm{Co(IO_3)_2}$; [13455-28-2]
(2) Tetrahydrofuran; $\mathrm{C_4H_8O}$; [109-99-9]
(3) Water; $\mathrm{H_2O}$; [7732-18-5]

Original Measurements:

H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659–61.

Variables:

Concentration of tetrahydrofuran: 0–40%
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 19. Solubility of $\mathrm{Co(IO_3)_3}$ in tetrahydrofuran–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	11.59
5	1.3	9.43
10	2.7	7.32
15	4.2	5.66
20	5.9	4.33
25	7.7	3.32
30	9.7	2.57
40	14.3	1.69

Auxiliary Information

Method/Apparatus/Procedure:

An excess of cobalt iodate crystals and the solvent mixtures were placed into glass-stopper bottles. The bottles were rotated in a thermostat at the selected temperature for 48 h. After the saturated solutions were allowed to settle, the solutions were withdrawn through a siphon tube equipped with a sintered-grass filter. The iodate content in the saturated solutions was determined iodometrically.

Source and Purity of Materials:

Cobalt iodate dihydrate was prepared by dropping solutions of equivalent quantities of cobalt nitrate (Wako guaranteed reagent) in a large volume of aqueous potassium nitrate solution. The precipitate was filtered, washed, and then dried under reduced pressure. Tetrahydrofuran was distilled over sodium hydroxide and then redistilled over sodium metal.

Estimated Error:

Solubility: precision no better than ± 0.05 %.
Temperature: precision ± 0.02 K.

Components:
(1) Cobalt iodate; $Co(IO_3)_2$; [13455-28-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. H. P. Cordfunke, J. Inorg. Nucl. Chem. **35**, 2699–705 (1973).

Variables:
Temperature: 293–399 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of the crystalline tetrahydrate, $Co(IO_3)_2 \cdot 4H_2O$, at 20 °C is given :

0.8 g $Co(IO_3)_2$ in 100 g H_2O (author) and 2.0×10^{-3} mol kg^{-1} (compilers)

and the solubility of the dihydrate, $Co(IO_3)_2 \cdot 2H_2O$ at 20 °C is given:

0.4 g $Co(IO_3)_2$ in 100 g H_2O (author) and 1.0×10^{-3} mol kg^{-1} (compilers).

Both values are from an extrapolation by the author. The solubilities at the other temperatures were given in a figure. The data were read from the figure by the compilers. The solubility values are given in Table 20.

TABLE 20. Solubility of various forms of $Co(IO_3)_2$ in water at various temperatures

Solute	t / °C	Mass ratio^{a}	$10^2 m_1$ / mol kg^{-1} ^{b}	$100x_1$ ^{b}
$Co(IO_3)_2 \cdot 4H_2O$	30	0.99; 1.00 (1.00)^{f}	2.4^{f}	0.044
	32	1.03	2.5	0.045
	33	1.05	2.6	0.046
	43	1.25	3.1	0.055
$Co(IO_3)_2 \cdot 2H_2O^{c}$	25	0.43	1.1	0.019
	33	0.48	1.2	0.021
	47	0.58	1.4	0.026
	57	0.65	1.6	0.029
	62	0.70	1.7	0.031
	64	0.65	1.6	0.029
	67	0.68	1.7	0.030
	71	0.70	1.7	0.031
	73	0.69	1.7	0.030
$Co(IO_3)_2 \cdot 2H_2O^{d}$	42	0.55	1.3	0.024
	55	0.62	1.5	0.027
	58	0.64	1.6	0.028
	71	0.73	1.8	0.032
	74	0.75	1.8	0.030
	82	0.79	1.9	0.035
	85	0.81	2.0	0.054

	89	0.85	2.1	0.037
	100	0.91	2.2	0.040
	102	0.51	1.2	0.022
	104[e]	0.66[e]	1.6	0.029
	107	0.50	1.2	0.022
	109	0.48	1.2	0.021
	112	0.49	1.2	0.022
$Co(IO_3)_2$	60	0.57	1.4	0.025
	69	0.56	1.4	0.025
	70	0.55	1.3	0.024
	77	0.57	1.4	0.025
	81	0.55	1.3	0.024
	90	0.56; 0.52 (0.54[f])	1.3[f]	0.024
	96	0.53	1.3	0.023
	103	0.53; 0.51 (0.52[f])	1.3[f]	0.023
	119	0.47	1.2	0.021
	126	0.46	1.1	0.020

[a] Mass ratio is reported as the amount (gram units) of $Co(IO_3)_2$ per 100 g of water in the original paper.

[b] Calculated by the compilers.

[c] Disappearance method.

[d] Analysis of the saturated solution.

[e] A metastable equilibrium.

[f] Average value.

The tetrahydrate is a metastable form transforming into dihydrate in a slow process. Anhydrous salt does not transform into dihydrate upon cooling below the transition temperature 102–104 $^{\circ}\mathrm{C}$.

The heat of solution of $\mathrm{Co(IO_3)_2 \cdot 6H_2O}$, $\mathrm{Co(IO_3)_2 \cdot 4H_2O}$, α-$\mathrm{Co(IO_3)_2 \cdot 2H_2O}$, β-$\mathrm{Co(IO_3)_2 \cdot 2H_2O}$ and $\mathrm{Co(IO_3)_2}$ in 6 mol dm^{-3} HNO_3 were determined: -7.46 ± 0.002, -6.87 ± 0.03, -3.50 ± 0.02, -1.15 and $+2.10 \pm 0.02$ kcal mol^{-1}, respectively.

Auxiliary Information

Method/Apparatus/Procedure:

Two different types of solubility measurements were employed: (1) the method in which the temperature is observed at which a certain amount of the hydrate has been solved in a given amount of water (disappearance method), (2) the method in which the iodate concentration is determined in the saturated solution which is in equilibrium with the solid hydrate. The iodate content was determined acidimetrically by titration with 0.1 mol dm^{-3} HCl in iodide medium. A simple and accurate determination of cobalt was carried out by titration with 0.05 mol dm^{-3} EDTA solution at pH = 6 with hexamine buffer and xylenol orange as the indicator. Small amounts of cobalt were determined by titration with EDTA using murexide as the indicator. The equilibrium solid phases were analyzed by X-rays. The heats of solution were determined in a calorimeter.

Source and Purity of Materials:

Cobalt iodate was prepared by mixing solutions of KIO_3 (or HIO_3) and $\mathrm{Co(NO_3)_2 \cdot 6H_2O}$ in the stoichiometric ratio. The hydrates were obtained as crystals from supersaturated solutions at room temperature. From 0.125–0.075 mol dm^{-3} $\mathrm{Co(IO_3)_2}$ solution, a mixture of the tetrahydrate and the dihydrate is obtained. From $<$ 0.07 mol dm^{-3} solutions the pure tetrahydrate can be obtained. From only slightly suppersaturated solutions the stable dihydrate was obtained.

Estimated Error:

Solubility: precision better than ± 4 % (by the compilers; reading-out procedure).

Temperature: reading-out procedure ± 1 K.

Components:

(1) Cobalt iodate; $Co(IO_3)_2$; [13455-28-2]
(2) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(3) Lithium nitrate; $LiNO_3$; [7790-69-4]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Fedorov, I. I. Shmyd'ko, A. M. Robov, L. S. Simaeva, V. A. Kukhtina, and V. E. Mironov, Zh. Neorg. Khim. **18**, 1274–9 (1973); Russ. J. Inorg. Chem. (Engl. Transl.) **18**, 673–5 (1973).

Variables:

Composition of $LiNO_3$ and $LiClO_4$
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 21. Solubility of $Co(IO_3)_2$ in water containing $LiNO_3$ and $LiClO_4$ at different ionic strength at 25 °C

I / mol dm^{-3}	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$10^2 c_1$ / mol dm^{-3}
0.5	0	0.5	1.62
	0.1	0.4	1.64
	0.2	0.3	1.66
	0.3	0.2	1.68
	0.4	0.1	1.73
	0.5	0	1.77
1.0	0	1.0	1.69
	0.2	0.8	1.73
	0.4	0.6	1.83
	0.6	0.4	1.87
	0.8	0.2	1.95
	1.0	0	2.01
2.0	0	2.0	1.61
	0.2	1.8	1.65
	0.5	1.5	1.73
	0.8	1.2	1.82
	1.0	1.0	1.88
	1.3	0.7	1.98
	1.5	0.5	2.05
	1.8	0.2	2.16
	2.0	0	2.24
3.0	0	3.0	1.40
	0.3	2.7	1.45
	0.6	2.4	1.49

	1.0	2.0	1.59
	1.3	1.7	1.68
	1.6	1.4	1.78
	2.0	1.0	1.91
	2.3	0.7	1.95
	2.6	0.4	2.06
	3.0	0	2.20
4.0	0	4.0	1.03
	0.4	3.6	1.09
	0.8	3.2	1.21
	1.2	2.8	1.30
	1.6	2.4	1.43
	2.0	2.0	1.52
	2.4	1.6	1.63
	2.8	1.2	1.75
	3.2	0.8	1.89
	3.6	0.4	2.03
	4.0	0	2.13

TABLE 22. Solubility product of $\mathrm{Co(IO_3)_2}$ at various ionic strength at 25 °C

I / mol dm^{-3}	$10^5 K_\mathrm{s}$ / mol^3 dm^{-9}
0	0.229 ± 0.015
0.5	1.70
1.0	1.93
2.0	1.66
3.0	1.09
4.0	0.436

The listed solubility products of cobalt iodate were calculated from $K_\mathrm{s} = 4c_1^3$, where s is the solubility of $\mathrm{Co(IO_3)_2}$ in lithium perchlorate solutions at various ionic strength. The stability constants of $\mathrm{Co(NO_3)_n^{2-n}}$ were also reported to calculate the solubility of cobalt iodate in various $LiNO_3$–$LiClO_4$ solutions.

The equilibrium solid phase was found to be $\mathrm{Co(IO_3)_2 \cdot 2H_2O}$.

Auxiliary Information

Method/Apparatus/Procedure:

Equilibrium in the system was reached by vigorous agitation of solutions for 5–6 h, and checked by sampling after various intervals of time. The concentration of cobalt iodate in the saturated solutions was determined iodometrically with an amperometric indication of the equivalent point. The solid equilibrium phase was analyzed: Co–13.1; IO_3–78.4; H_2O–8.5 mass%, Calculated for $\mathrm{Co(IO_3)_2 \cdot 2H_2O}$: Co–13.2; IO_3–78.3; H_2O–8.5 mass %.

Source and Purity of Materials:

No information was given.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: nothing specified.

Components:

(1) Cobalt iodate; $\mathrm{Co(IO_3)_2}$; [13455-28-2]
(2) Lithium chloride; LiCl; [7447-41-8]
or (2) Lithium bromide; LiBr; [7550-35-8]
or (2) Lithium sulfate; Li_2SO_4; [10377-48-7]
or (2) Lithium nitrate; $LiNO_3$; [7790-69-4]
or (2) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Fedorov, A. M. Robov, I. I. Shmyd'ko, T. N. Koneva, L. S. Simaeva, V. A. Kukhtina, Zh. Fiz. Khim. **50**, 2213–5 (1976); Russ. J. Phys. Chem. (Engl. Transl.) **50**, 1330–2 (1976).

Variables:

Concentration of lithium salts: 0–4.0 mol dm^{-3}
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 23. Solubility of $\mathrm{Co(IO_3)_2}$ in aqueous lithium salt solutions at 25 °C

Lithium salt	c_2 / mol dm^{-3}	$10^2 c_1$ / mol dm^{-3}
None	—	1.13
LiCl	0.5	1.89
	1.0	2.09
	2.0	2.20
	3.0	2.14
	4.0	2.09
LiBr	0.5	1.92
	1.0	2.05
	2.0	2.10
	3.0	2.15
	4.0	1.57
Li_2SO_4	0.25	2.56
	0.50	3.37
	1.0	4.02
	1.5	4.24
	2.0	3.78
$LiClO_4$	0.01	1.21
	0.05	1.27
	0.10	1.33
	0.25	1.48
	0.50	1.62
	1.00	1.69
	2.00	1.61

	3.00	1.40
	4.00	1.03
$LiNO_3$	0.01	1.17
	0.05	1.25
	0.10	1.32
	0.25	1.59
	0.40	1.70
	0.50	1.76
	1.00	2.01
	2.00	2.19
	3.00	2.20
	4.00	2.13

The authors stated that the solid phase is $\mathrm{Co(IO_3)_2 \cdot 2H_2O}$.

$\mathrm{p}K_\mathrm{s}^\circ$ values of $\mathrm{Co(IO_3)_2}$ found after extrapolation to the zero ionic strength at 298 K are: 5.75 (in LiCl), 6.12 (in $\mathrm{Li_2SO_4}$), 5.70 (in LiBr). 5.81 (in $\mathrm{LiNO_3}$), 5.81 (in $\mathrm{LiClO_4}$), and 5.77 (in pure $\mathrm{H_2O}$).

Auxiliary Information

Method/Apparatus/Procedure:

The experimental method used was similar to that of Fedorov et al. (1). Equilibration required 4–6 h of intensive mixing of the solution and salts. The solubilities were determined by iodometric titration with amperometric end point detection. Each solubility value given in the table above is the mean of 3–4 determinations.

Source and Purity of Materials:

The source and purity of materials were not given.

Estimated Error:

Solubility: reproducibility within 2-3%.

Temperature: nothing specified.

References:

[1] V. A. Fedorov, I. I. Shmyd'ko, A. M. Robov, L. S. Simaeva, V. A. Kukhtina, and V. E. Mironov, Zh. Neorg. Khim. **18**, 1274 (1973); Russ. J. Inorg. Chem. (Engl. Transl.) **18**, 673 (1973).

Components:
(1) Cobalt iodate; $Co(IO_3)_2$; [13455-28-2]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
or (2) 2-Propanone (acetone); C_3H_6O; [67-64-1]

Original Measurements:
M. Maneva and V. Koleva, God. Vissh. Khimikotekhnol. Inst., Sofia, **31** (3), 111–25 (1992, Pub. 1993).

Variables:
Organic solvents
One temperature: 293 K

Prepared By:
Hiroshi Miyamoto, Christo Balarew, and Cezary Guminski

Experimental Data

TABLE 24. Solubility of $Co(IO_3)_2$ in various organic solvents at 20 °C

Organic solvent	c_1 / mol dm^{-3}
Methanol	2×10^{-5}
Ethanol	1×10^{-5}
2-Propanone	1×10^{-5}

$Co(IO_3)_2 \cdot 4H_2O$ is the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:
Saturated solutions were prepared by mixing the salt with the corresponding solvents at the selected temperature for up to 12 h. The resulting solutions were filtered at this temperature in a glass apparatus. The cobalt content was determined by atomic absorption spectroscopy. The equilibrium solid phase was determined by X-ray diffraction and analyzed for the contents of Co^{2+}, IO_3^- and H_2O.

Source and Purity of Materials:
$Co(IO_3)_2 \cdot 4H_2O$ was obtained by mixing cobalt nitrate and iodic acid (both pure for analysis) aqueous solutions in stoichiometric ratio at “proper”, but unspecified temperature. The crystals were washed with water to absence of NO_3^- ion in the solution and dried in air. Compositions of the product were analyzed: Co^{2+} — by complexometric titration, IO_3^- — by iodometric titration and H_2O — by the Fischer’s method and thermogravimetry. Distilled water was used. Methanol, ethanol and acetone were dehydrated with molecular sieves (porosity of 300 pm) and distilled; the final water content was maximally 0.5 %.

Estimated Error:
Solubility: nothing specified.
Temperature: precision ± 0.1 K.

E. Nickel Iodate

Components:
(1) Nickel iodate; $Ni(IO_3)_2$; [13477-98-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. March 2002.

Critical Evaluation:

The Binary System

Earliest studies on the preparation and the solubility of nickel iodate have been summarized by Mellor (1). Because these articles are very old the evaluators could not get them except the Meusser's paper (2). Data for the solubility of nickel iodate in water have been reported in 7 publications (2–8). Meusser (2) reported the solubility in water in mass % over a wide range of temperatures (273–363 K). The article has been the most comprehensive source of knowledge on nickel iodate in the early years. Cordfunke (3) also studied the solubility for the binary $Ni(IO_3)_2$–H_2O system as a function of temperature (293–334 K). Any numerical data have not been reported in his article except the value at 293 K. The results for both crystalline and amorphous forms of the nickel iodate dihydrate as a function of temperature were shown in a figure. Therefore, the corresponding data points of Cordfunke were read out by the evaluators. The paper by Lepeshkov et al. (4) was concerned on isothermal studies of a ternary system at 323 K which included data for the binary $Ni(IO_3)_2$–H_2O system. Miyamoto's groups (5, 6) have reported the molarity solubilities of nickel iodate in aqueous–organic solvent mixtures at 298 K which includes data in pure water. Fedorov's groups (7, 8) have reported the molarity solubilities of nickel iodate in aqueous electrolyte solutions and obtained the solubility product of nickel iodate at various ionic strength at 298 K. All experimental solubility data including the doubtful data are tabulated and shown in the figure.

TABLE 25. Summary for solubility studies of $Ni(IO_3)_2$ in water

T / K	10^2c_1 / mol dm^{-3}	10^2m_1 / mol kg^{-1} [a]	$100x_1$ [a]	Analytical method	Reference
		Solid phase: $Ni(IO_3)_2 \cdot 4H_2O$ (intermediate metastable)			
273	—	1.80	0.033	Iodometry	(2)
291	—	2.50	0.045	Iodometry	(2)
303	—	3.50	0.063	Iodometry	(2)
		Solid phase: $Ni(IO_3)_2 \cdot 3H_2O$ (a mixture of di- and tetahydrates)			
298	1.14[c]	—	—	Iodometry	(8)
298	1.82	—	—	Iodometry	(8)
		Solid phase: $Ni(IO_3)_2 \cdot 2H_2O$ (amorphous)			
273	—	1.30	0.024	Iodometry	(2)
291	—	1.68	0.030	Iodometry	(2)
293	—	1.74	0.031	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)

303	—	2.12	0.038	Iodometry	(2)
323	—	4.44	0.080	Iodometry	(2)
Solid phase: $Ni(IO_3)_2 \cdot 2H_2O$ (crystalline)					
281	—	1.28	0.023	Iodometry	(2)
291	—	1.35	0.024	Iodometry	(2)
293	1.54	—	—	Iodometry	(6)
298	1.62	—	—	Iodometry	(6)
298	1.65	—	—	Iodometry	(5)
302	—	1.54[b]	0.028[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
303	1.70	—	—	Iodometry	(6)
306	—	1.66[b]	0.030[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
313	—	1.71[b]	0.031[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
318	—	1.84[b]	0.033[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
319	—	1.91[b]	0.034[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
323	—	1.93[b]	0.035[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
323	—	2.00	0.036	Iodometry	(2)
323	—	2.04	0.037	Complexometry	(4)
326	—	1.98[b]	0.036[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
328	—	2.03[b]	0.037[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
333	—	2.13[b]	0.038[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
334	—	2.11[b]	0.038[b]	Acidimetry (IO_3^-), Gravimetry (Ni^{2+})	(3)
348	—	2.55	0.046	Iodometry	(2)
353	—	2.77	0.050	Iodometry	(2)
Solid phase: $Ni(IO_3)_2$ (doubtful data)					
303	—	2.81	0.051	Iodometry	(2)
323	—	2.65	0.048	Iodometry	(2)
348	—	2.52	0.045	Iodometry	(2)
363	—	2.44	0.044	Iodometry	(2)

[a] Calculated by the evaluators

[b] Read out from the figure and calculated by the evaluators

[c] Calculated from solubility product at $I = 0$ by the evaluators

The following equilibrium solid phases have been reported in these solubility studies:

$Ni(IO_3)_2$	[13477-98-0]	(2)
$Ni(IO_3)_2 \cdot 2H_2O$	[24651-06-7]	(2), (3), (4), (5), (6), (12)
$Ni(IO_3)_2 \cdot 3H_2O$	[42910-38-3]	(7), (8)
$Ni(IO_3)_2 \cdot 4H_2O$	[13477-99-1]	(2)

Cordfunke (4) examined the preparation of the hydrated nickel iodates, redetermined both the solubilities in water and the composition of equilibrium solid phases. Cordfunke stated that only the solubility of $Ni(IO_3)_2 \cdot 2H_2O$ corresponds to real equilibrium since it was not possible to obtain the tetrahydrate in a pure form. The results of his measurements were in reasonable agreement with Meusser's data for the "β-dihydrate". As can be expected the solubility of the amorphous form of the dihydrate was much higher than that of the crystalline dihydrate. The solubility of the amorphous form at 293 K agrees well with the data given by Meusser (2) for the α-dihydrate. Cordfunke concluded that both substances are identical and only one crystalline dihydrate of nickel iodate exists.

Nassau's group (10) has reviewed the solubility studies on nickel iodate and stated (from the solubility study by Meusser (2)) that in the presence of water, α-$Ni(IO_3)_2 \cdot 2H_2O$ is stable below 75 °C, β-$Ni(IO_3)_2 \cdot 2H_2O$ above 75 °C, and $Ni(IO_3)_2 \cdot 4H_2O$ was unstable at all temperatures. Nassau's group has studied in details the characterization of nickel iodate anhydride and hydrates by differential thermal and thermogravimetric analyses and optical absorption spectroscopy, although a solubility study was not performed. Abrahams et al. (11) have studied the crystallographic, magnetic and nonlinear optical properties of the nickel iodate anhydride and hydrates. Existence of α-$Ni(IO_3)_2 \cdot 4H_2O$ (green), β-$Ni(IO_3)_2 \cdot 4H_2O$ (medium-green), $Ni(IO_3)_2 \cdot 2H_2O$ (emerald green), and β-$Ni(IO_3)_2$ (yellow) in the presence of water or nitric acid solution was established in both studies (10, 11). Anhydrous α-$Ni(IO_3)_2$ (green) was only obtained in the absence of water. Nassau et al. (10) reported that a fluffy precipitate, α-$Ni(IO_3)_2 \cdot 4H_2O$, formed within a few hours at room temperature by a precipitation of $Ni(NO_3)_2$ solution with HIO_3 solution. The α-tetrahydrate was the first compound to precipitate from the solution; it was rapidly converted to β-$Ni(IO_3)_2 \cdot 4H_2O$ in the solution or in a contact of the salt with water. The appearance of the β-tetrahydrate is an intermediate metastable phase on the way to stability. In the presence of water it was further slowly converted into $Ni(IO_3)_2 \cdot 2H_2O$. In the presence of a small amount of water, a powdered mixture of all three hydrates, α-$Ni(IO_3)_2 \cdot 4H_2O$, β-$Ni(IO_3)_2 \cdot 4H_2O$ and $Ni(IO_3)_2 \cdot 2H_2O$, converts rapidly to the dihydrate. Nassau et al. concluded that any solubility data depend on the length of equilibration time used as well as on presence of the salt nuclei. The group of Nassau's (10, 11) identified only one crystalline form of dihydrate and found Messuer's α-dihydrate (2) to be in fact the β-tetrahydrate which is unstable in the presence of water. Therefore the solubility data of the α-dihydrate reported by Messuer are unreliable because of the appearance of the metastable state as the equilibrium solid phase.

Lepeshkov et al. (4) measured the solubility of nickel iodate in equilibrium with $Ni(IO_3)_2 \cdot 2H_2O$ as the solid phase at 323 K. The composition of equilibrium solid phase was characterized by thermal, thermogravimetric, X-ray diffraction and infrared spectroscopic methods. The authors have identified only one dihydrate and have not reported other hydrate salts.

Fedorov's groups (7, 8) reported the solubility of nickel iodate with $Ni(IO_3)_2 \cdot 3H_2O$ as the equilibrium solid phase. They determined the composition of this phase by chemical analysis and stated that it is the most thermodynamically stable hydrate. Messuer (2) was unable to prepare the trihydrate and stated that the trihydrate salt was probably a mixture of the dihydrated and tetrahydrate salt. Neither Cordfunke or Nassau's groups have reported the existence of $\mathrm{Ni(IO_3)_2 \cdot 3H_2O}$. Maneva and Koleva (13) discussed the role of water in the α-$\mathrm{Ni(IO_3)_2 \cdot 4H_2O}$ on its solubility in a medium of lyophilic solvents.

As a result of the above described observations, the evaluators fitted the observed solubility data of $\mathrm{Ni(IO_3)_2 \cdot 2H_2O}$ (crystalline) based on mole fraction to the smoothing equation described below. The detailed information of the calculation procedure for the smoothing equation has been described in the Preface to this volume. The solubility values calculated from the smoothing equation are tabulated in Table 26; these values are designated as recommended values.

$$Y_x = \frac{-10385.6594}{(T/\mathrm{K})} - 73.2126304 \ln{(T/\mathrm{K})} + 386.469077 + 0.159339785(T/\mathrm{K}), \qquad (37)$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^2(3+2)^{3+2}}{2^2(1+x)^{3+2}}\right). \qquad (38)$$

As the results of the calculation, $\sigma_Y = 0.080$ and $\sigma_x = 9.3 \times 10^{-6}$ were obtained.

TABLE 26. Recommended solubility of $\mathrm{Ni(IO_3)_2}$ (solid phase: dihydrate, crystalline) in water

T / K	$100x_1$	10^2m_1 / mol kg^{-1}
281	0.023	1.26
283	0.023	1.29
293	0.026	1.42
298	0.027	1.50
303	0.028	1.57
313	0.031	1.75
323	0.035	1.94
333	0.039	2.17
343	0.044	2.42

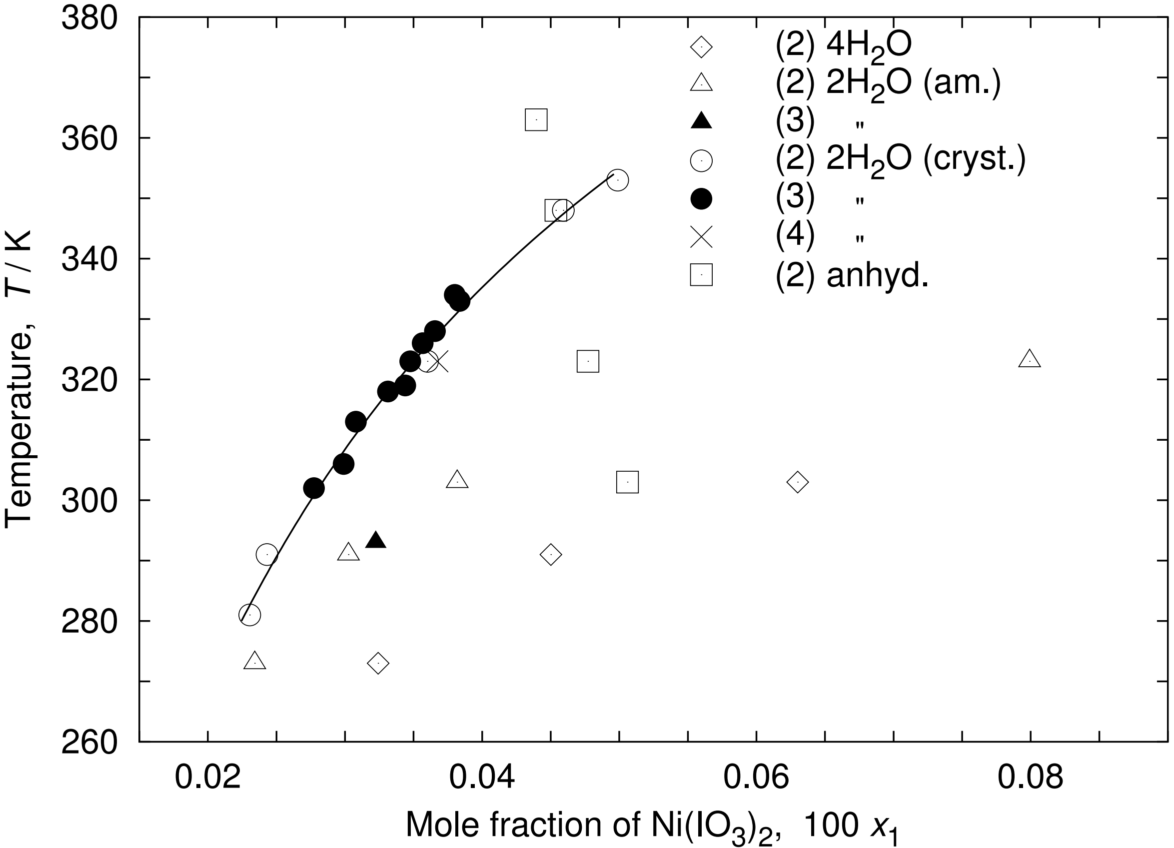

FIG. 5. Solubility of nickel(II) iodate hydrates in water (The tetrahydrate form is an intermediate metastable. The dihydrate indicated by (am.) is the amorphous form. Solubilities of anhydrate were doubtful (see text).)

The Multicomponent System

Miyamoto's group studied the solubilities in two aqueous-organic solvent mixtures: with tetrahydrofuran (5) and *N*,*N*-dimethylformamide (6). The solubility of nickel iodate decreased with an increase of the organic solvent concentration in both mixtures.

Maneva and Koleva (13) measured the solubility of nickel iodate tetrahydrate in lyophilic organic solvents such as methanol, ethanol and acetone with 0.5 % water content at 293 K. The solubility values in these solvents are about one-thousandth of that in water. The role of the organic solvent affinity toward water included in the crystal has been discussed as the possible course of the dehydration process.

Lepeshkov et al. (4) studied the solubility in the ternary $\mathrm{Ni(IO_3)_2}$–$\mathrm{KIO_3}$–$\mathrm{H_2O}$ system. The solubility isotherm at 323 K has crystallization branches of $\mathrm{KIO_3}$, $\mathrm{Ni(IO_3)_2 \cdot 2H_2O}$ and $\mathrm{K_2[Ni(IO_3)_4(H_2O)_2]}$ (dipotassium diaquatetraiodatonickelate). The reader finds the compilation of this system in (14). Fedorov et al. (7) measured the nickel iodate solubilities in aqueous solutions of lithium nitrate, chloride, bromide, sulfate and perchlorate at 298 K. The thermodynamic solubility product of $(5.9 \pm 0.3) \times 10^{-6}$ mol^3 dm^{-9} was calculated from these solubility data after extrapolation to zero ionic strength. The solubility value of 1.14×10^{-2} mol dm^{-3} recalculated by the evaluators is distinctly different from the data observed by the other investigators at 298

K because the activity coefficients were taken into account. Fedorov et al. (8) also measured the solubilities of nickel iodate in aqueous solutions containing lithium perchlorate and nitrate at 298 K. The ionic strength of the aqueous solution was adjusted at 0.5, 1.0, 2.0, 3.0, and 4.0. Results of the solubility were used to calculate the complexation stability constant of nickel and nitrate ions.

References:

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14. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, UK, 1987), p. 401.

Components:
(1) Nickel iodate; $\mathrm{Ni(IO_3)_2}$; [13477-98-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
A. Meusser, Ber. Dtsch. Chem. Ges. **34**, 2432–42 (1901).

Variables:
Temperature: 273–363 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 27. Experimental solubility of various forms of $\mathrm{Ni(IO_3)_2}$ in water at various temperatures

Equilibration time / h	t / °C	$100w_1$	$100x_1$[a]	10^2m_1 / mol kg^{-1} [a]
		Solid phase: $\mathrm{Ni(IO_3)_2\cdot 4H_2O}$		
20	0	0.73	0.033	1.86
12	18	1.01	0.045	2.50
0.25	20	1.41	0.063	3.50
		Solid phase: α-$\mathrm{Ni(IO_3)_2\cdot 2H_2O}$		
120	8	0.53	0.024	1.30
6	18	0.68	0.030	1.68
8	30	0.86	0.038	2.12
0.25	50	1.78	0.080	4.44
		Solid phase: β-$\mathrm{Ni(IO_3)_2\cdot 2H_2O}$		
24	8	0.52	0.024	1.28
3	18	0.55	0.024	1.35
6	50	0.81	0.036	2.00
2	75	1.03	0.046	2.55
0.167	80	1.12	0.050	2.77
		Solid phase: $\mathrm{Ni(IO_3)_2}$		
290	30	1.135	0.051	2.81
144	50	1.07	0.048	2.65
26	75	1.02	0.045	2.52
20	90	0.988	0.044	2.44

[a] Calculated by the compilers

Auxiliary Information

Method/Apparatus/Procedure:

The saturated solutions were equilibrated with nickel iodate for a long time shaking. The compilers assumed that the solubility of nickel iodate was determined iodometrically.

Source and Purity of Materials:

Nickel iodate tetrahydrate was prepared from dilute solutions of nickel nitrate and sodium iodate or of iodic acid and nickel nitrate. Crystallization was carried out at 0–10 $^\circ\mathrm{C}$. The analytical results of the products were in good agreement with those calculated from $\mathrm{Ni(IO_3)_2 \cdot 4H_2O}$ formula. When the crystallization was performed at $\sim$ 30, $\sim$ 50 and 120 $^\circ\mathrm{C}$ one obtained: α-$\mathrm{Ni(IO_3)_2 \cdot 2H_2O}$, β-$\mathrm{Ni(IO_3)_2 \cdot 2H_2O}$ and anhydrous $\mathrm{Ni(IO_3)_2}$, respectively.

Estimated Error:

Nothing specified.

Components:
(1) Nickel iodate; $Ni(IO_3)_2$; [13477-98-0]
(2) Tetrahydrofuran; C_4H_8O; [109-99-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, Nippon Kagaku Zasshi **1972**, 659–61.

Variables:
Concentration of tetrahydrofuran: 0–40 mass%
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 28. Solubility of $Ni(IO_3)_2$ in tetrahydrofuran–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	16.52
5	1.3	11.83
10	2.7	8.34
15	4.2	6.12
20	5.9	4.52
25	7.7	3.42
30	9.7	2.76
40	14.3	1.55

Auxiliary Information

Method/Apparatus/Procedure:
An excess of nickel iodate dihydrate crystals and the solvent mixtures were placed into glass-stoppered bottles. The bottles were rotated in a thermostat at the selected temperature for 48 h. After the saturated solutions were allowed to settle, they were withdrawn through a siphon tube equipped with a sintered-glass filter. The iodate content in the saturated solutions was determined iodometrically.

Source and Purity of Materials:
$Ni(IO_3)_2 \cdot 2H_2O$ was prepared by dropping solutions of equivalent quantities of nickel nitrate (Wako guaranteed reagent) and potassium iodate (Wako guaranteed reagent) in a large volume of aqueous potassium nitrate solution. The precipitate was filtered, washed and then dried under reduced pressure. Tetrahydrofuran was distilled over sodium hydroxide and then redistilled over sodium metal.

Estimated Error:
Solubility: precision between ± 3 % and ± 0.3 % (compiler).
Temperature: precision ± 0.02 K.

Components:
(1) Nickel iodate; $Ni(IO_3)_2$; [13477-98-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. H. P. Cordfunke, J. Inorg. Nucl. Chem. **35**, 2699–2705 (1973).

Variables:
Temperature: 293–334 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

The solubility of the crystalline dihydrate, $Ni(IO_3)_2 \cdot 2H_2O$ at 20 °C was reported:

0.63 g $Ni(IO_3)_2$ / 100 g H_2O (author)
or 1.5×10^{-2} mol kg^{-1} (compilers).

The solubilities at the other temperatures were shown in a figure. The results were read from the figure by the compilers. The tetrahydrate is a metastable form slowly transforming into the dihydrate and it was impossible to measure the tetrahydrate solubility.

TABLE 29. Solubility of various forms of $Ni(IO_3)_2 \cdot 2H_2O$ in water at various temperatures

Solute	t / °C	Mass ratio[a]	$10^2 m_1$ / mol kg^{-1} [b]
Amorphous $Ni(IO_3)_2 \cdot 2H_2O$	20	0.71	1.7
Crystalline $Ni(IO_3)_2 \cdot 2H_2O$	20	0.63	1.5
	33	0.68	1.7
	40	0.70	1.7
	45	0.75	1.8
	46	0.78	1.9
	50	0.79	1.9
	53	0.81	2.0
	55	0.83	2.0
	60	0.87	2.1
	61	0.86	2.1

[a] Mass ratio is reported as the amount of $Ni(IO_3)_2$ g per 100 g of H_2O in the original paper.
[b] Calculated by the compilers

Auxiliary Information

Method/Apparatus/Procedure:

Two different types of solubility measurements were employed: (1) the method in which the temperature is observed at which a certain amount of the hydrate has been solved in a given amount of water (disappearance method). (2) the method in which the concentration of iodate is determined in the saturated solution which is an equilibrium with the solid hydrate.

The iodate concentration was determined acidimetrically by titration with 0.1 mol dm^{-3} HCl in iodine medium. The nickel content was determined gravimetrically after homogeneous precipitation of nickel(II) with dimethylglyoxime in 1-propanol and ageing the precipitate overnight on a water bath. The equilibrium solid phases were analyzed by X-rays.

Source and Purity of Materials:

Nickel iodate was prepared by mixing solutions of KIO_3 (or HIO_3) and $Ni(NO_3)_2 \cdot 6H_2O$ in the stoichiometric ratio, and the pure tetrahydrate crystals were obtained at times from 1 d to several weeks if the product was less concentrated than 0.111 mol dm^{-3}.

Estimated Error:

Solubility: precision better than ± 2 % (compilers); reading-out procedure ± 0.005 g / 100 g H_2O.

Temperature: reading-out procedure ± 1 K.

Components:

(1) Nickel iodate; $Ni(IO_3)_2$; [13477-98-0]
(2) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(3) Lithium nitrate; $LiNO_3$; [7790-69-4]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Fedorov, I. I. Shmyd'ko, A. M. Robov, L. S. Simaeva, V. A. Kukhtina, and V. E. Mironov, Zh. Neorg. Khim. **18**, 1274–9 (1973); Russ. J. Inorg. Chem. (Engl. Transl.) **18**, 673–5 (1973).

Variables:

Composition of $LiNO_3$ and $LiClO_4$
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 30. Solubility of $Ni(IO_3)_2$ in water containing $LiNO_3$ and $LiClO_4$ at 25 °C

I / mol dm^{-3}	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	10^2c_1 / mol dm^{-3}
0.5	0	0.5	2.57
	0.1	0.4	2.58
	0.2	0.3	2.59
	0.3	0.2	2.60
	0.4	0.1	2.61
	0.5	0	2.62
1.0	0	1.0	2.59
	0.2	0.8	2.69
	0.4	0.6	2.79
	0.6	0.4	2.88
	0.8	0.2	2.99
	1.0	0	3.09
2.0	0	2.0	2.41
	0.2	1.8	2.50
	0.5	1.5	2.62
	0.8	1.2	2.77
	1.0	1.0	2.83
	1.3	0.7	3.01
	1.5	0.5	3.12
	1.8	0.2	3.29
	2.0	0	3.44
3.0	0	3.0	2.12
	0.3	2.7	2.13
	0.6	2.4	2.25
	1.0	2.0	2.39

	1.3	1.7	2.48
	1.6	1.4	2.67
	2.0	1.0	2.69
	2.3	0.7	2.76
	2.6	0.4	2.88
	3.0	0	3.11
4.0	0	4.0	1.54
	0.4	3.6	1.66
	0.8	3.2	1.79
	1.2	2.8	1.54[a], 1.95[b]
	1.6	2.4	2.11
	2.0	2.0	2.28
	2.4	1.6	2.47
	2.8	1.2	2.66
	3.2	0.8	2.86
	3.6	0.4	3.08
	4.0	0	3.31

[a] Erroneously experimental value reported by the authors.

[b] Calculated value by the authors from the stability constants (β_1 and β_2) of nitrate-complexes and the solubility product of nickel iodate in $LiIO_3$ solution at I = 4.0 mol dm^{-3}. Detailed information on the calculation was reported in the original paper.

TABLE 31. Solubility product of $Ni(IO_3)_2$ at various ionic strength at 25 °C

I / mol dm^{-3}	$10^5 K_s$ / mol^3 dm^{-9}
0	0.870 ± 0.050
0.5	6.80
1.0	6.92
2.0	5.60
3.0	3.80
4.0	1.46

The listed solubility products of nickel iodate were calculated from $K_{\rm s} = 4c_1^3$, where c_1 is the solubility of $\rm Ni(IO_3)_2$ in lithium perchlorate solutions at various ionic strength. The stability constants of $\rm Ni(NO_3)_n^{2-n}$ were also reported to calculate the solubility of nickel iodate in various $LiNO_3$–$LiClO_4$ solutions

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of nickel iodate in $LiNO_3$–$LiClO_4$ solutions was measured at the selected temperature and ionic strength changed between 0.5 and 4.0. Equilibrium in the system was reached by vigorous agitation of the compound for 5–6 h, and was checked by sampling the solution after various intervals of time. The concentration of nickel iodate in the saturated solution was determined iodometrically with an amperometric indication of the equivalent point. The solid phase was analyzed: Ni–12.7; IO_3–75.7; H_2O–11.6 mass %, calculated contents: for $\rm Ni(IO_3)_2 \cdot 3H_2O$: Ni–12.7; IO_3–75.6; H_2O–11.7 mass %.

Source and Purity of Materials:

No information given.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: nothing specified.

Components:

(1) Nickel iodate; $Ni(IO_3)_2$; [13477-98-0]
(2) Lithium chloride; LiCl; [7447-41-8]
or (2) Lithium bromide; LiBr; [7550-35-8]
or (2) Lithium sulfate; Li_2SO_4; [10377-48-7]
or (2) Lithium nitrate; $LiNO_3$; [7790-69-4]
or (2) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Fedorov, A. M. Robov, I. I. Shmyd'ko, T. N. Koneva, L. S. Simaeva, and V. A. Kukhtina, Zh. Fiz. Khim. **50**, 2213–5 (1976); Russ. J. Phys. Chem. (Engl. Transl.) **50**, 1330–2 (1976).

Variables:

Concentration of lithium salts: 0–4.0 mol dm^{-3}
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 32. Solubility of $Ni(IO_3)_2$ in aqueous lithium salt solutions at 25 °C

Lithium salt	c_2 / mol dm^{-3}	10^2c_1 / mol dm^{-3}
None	—	1.82
LiCl	0.5	2.75
	1.0	3.00
	2.0	3.05
	3.0	3.09
	4.0	2.86
LiBr	0.5	2.72
	1.0	2.98
	2.0	3.08
	3.0	3.06
	4.0	2.87
Li_2SO_4	0.5	3.91
	1.0	4.86
	2.0	5.21
	3.0	5.73
	4.0	5.30
$LiNO_3$	0.01	1.91
	0.05	1.97
	0.10	2.17
	0.25	2.42
	0.40	2.66
	0.50	2.70
	1.00	3.09

	2.00	3.41
	3.00	3.39
	4.00	3.31
$LiClO_4$	0.01	2.03
	0.05	2.04
	0.10	2.13
	0.25	2.40
	0.50	2.57
	1.00	2.59
	2.00	2.41
	3.00	2.12
	4.00	1.54

pK_s° values found after extrapolation to the zero ionic strength at 298 K are: 5.22 (in LiCl), 5.24 (in LiBr), 5.24 (in Li_2SO_4), 5.24 (in $LiNO_3$), 5.20 (in $LiClO_4$), and 5.20 (in pure H_2O).

The authors stated that the solid phase is $\mathrm{Ni(IO_3)_2 \cdot 3H_2O}$ independently of the other salt composition.

The solubility results for the ionic strengths 0.5–4.0 were reported also in (1).

Auxiliary Information

Method/Apparatus/Procedure:

The experimental method used was similar to that of Fedorov et al. (1). Equilibrium required 4–6 h of intensive mixing of the solutions and salts. The solubilities were determined by iodometric titration of the iodate with amperometric end point detection. Each solubility value given in the table above is the mean of 3–4 determinations.

Source and Purity of Materials:

The source and purities of materials were not given.

Estimated Error:

Solubility: reproducibility within 2–3 %.

Temperature: nothing specified.

References:

[1] V. A. Fedorov, I. I. Shmyd'ko, A. M. Robov, L. S. Simaeva, V. A. Kukhtina, and V. E. Mironov, Zh. Neorg. Khim. **18**, 1274 (1973); Russ. J. Inorg. Chem. (Engl. Transl.) **18**, 673 (1973).

Components:

(1) Nickel iodate; $\mathrm{Ni(IO_3)_2}$; [13477-98-0]

(2) *N*,*N*-Dimethylformamide (DMF); $\mathrm{C_3H_7NO}$; [68-12-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, M. Yamamoto, and Y. Maruyama, Nippon Kagaku Kaishi **1979**, 546–8.

Variables:

Concentration of DMF: 0–41 mass %

Temperature: 293.15–303.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 33. Solubility of $\mathrm{Ni(IO_3)_2}$ in *N*,*N*-dimethylformamide–water mixtures at various temperatures

t / °C	$100w_2$	$100x_2$[a]	10^3c_1 / mol dm^{-3}
20	0	0	15.42
	4.85	1.24	11.79
	10.04	2.68	9.02
	15.20	4.17	6.80
	19.80	5.74	5.31
	25.01	7.60	4.07
	29.98	9.55	3.01
	40.19	14.2	1.50
25	0	0	16.23
	4.77	1.22	12.84
	9.85	2.62	9.77
	15.11	4.20	7.26
	19.71	5.71	5.74
	24.87	7.54	4.31
	29.95	9.53	3.18
	41.21	14.7	1.50
30	0	0	16.95
	5.30	1.36	13.33
	10.21	2.73	10.16
	15.05	4.18	7.80
	19.95	5.79	5.92
	25.26	7.69	4.46
	29.84	9.49	3.46
	40.94	14.6	1.65

[a] Calculated by the compiler

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:

An excess of nickel iodate crystals and the solvent mixtures were placed into glass–stoppered bottles. The bottles were placed into a thermostat at given temperature and rotated for 72 h. After saturation, the solution was separated from the solid phase using a glass-sintered filter. The saturated solutions were diluted with water, the iodate content was determined iodometrically.

Source and Purity of Materials:

Nickel iodate dihydrate was prepared by adding both solutions of nickel nitrate and potassium iodate to aqueous solution of potassium nitrate under heating. The precipitate was washed and dried at room temperature. Dimethylformamide (from Mitsubishi Gas Chem. Co) was distilled under reduced pressure. After the product was dried over sodium carbonate, the distillation of the solvent was repeated three times.

Estimated Error:

Solubility: probable error between $\pm 1 \times 10^{-6}$ and 1.4×10^{-4} mol dm^{-3}.
Temperature: precision ± 0.02 K.

Components:
(1) Nickel iodate; $\mathrm{Ni(IO_3)_2}$; [13477-98-0]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
or (2) 2-Propanone (acetone); C_3H_6O; [67-64-1]

Original Measurements:
M. Maneva and V. Koleva, God. Vissh. Khimikotekhnol. Inst., Sofia, **31** (3), 111–25 (1992, Pub. 1993).

Variables:
Organic solvents
One temperature: 293 K

Prepared By:
Hiroshi Miyamoto, Christo Balarew, and Cezary Guminski

Experimental Data

TABLE 34. Solubility of $\mathrm{Ni(IO_3)_2}$ in various organic solvents at 20 °C

Organic solvent	c_1 / mol dm^{-3}
Methanol	2×10^{-5}
Ethanol	1×10^{-5}
2-Propanone	1×10^{-5}

$\alpha\text{-Ni(IO}_3)_2 \cdot 4\mathrm{H_2O}$ is the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:
Saturated solutions were prepared by mixing the salt with the corresponding solvents at the selected temperature for up to 12 h. The resulting solutions were filtered at this temperature in a glass apparatus. The nickel content was determined by atomic absorption spectroscopy. The equilibrium solid phase was determined by X-ray diffraction and analyzed for the contents of Ni^{2+}, IO_3^- and H_2O.

Source and Purity of Materials:
$\alpha\text{-Ni(IO}_3)_2 \cdot 4\mathrm{H_2O}$ was obtained by mixing nickel nitrate and iodic acid (both pure for analysis) aqueous solutions in stoichiometric ratio at “proper”, but unspecified temperature. The crystals were washed with water to absence of NO_3^- ion in the solution and dried in air. Composition of the product was analyzed: Ni^{2+} — by complexometric titration, IO_3^- — by iodometric titration and H_2O — by the Fischer’s method and thermogravimetry. Distilled water was used. Methanol, ethanol and acetone were dehydrated with molecular sieves (porosity of 300 pm) and distilled; the final water content was maximally 0.5 %.

Estimated Error:
Solubility: nothing specified.
Temperature: precision ± 0.1 K.

Components:

(1) Zirconium iodate; $Zr(IO_3)_4$; [22446-84-0]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Ryo Miyamoto, Hirosaki University, Hirosaki, Japan. September 2001.

Critical Evaluation:

The Binary System

Solubility data for $Zr(IO_3)_4$ in pure water at 298 K have been reported in 3 publications by group of Shklovskaya (1–3). These studies deal with ternary systems, and the binary aqueous system is given as one point on the phase diagrams. The stable solid in equilibrium with the saturated solution was found to be anhydrous zirconium iodate. The reported solubility of 2.5×10^{-6} mol kg^{-1} is selected as a tentative value at 298 K.

The Ternary System

Solubility data for the ternary aqueous systems have been reported in 3 publications (1–3) which are summarized in Table 35.

TABLE 35. Summary for solubility studies of $Zr(IO_3)_4$ in the ternary systems with alkali metal iodate at 298 K

Ternary system	Solid phases	Reference
$Zr(IO_3)_4$–$LiIO_3$–H_2O	$Zr(IO_3)_4$; $LiIO_3$	(1) compiled in (4)
$Zr(IO_3)_4$–$NaIO_3$–H_2O	$Zr(IO_3)_4$; $NaIO_3 \cdot H_2O$; solid solution based on $Zr(IO_3)_4$	(3)
$Zr(IO_3)_4$–$RbIO_3$–H_2O	$Zr(IO_3)_4$; $RbIO_3$; solid solution based on $Zr(IO_3)_4$	(3)
$Zr(IO_3)_4$–$CsIO_3$–H_2O	$Zr(IO_3)_4$; $CsIO_3$	(2)

The dominant feature of the ternary system with $NaIO_3$ and $RbIO_3$ is the formation of solid solutions. The aqueous ternary systems with $LiIO_3$ and $CsIO_3$ are simple eutonic and no double salts were found. The detailed compilation of the ternary $Zr(IO_3)_4$–$LiIO_3$–H_2O system was given in (4).

References:

1. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and N. I. Troitskaya, Zh. Neorg. Khim. **27**, 257 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 145 (1982).

2. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **29**, 2970 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 1698 (1984).

3. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 274 (1985); Russ. J. Inorg. Chem. (Engl.Transl.) **30**, 156 (1985).

4. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 307.

Components:

(1) Zirconium iodate; $\mathrm{Zr(IO_3)_4}$; [22446-84-0]
(2) Cesium iodate; $CsIO_3$; [13454-81-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **29**, 2970–1 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 1698–9 (1984).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 36. Composition of saturated solutions in the ternary $\mathrm{Zr(IO_3)_4}$–$CsIO_3$–H_2O system at 25 °C

$\mathrm{Zr(IO_3)_4}$		$CsIO_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.00020[c]	4.6×10^{-6}	—	—	A
0.000012	2.7×10^{-6}	0.23	0.013	A
0.00011	2.5×10^{-6}	0.46	0.027	A
0.00010	2.3×10^{-6}	0.69	0.041	A
0.000080	1.8×10^{-6}	0.92	0.054	A
0.000079	1.8×10^{-6}	1.10	0.065	A
0.000075	1.7×10^{-6}	1.35	0.0800	A
0.000070	1.6×10^{-6}	1.50	0.0891	A
0.000078	1.8×10^{-6}	1.75	0.104	A
0.000091	2.1×10^{-6}	2.08	0.124	A
0.00012[d]	2.8×10^{-6}	2.29[d]	0.137	A+B
—	—	2.61[c]	0.157	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{Zr(IO_3)_4}$; B = $CsIO_3$.

[c] For binary systems the compilers compute the following values:

Solubility of $CsIO_3$ = 0.087 mol kg^{-1}
Solubility of $\mathrm{Zr(IO_3)_4}$ = 2.5×10^{-6} mol kg^{-1}.

[d] Eutonic composition.

Method/Apparatus/Procedure:

The isothermal method was used. The solutions and salts were equilibrated for 20–25 d. Samples of the coexisting phases were analyzed for cesium by flame emission spectroscopy. The cesium concentration was determined by comparing the sample solution being analyzed, previously buffered with 2 % sodium chloride solution, with reference solutions containing cesium and 2 % sodium chloride. The influences of the iodate and zirconium ions on the intensity of the emission of the cesium atoms were checked. The zirconium concentration in samples of the liquid phase was determined photometrically using Arsenazo III, after preliminary reduction of the iodate ion by hydroxylamine. The zirconium concentration in the sample of "solid residues" was determined by complexometric titration using xylenol orange indicator. The iodate content was determined iodometrically. The solid phases were identified by the method of "residues" and confirmed by X-ray diffraction.

Source and Purity of Materials:

"High purity" cesium iodate was used. Zirconium iodate was obtained from reaction of hydrated zirconium oxide and 60–70 % iodic acid at room temperature (1). The mixture was diluted 30 times and heated up to 60–80 °C. The resulting salt was dried; an analysis gave: 11.50 mass % Zr and 89.1 mass % IO_3, and calculation: 11.53 mass % Zr and 88.47 mass % IO_3. X-rays patterns confirmed structure of anhydrous $\mathrm{Zr(IO_3)_4}$.

Estimated Error:

Solubility: precision in cesium analysis ± (3–8) %.
Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and N. I. Troitskaya, Zh. Neorg. Khim. **27**, 257 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 145 (1982).

Components:

(1) Zirconium iodate; $\mathrm{Zr(IO_3)_4}$; [22446-84-0]
(2) Sodium iodate; $NaIO_3$; [7681-55-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 274–6 (1985); Russ. J. Inorg. Chem. (Engl.Transl.) **30**, 156–7 (1985).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 37. Composition of saturated solutions in the ternary $\mathrm{Zr(IO_3)_4}$–$NaIO_3$–H_2O system at 25 °C

$\mathrm{Zr(IO_3)_4}$		$NaIO_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.00020[c]	4.6×10^{-6}	—	—	A
0.00019	4.4×10^{-6}	0.78	0.072	B
0.00017	3.9×10^{-6}	1.56	0.144	B
0.00015	3.5×10^{-6}	2.49	0.232	B
0.00014	3.3×10^{-6}	3.22	0.302	B
0.00013	3.1×10^{-6}	4.07	0.385	B
0.00012	2.9×10^{-6}	4.89	0.466	B
0.00011	2.6×10^{-6}	5.43	0.520	B
0.00010	2.4×10^{-6}	6.22	0.600	B
0.00009	2.2×10^{-6}	7.24	0.706	B
0.00008	2.0×10^{-6}	7.92	0.777	B
0.00010[d]	2.5×10^{-6}	8.60[d]	0.849	C
—	—	8.66[c]	0.856	D

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{Zr(IO_3)_4}$; B = solid solution based on $\mathrm{Zr(IO_3)_4}$; C = solid solution based on $\mathrm{Zr(IO_3)_4}$ + $NaIO_3 \cdot H_2O$; D = $NaIO_3 \cdot H_2O$.

[c] For binary systems the compilers compute the following values:

Solubility of $NaIO_3$ = 0.479 mol kg^{-1}
Solubility of $\mathrm{Zr(IO_3)_4}$ = 2.5×10^{-6} mol kg^{-1}.

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied isothermally. Equilibrium was established in 25–30 d. Samples of the coexisting phases were analyzed for sodium by emission spectroscopy. An influence of zirconium and iodate ions on the sodium determination was found to be negligible. The zirconium concentration in the samples of the liquid phase was determined spectrophotometrically with Arsenazo III after preliminary reduction of the iodate with hydroxylamine. Zirconium in samples of the "solid residues" was determined by complexometric titration. In addition, samples were analyzed for the iodate ion iodometrically and the zirconium concentration was checked by the difference. The solid phases were identified by the method of "residues" and the result was checked by X-ray diffraction.

Source and Purity of Materials:

"Chemically pure" sodium iodate monohydrate was recrystallized twice. Zirconium iodate was synthesized from freshly prepared zirconium oxide hydrate and 60–70 % iodic acid at room temperature (1). The mixture was diluted 30 times and heated up to 60–80 °C. The resulting salt was dried; an analysis gave: 11.50 mass % Zr and 89.1 mass % IO_3, and calculation: 11.53 mass % Zr and 88.47 mass % IO_3. X-ray patterns confirmed structure of anhydrous $\mathrm{Zr(IO_3)_4}$.

Estimated Error:

Solubility: precision in sodium analysis ± (3–8) %.

Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and N. I. Troitskaya, Zh. Neorg. Khim. **27**, 257 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 145 (1982).

Components:

(1) Zirconium iodate; $\mathrm{Zr(IO_3)_4}$; [22446-84-0]
(2) Rubidium iodate; $RbIO_3$; [13446-76-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 274–6 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 156–7 (1985).

Variables:

Concentration of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 38. Composition of saturated solutions in the ternary $\mathrm{Zr(IO_3)_4}$–$RbIO_3$–H_2O system at 25 °C

$\mathrm{Zr(IO_3)_4}$		$RbIO_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.00020[c]	4.6×10^{-6}	—	—	A
0.00019	4.3×10^{-6}	0.36	0.025	B
0.00016	3.7×10^{-6}	0.76	0.053	B
0.00012	2.8×10^{-6}	1.00	0.0698	B
0.00011	2.5×10^{-6}	1.24	0.0868	B
0.00010	2.3×10^{-6}	1.43	0.100	B
0.00009	2.1×10^{-6}	1.64	0.115	B
0.00007	1.6×10^{-6}	1.79	0.126	B
0.00006	1.4×10^{-6}	2.02	0.142	B
0.00008[d]	1.9×10^{-6}	2.31[d]	0.163	C
—	—	2.36[c]	0.167	D

[a] Calculated by the compilers.

[b] The solid phases are: A= $\mathrm{Zr(IO_3)_4}$; B = solid solution based on $\mathrm{Zr(IO_3)_4}$; C = solid solution based on $\mathrm{Zr(IO_3)_4}$ + $RbIO_3$; D = $RbIO_3$.

[c] For binary systems the compilers compute the following values:

$$\text{Solubility of } \mathrm{RbIO_3} = 0.0928 \text{ mol kg}^{-1}$$
$$\text{Solubility of } \mathrm{Zr(IO_3)_4} = 2.5 \times 10^{-6} \text{ mol kg}^{-1}.$$

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied isothermally. Equilibrium was established in 25–30 d. Samples of the coexisting phases were analyzed for rubidium by emission spectroscopy. It was found that an influence of zirconium and iodate ions on the rubidium determination was negligible. The zirconium concentration in the samples of the liquid phase was determined spectrophotometrically with Arsenazo III after preliminary reduction of the iodate with hydroxylamine. The zirconium content in the "solid residues" was determined by complexometric titration. In addition, samples were analyzed for the iodate ion by iodometric titration and the zirconium concentration was checked by the difference. The solid phases were identified by the method of "residues" and the result was checked by X-ray diffraction.

Source and Purity of Materials:

"High purity" rubidium iodate was used. Zirconium iodate was synthesized from freshly prepared zirconium oxide hydrate and 60–70 % iodic acid at room temperature (1). The mixture was distilled 30 times and heated up to 60–80 °C. The product was dried; an analysis gave: 11.50 mass % Zr and 89.1 mass % IO_3, and calculation: 11.53 mass % Zr and 88.47 mass % IO_3. X-ray patterns confirmed structure of anhydrous $Zr(IO_3)_4$.

Estimated Error:

Solubility: precision in rubidium analysis ± (3–8) %.
Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and N. I. Troitskaya, Zh. Neorg. Khim. **27**, 257 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 145 (1982).

Components:

(1) Hafnium iodate; $Hf(IO_3)_4$; [19630-06-9]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata university, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. November 2001.

Critical Evaluation:

The Binary System

Data for the solubility of $Hf(IO_3)_4$ in water at 298 K have been reported in 3 publications (1–3). The reported data were measured by a groups of Shklovskaya. The value of 4.2×10^{-6} mol kg^{-1} is designated as a tentative solubility. All detailed compilations of (1–3) may be found in (4). The equilibrium solid phase consists of anhydrous $Hf(IO_3)_4$ (1–3).

The Ternary System

TABLE 39. Summary for solubility studies of $Hf(IO_3)_4$ in the ternary systems with alkali metal iodates at 298 K

Ternary system	Solid phases	Reference
$Hf(IO_3)_4$–$LiIO_3$–H_2O	$Hf(IO_3)_4$; $LiIO_3$; solid solution	(1)
$Hf(IO_3)_4$–$NaIO_3$–H_2O	$Hf(IO_3)_4$; $NaIO_3 \cdot H_2O$; solid solution	(2)
$Hf(IO_3)_4$–$RbIO_3$–H_2O	$Hf(IO_3)_4$; $RbIO_3$; solid solution	(3)
$Hf(IO_3)_4$–$CsIO_3$–H_2O	$Hf(IO_3)_4$; $CsIO_3$; solid solution	(3)

Deabriges and Rohmer (5) studied the ternary HfO_2–HIO_3–H_2O system at 298 K. The solid phase equilibrated with the saturated solution was amorphous hydrated hafnium oxide. The authors stated that the crystalline phase, $\mathrm{Hf(IO_3)_4}$ and $\mathrm{Hf(IO_3)_4 \cdot HIO_3 \cdot 3H_2O}$ were not soluble, what is in agreement with the value observed by (1–3).

References:

1. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and A. G. Tokareva, Zh. Neorg. Khim. **26**, 1701 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 919 (1981).

2. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and S. I. Timofeev, Zh. Neorg. Khim. **28**, 2435 (1983); Russ. J. Inorg. Chem. (Engl. Transl.) **28**, 1384 (1983).

3. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **29**, 1346 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 773 (1984).

4. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), pp. 308, 370, 441 and 456.

5. J. Deabriges, Ph. D. Thesis, Univ. of Strasbourg, France (1969); J. Deabriges and R. Rohmer, Bull. Soc. Chim. Fr. **1968**, 521.

Components:

(1) Hafnium oxide; HfO_2; [12055-23-1]
(2) Iodic acid; HIO_3; [7782-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

J. Deabriges, Ph. D. Thesis, Univ. of Strasbourg, France (1969); J. Deabriges and R. Rohmer, Bull. Soc. Chim. Fr. **1968**, 521–7.

Variables:

Concentration of iodic acid
One temperature: 298 K

Prepared By:

J. J. Counioux and Hiroshi Miyamoto

Experimental Data

TABLE 40. Solubility of HfO_2 in aqueous solutions of HIO_3 at 25 °C

I_2O_5		HfO_2	
$100w_2$	$100x_2$	$100w_1$	$100x_1$
6.1	0.36	0.039	0.0035
7.7	0.45	0.048	0.0044
10.8	0.65	0.17	0.0016
15.4	0.98	0.50	0.0050
18.8	12.5	0.96	0.10
21.0	1.44	1.35	0.15
24.9	1.79	1.43	0.16
28.7	2.17	1.50	0.18
37	3.1	1.6	0.22
40	3.6	1.7	0.24
46	4.5	1.9	0.30
47	4.7	2.2	0.35
51	5.6	2.4	0.41
57	7.0	2.6	0.51

The solid phase is amorphous hydrated hafnium oxide. The crystal phases, $\mathrm{Hf(IO_3)_4}$ and $\mathrm{Hf(IO_3)_4 \cdot HIO_3 \cdot 3H_2O}$, are observably not soluble.

Auxiliary Information

Method/Apparatus/Procedure:

Isothermal method was used. The mixtures were prepared from aqueous of iodic acid and hafnium oxide. They are shaken during one month at constant temperature. Then the solid and the liquid phases were analyzed. Iodate content was determined by iodometry after reduction with KI. Hafnium content was determined by gravimetry as $\mathrm{Hf(C_6H_5CHOHCOO)_4}$ (1), after precipitation with mandelic acid.

Source and Purity of Materials:

Hydrated hafnium oxide from Nobel–Bozel was used. The principal impurities are sulfate ion and 3 % Zr. This oxide was purified by dissolution with hydrochloric acid and subsequent precipitation with ammonia.

Estimated Error:

Nothing specified.

References:

[1] R. B. Hahn and P. T. Joseph, J. Am. Chem. Soc. **79**, 1298 (1957).

PART 2. COMPILATION AND EVALUATION OF SOLUBILITY DATA OF HALATES OF METALS IN THE MAIN GROUPS 12–14, AND AMMONIUM HALATES

V. CHLORATES OF ELEMENTS IN THE MAIN GROUPS 12–14 AND AMMONIUM CHLORATE

A. Zinc Chlorate

Components:

(1) Zinc chlorate; $\mathrm{Zn(ClO_3)_2}$; [10361-95-2]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

Data for the solubility of zinc chlorate in water have been reported in two publications (1, 2). Myllius and Funk (1) have reported the solubility at 291 K. Meusser (2) measured the solubility over a temperature range of 255–328 K.

Depending on temperature and composition, several equilibrium solid phases of varying degree of hydration have been reported. The following solid phases have been identified.

$\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$	[not available]
$\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$	[not available]
$\mathrm{Zn(ClO_3)_2 \cdot 2H_2O}$	[not available]
$\mathrm{Zn(ClO_3)_2}$	[10361-95-2]

The anhydrous salt was not observed over the temperature range investigated (255–328 K). $\mathrm{Zn(ClO_3)_2 \cdot 2H_2O}$ was prepared at 328 K, however due to its decomposition the corresponding solubility was not measured. Myllius and Funk (1) claimed that the equilibrium solid phase up to 333 K is $\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$. According to thermal analysis performed by Meusser (2) and the liquidus shape shown in Fig. 6, $\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$ undergoes peritectic transformation to $\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$ at 288 K and $\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$ is peritectically decomposed to $\mathrm{Zn(ClO_3)_2 \cdot 2H_2O}$ at 328 K. The phase equilibria proposed by Meusser (2) are more convincing than those of (1).

The solubility values reported by Myllius and Funk (1) and by Meusser (2) were given in Table 41; the solubilities read-out from Fig. 1 of (2) were also listed in. These data were used to fit to the smoothing equation given in the Preface in this volume. The detailed information of the calculation procedure for the smoothing equation has been described therein.

TABLE 41. Summary for solubility studies of $\mathrm{Zn(ClO_3)_2}$ in water at various temperatures

t / °C	T / K	Molar ratio[a]	$100w_1$	$100x_1$[b]
		Solid phase: Ice		

0[c]	273.15	—	0[c]	0
−9	264	2.80	26.54	2.73
−13	260	3.36	30.27	3.26
Solid phase: $Zn(ClO_3)_2 \cdot 6H_2O$				
−30[d]	243	9.39[d]	54.77[b]	8.58
−20[d]	253	9.70[d]	55.57[b]	8.84
−18	255	9.70	55.62	8.86
−10[d]	263	10.21[d]	56.82[b]	9.27
0	273	11.08	59.19	10.11
8	281	11.72	60.20	10.50
10[d]	283	12.89[d]	62.44[b]	11.42
12.5[d]	285.7	14.00[d]	64.35[b]	12.28
13.9[d]	287.1	15.00[d]	65.92[b]	13.04
15	288	16.64	67.32	13.79
18	291	—	65[e]	12.6
Solid phase: $Zn(ClO_3)_2 \cdot 4H_2O$				
18	291	15.39	66.52	13.35
20[d]	293	15.50[d]	66.65[b]	13.42
30	303	16.20	67.66	13.95
40	313	17.29	69.06	14.76
50[d]	323	19.71[d]	71.77[b]	16.47
55	328	24.00	75.44	19.24

[a] Molar ratio is amount of $Zn(ClO_3)_2$ mol per 100 mol of water.

[b] Calculated by the compilers

[c] Pure water

[d] Read out by the compilers from Fig. 1 of the original paper by Meusser (2)

[e] Reported by Myllius and Funk (1)

(1) Smoothing equation for $\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$ as the solid phase (243–289 K):

$$Y_x = \frac{-25454.4251}{(T/\mathrm{K})} - 228.55305 \ln{(T/\mathrm{K})} + 1236.27625 + 0.511576164(T/\mathrm{K}), \tag{39}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^6(3+6)^{3+6}}{6^6(1+x)^{3+6}}\right). \tag{40}$$

$\sigma_Y = 0.060$ and $\sigma_x = 0.0067$ were obtained.

(2) Smoothing equation for $\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$ as the solid phase (289–328 K):

$$Y_x = \frac{-434495.377}{(T/\mathrm{K})} - 2877.95678 \ln{(T/\mathrm{K})} + 16433.1795 + 4.77006994(T/\mathrm{K}), \tag{41}$$

where

$$Y_x = \ln\left(\frac{x^3(1-x)^4(3+4)^{3+4}}{4^4(1+x)^{3+4}}\right). \tag{42}$$

$\sigma_Y = 0.014$ and $\sigma_x = 0.0019$ were obtained.

(3) Smoothing equation for ice as the solid phase (260–273 K):

$$x = -0.000125484562(T/\mathrm{K})^2 + 0.0644252851(T/\mathrm{K}) - 8.23524769 \tag{43}$$

Two reported solubilities and melting point of ice gave this quadratic equation.

The tentative solubility values calculated from the smoothing equation are given in Table 42 and the smoothing curves are plotted in Fig. 6.

TABLE 42. Tentative solubility of $\mathrm{Zn(ClO_3)_2}$ in water

T / K	$100x_1$
Solid phase: Ice	
270	1.2
265	2.5
260	3.3
Solid phase: $\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$	
253	8.8
258	9.0
263	9.2
268	9.6
273	10.1
278	10.7
283	11.5
288	12.7
Solid phase: $\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$	
290	13.2
293	13.5
298	13.8
303	14.0
308	14.2

313	14.6
318	15.4
323	16.7
328	19.1

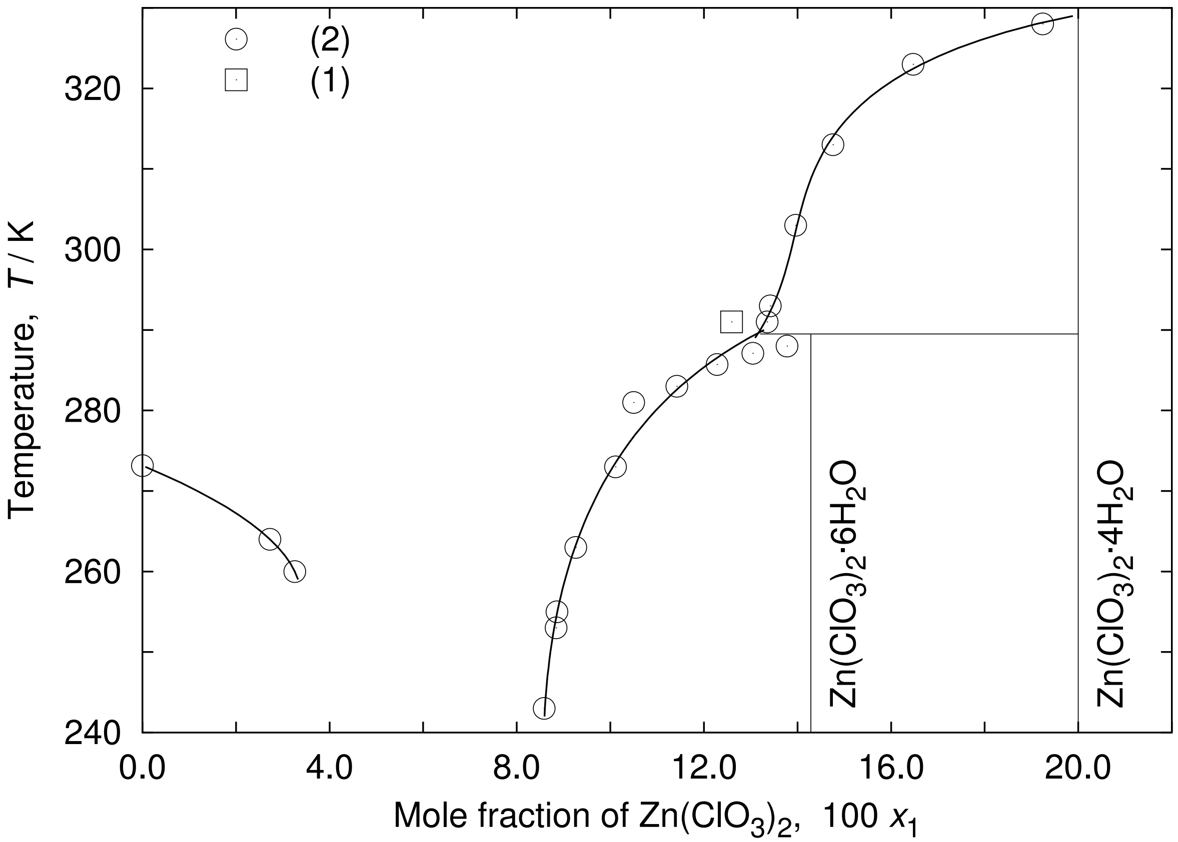

FIG. 6. Partial phase diagram of the $Zn(ClO_3)_2$–H_2O system

References:

1. F. Myllius and R. Funk, Ber. Dtsch. Chem. Ges. **30**, 1716 (1897).
2. A. Meusser, Ber. Dtsch. Chem. Ges. **35**, 1414 (1902).

Components:
(1) Zinc chlorate; $Zn(ClO_3)_2$; [10361-95-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. Myllius and R. Funk, Ber. Dtsch. Chem. Ges. **30**, 1716–25 (1897).

Variables:
One temperature: 291 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of zinc chlorate in water at 18 °C:

65 mass %	(authors)
186.2 g / 100 g H_2O	(authors)
8.02 mol kg^{-1}	(compiler)

The density of the saturated solution was given as 1.914 g cm^{-3}.

The melting point of $Zn(ClO_3)_2 \cdot 6H_2O$ was reported at 60 °C. $Zn(ClO_3)_2 \cdot 6H_2O$ was reported as the equilibrium solid.

Auxiliary Information

Method/Apparatus/Procedure:
The zinc chlorate hydrate and water were placed in a bottle and agitated in a constant temperature bath for more than 1 h. After the saturated solution settled, aliquots of the clear solution were withdrawn with a pipet and weighed. $Zn(ClO_3)_2$ was determined by evaporation to dryness.

Source and Purity of Materials:
The salt was stated to be of a “pure grade” and trace impurities negligible.

Estimated Error:
Solubility: precision within 1 %.
Temperature: nothing specified.

Components:

(1) Zinc chlorate; $\mathrm{Zn(ClO_3)_2}$; [10361-95-2]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

A. Meusser, Ber. Dtsch. Chem. Ges. **35**, 1414–24 (1902).

Variables:

Temperature: 255–328 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 43. Experimental solubility of $\mathrm{Zn(ClO_3)_2}$ in water at various temperatures

t / °C	$100w_1$	$100x_1$[a]	Solid phase[b]
-9	26.54	2.73	A
-13	30.27	3.26	A
-18	55.62	8.86	B
0	59.19	10.11	B
8	60.20	10.50	B
15	67.32	13.78	B
18	66.52	13.35	C
30	67.66	13.96	C
40	69.06	14.76	C
55	75.44	19.24	C

[a] Calculated by the compilers.

[b] The solid phases are: A = ice, B = $\mathrm{Zn(ClO_3)_2 \cdot 6H_2O}$, C = $\mathrm{Zn(ClO_3)_2 \cdot 4H_2O}$

The author observed the following phase transition of solid phases:

$$\mathrm{Zn(ClO_3)_2 \cdot 6H_2O \longrightarrow Zn(ClO_3)_2 \cdot 4H_2O} \text{ at 14.5–15 °C,}$$
$$\mathrm{Zn(ClO_3)_2 \cdot 4H_2O \longrightarrow Zn(ClO_3)_2 \cdot 2H_2O} \text{ at} > 55 \text{ °C}$$

$Zn(ClO_3)_2 \cdot 2H_2O$ was prepared at 65 °C but due to its decomposition any solubility was not measured. Specific density of the saturated solution at 18 °C was determined to be 1.916 g cm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Zinc chlorate hydrate and water were placed in a test tube and shaken for 1 h. Temperature was probably controlled by a thermostat. Aliquots of saturated solution were withdrawn with a pipet, and zinc content was determined gravimetrically as ZnS. Analysis of the solid phase gave the following results: $Zn(ClO_3)_2 \cdot 6H_2O$—67.57 mass % $Zn(ClO_3)_2$ and 32.43 mass % H_2O, this gives a mole ratio of H_2O to $Zn(ClO_3)_2$ of 6.18; $Zn(ClO_3)_2 \cdot 4H_2O$—75.44 mass % $Zn(ClO_3)_2$ and 24.56 mass % H_2O, this gives a mole ratio of H_2O to $Zn(ClO_3)_2$ of 4.20. Thermal analysis of the solid was performed with the use of a thermometer.

Source and Purity of Materials:

Zinc chlorate was prepared in this laboratory by the method reported in the literature. “Very pure” salts from Kahlbaum were used for the synthesis.

Estimated Error:

Solubility: precision is not better than ± 1 % (compilers); the difference between the theoretical and determined stoichiometry coefficients for the solid phases was equal or lower than 5 %.
Temperature: precision no better than ± 0.5 K (compilers).

Components:
(1) Cadmium chlorate; $Cd(ClO_3)_2$; [22750-54-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
A. Meusser, Ber. Dtsch. Chem. Ges. **35**, 1414–24 (1902).

Variables:
Temperature: 253–338 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 44. Experimental solubility of $Cd(ClO_3)_2$ in water at various temperatures

t / °C	T / K	$100w_1$	Molar ratio[a]	Molar ratio[b]
		Solid phase: Ice		
-6.5	266.7	26.18	3.07	2.29
-13	260	52.36	9.52	7.09
		Solid phase: $Cd(ClO_3)_2 \cdot 2H_2O$		
-20	253	72.18	22.47	16.73
-15	258	72.53	22.87	17.03
0	273	74.95	25.92	19.30
18	291	76.36	27.98	20.83
49	322	80.08	34.82	25.93
65	338	82.95	42.14	31.38

[a] Molar ratio is amount of $Cd(ClO_3)_2$ mol per 100 mol of water.

[b] Recalculated by the compilers from the reported mass % solubility using the 2003 IUPAC recommended atomic weights.

The molar ratio solubilities of cadmium chlorate recalculated by the compilers from the reported mass % solubilities are also given in Table 44. The compilers cannot reporduce the molar ratio solubilities from the reported mass % solubilities. Redetermination of cadmium chlorate solubility requires to decide the recommended solubility salt.

The melting point of the equilibrium solid phase, $\mathrm{Cd(ClO_3)_2 \cdot 2H_2O}$, was found at 79 °C. Specific density of the saturated solution at 18 °C was found to be 2.284 g $\mathrm{cm^{-3}}$. The anhydrous salt was not observed within the temperature range 253–338 K.

Auxiliary Information

Method/Apparatus/Procedure:

Cadmium chlorate hydrate and water were placed in a test tube and shaken for 1 h. Temperature was probably controlled by a thermostat. The detailed determination of the cadmium chlorate content was not reported. Analysis of the solid phase gave the following results: 87.84 mass % $\mathrm{Cd(ClO_3)_2}$ and 12.16 mass % H_2O. This gives a mole ratio of H_2O to $\mathrm{Cd(ClO_3)_2}$ of 2.14. Thermal analysis of the solid was observed by using a thermometer.

Source and Purity of Materials:

Cadmium chlorate was prepared by the double decomposition of cadmium sulfate and barium chlorate. The crystals were recrystallized, if needed. The dihydrated salt was obtained. “Very pure” salts from Kahlbaum were used for the synthesis.

Estimated Error:

Solubility: the difference between the theoretical and determined stoichiometry coefficient is about 7 %; solubility determination precision is not better than ± 2 % (compilers).
Temperature: precision no better than ± 0.5 °C (compilers).

Components:
(1) Thallium chlorate; $TlClO_3$; [13454-30-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. April 2002.

Critical Evaluation:

The Binary System

Data for the solubility of thallium chlorate in water have been reported in 3 publications (1–3). Muir (1) reported solubilities over a wide temperature range. Roozeboom (2) and Noyes and Farrell (3) measured the solubility at only selected temperatures. Rabe (4) reported solubilities of $TlClO_3$ in water at 273, 323, and 373 K, quoting Graham-Otto from 1884 as a source. In fact these data were originally calculated from a solubility equation by Muir (1). These data are not taken into account in a selection of the recommended values. All solubility data for this binary system were studied in elder times, and the evaluators did not find solubility studies in recent years through a survey of literature. A summary of solubility studies for the binary $TlClO_3$–H_2O system is given in Table 45. Anhydrous salt is in equilibrium with the saturated solution (1). All solubility results are presented in Fig. 7.

TABLE 45. Summary for solubility studies of $TlClO_3$ in water at various temperatures

T / K	m_1 / mol kg^{-1}	$100x_1$	Reference
283	0.08907[a]	0.1666	(2)
283.7	0.0983	0.1768	(1)
287.0	0.1039	0.1868	(1)
291.7	0.1289	0.2317	(1)
293.2	0.1333[a]	—[b]	(3)
298.0	0.1751	0.3145	(1)
304.1	0.2171	0.3896	(1)
312.7	0.2943	0.5274	(1)
320.8	0.3669	0.6566	(1)
329.2	0.5739	1.023	(1)
342.7	0.8772	1.556	(1)
354.7	1.139	2.011	(1)

[a] The solubility value based on mol dm^{-3} units.

[b] No information of the solution density.

The solubility of thallium chlorate given by Muir (1) and Roozeboom (2) were used to obtain a smoothing equation. Given solubility data of both m_1 by Muir and c_1 by Roozeboom were converted to mole fraction solubilities, and then the smoothing equation was estimated. The detailed information of the calculation procedure for the smoothing equation has been described earlier.

$$x = \frac{-100.766982}{(T/\mathrm{K})} - 1.29671953\ln(T/\mathrm{K}) + 6.733295 + 0.00333965616(T/\mathrm{K}), \tag{44}$$

The value $\sigma_x = 0.00044$ was obtained. The calculated smoothing curve is plotted in the Fig. 7. The solubilities calculated from the fitting equation is designated as tentative solubilities and those at round temperatures are given in Table 46.

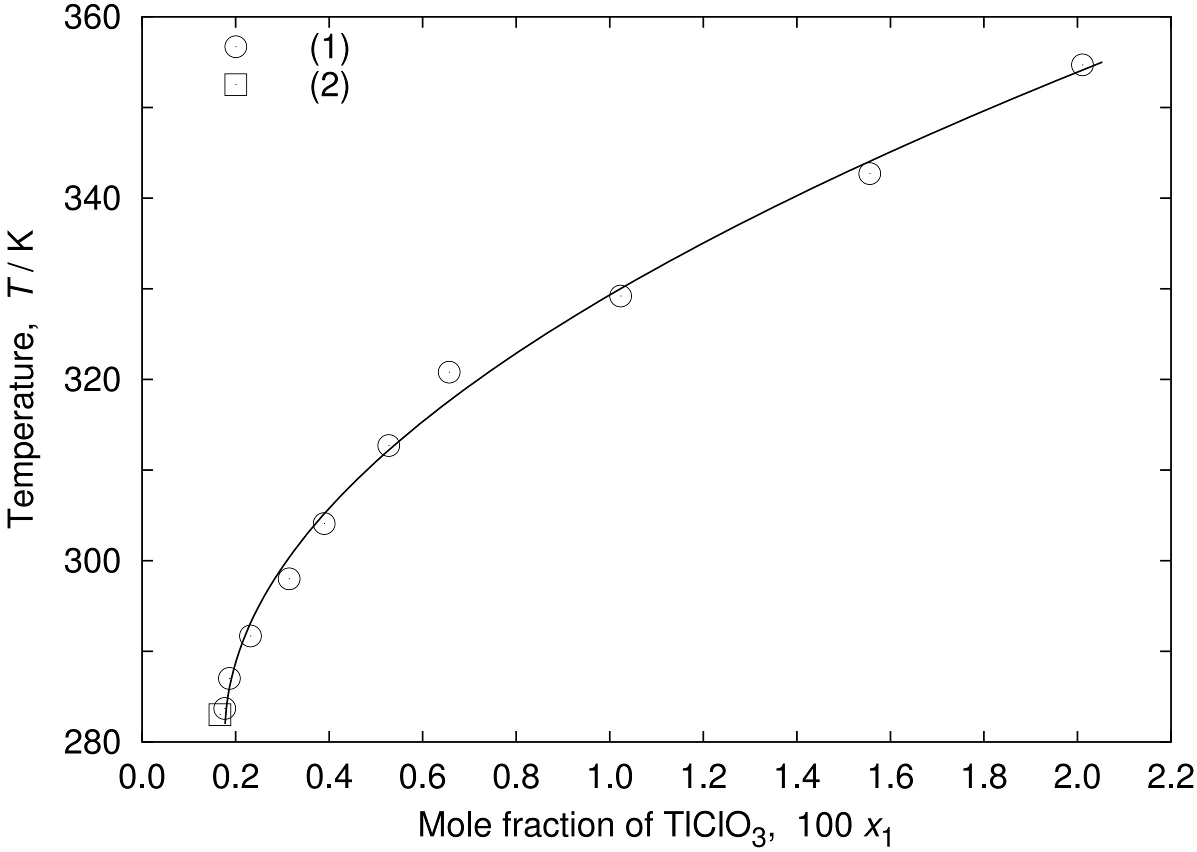

FIG. 7. Temperature dependence of solubility of $TlClO_3$ in water

TABLE 46. Tentative solubility of $TlClO_3$ in water

T / K	$100x_1$
283	0.179
288	0.196
293	0.23
298	0.28
303	0.35
308	0.44
313	0.55

318	0.67
323	0.80
328	0.96
333	1.13
338	1.31
343	1.51
348	1.73
353	1.96

The Ternary System

Roozeboom (2) studied the solubility of the ternary $TlClO_3$–$KClO_3$–H_2O system at 283 K. Noyes and Farrell (3) have reported that 1 dm^3 of aqueous solution saturated with $TlClO_3$ and Tl_2SO_4 contains 30.4 g $TlClO_3$ and 34.34 g Tl_2SO_4 at 293 K.

References:

1. J. Muir, J. Chem. Soc. **29**, 857 (1876).
2. H. W. B. Roozeboom, Z. Phys. Chem. **8**, 531 (1891).
3. A. A. Noyes and F. S. Farrell, J. Am. Chem. Soc. **33**, 1650 (1911).
4. W. O. Rabe, Z. Anorg. Chem. **31**, 154 (1902).

Components:

(1) Thallium chlorate; $TlClO_3$; [13453-30-0]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

J. Muir, J. Chem. Soc. **29**, 857–9 (1876).

Variables:

Temperature: 283.7–354.7 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 47. Solubility of $TlClO_3$ in water at various temperatures

t / °C	Mass ratio[a]	$100w_1$[b]	m_1 / mol kg^{-1} [b]	$100x_1$[b]
10.5	2.83	2.75	0.0983	0.177
13.8	2.99	2.90	0.104	0.187
18.5	3.71	3.58	0.129	0.232
24.8	5.04	4.80	0.175	0.314
30.9	6.25	5.88	0.217	0.390
39.5	8.47	7.81	0.294	0.527
47.6	10.56	9.55	0.367	0.656
56.0	16.52	14.18	0.574	1.02
69.5	25.25	20.16	0.877	1.56
81.5	32.79	24.69	1.14	2.01

[a] Mass ratio was expressed as the amount of $TlClO_3$ in g per 100 g of water in the original paper.

[b] Calculated by the compilers

The author fitted the solubilities based on the mass ratio (g $TlClO_3$ per 100 g H_2O) to the following equation:

$$\text{Solubility of TlClO}_3 = 1.99 + 0.05415\,t + 0.00139\,t^2 + 0.00036\,t^3 \qquad (45)$$

where t is the temperature in °C. A difference between the calculated and experimental data is between -9.8 % and $+6.0$ %. The solubilities at round temperatures calculated from the fitting equation were reported in the original paper.

Auxiliary Information

Method/Apparatus/Procedure:

No information is given concerning preparation of saturated solutions and their separation from the $TlClO_3$ deposit. Thallium content was analyzed by TlI precipitation. Chlorate was transformed to iodine by addition of potassium iodide excess and iodine liberated was reduced by Cu–Zn alloy to iodides and precipitated as AgI.

Source and Purity of Materials:

Thallium chlorate was prepared by mixing equivalent quantities of thallium sulfate and barium chlorate, filtering the mixture from the barium sulfate, and concentrating the solution. The purity of the thallium chlorate was finally 99.0 % and it was proved to be essentially anhydrous salt. Analyses of the salt gave: 70.69 % Tl, 12.02 % Cl. The calculated composition was: 70.91 % Tl, 12.36 % Cl and 16.73 % O.

Estimated Error:

Solubility: nothing specified but it seems to be several % in precision (compilers).

Temperature: nothing specified.

Components:

(1) Thallium chlorate; $TlClO_3$; [13453-30-0]
(2) Potassium chlorate; $KClO_3$; [3811-04-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. W. B. Roozeboom, Z. Phys. Chem. **8**, 531–5 (1891).

Variables:

Composition of $TlClO_3$ and $KClO_3$ mixed crystals
One temperature: 283 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 48. Solubility for the ternary $TlClO_3$–$KClO_3$–H_2O system at 10 °C

γ_1 / g dm^{-3}	10^3c_1 / mol dm^{-3} [a]	γ_2 / g dm^{-3}	10^3c_2 / mol dm^{-3} [a]	ρ / g cm^{-3}	$100y_2$ [b]
25.637	89.07	—	—	1.0210	0
19.637	68.22	6.884	56.2	1.0222	2.00
12.001	41.69	26.100	213.0	1.0278	12.61
9.036	31.39	40.064	326.9	1.0338	25.01
7.885	27.39	46.497	379.4	1.0359	36.30, 97.93
7.935	27.57	49.535	379.7	1.0360	36.30, 97.93
6.706	23.30	46.410	378.7	1.0357	99.28
6.723	23.30	47.109	384.4	1.0363	99.60
4.858	16.84	47.312	386.1	1.0345	99.62
2.769	9.62	47.134	384.6	1.0330	99.67
—-	—	49.925	407.4	1.0330	100

[a] Recalculated by the compiler.

[b] The value was expressed as the amount in mol % of $KClO_3$ in mixed crystals ($TlClO_3$ + $KClO_3$).

Auxiliary Information

Method/Apparatus/Procedure:

Solutions of $TlClO_3$ and $KClO_3$ were mixed in different proportion, such amounts being taken that no more than 1 or 2 g of the salts would separate from 1 dm^{-3} of the solution. The solutions were placed in flasks. The flasks were held in a large water-bath maintained at the selected temperature. Allowing the solution to settle for more than 2 d, a portion of the solution was evaporated to determine the total salt content. Other portion was used to determine Tl as TlI precipitate which was weighed after drying at about 100 °C. The composition of the crystals was also determined.

Source and Purity of Materials:

$TlClO_3$ was prepared as it follows. Pure thallium was dissolved in sulfuric acid under heating, the resulted Tl_2SO_4 crystals were separated from the solution by cooling. $Ba(OH)_2$ was added to Tl_2SO_4 solution and excess $Ba(OH)_2$ was removed from the solution by passing through CO_2. TlOH solution was neutralized by pure chloric acid, and $TlClO_3$ crystals were obtained. The precipitate was recrystallized several times. $KClO_3$ was recrystallized.

Estimated Error:

Solubility: nothing specified; precision no better than ± 0.5 % (compilers).
Temperature: nothing specified.

Components:

(1) Thallium chlorate; $TlClO_3$; [13453-30-0]
(2) Thallium sulfate; Tl_2SO_4; [7446-18-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

A. A. Noyes and F. S. Farrell, J. Am. Chem. Soc. **33**, 1650–63 (1911).

Variables:

Composition of solutions
One temperatures: 293 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 49. Solubility of $TlClO_3$ in water at 20 °C

V / cm^3 solution	m_1 / g $TlClO_3$	γ_1 / g dm^{-3} [a]	c_1 / mol dm^{-3} [b]
50.01	1.9200	38.39[c]	0.1334
50.13	1.9343	38.59[d]	0.1341
50.01	1.9220	38.41[c]	0.1334
50.13	1.9380	38.67[d]	0.1343
50.07	1.9220	38.38[c]	0.1333
50.13	1.9330	38.57[d]	0.1340
49.97	1.9219	38.47[c]	0.1337
50.01	1.9332	38.65[d]	0.1343
50.07	1.9266	38.44[c]	0.1335
49.97	1.9312	38.65[d]	0.1343

[a] $\gamma_1 = m_1/V$: mass concentration of $TlClO_3$, where m_1 = mass of $TlClO_3$ and V = volume of solution.
[b] Calculated by the compilers.
[c] undersaturation
[d] supersaturation

The mean value calculated by the compilers is 0.1338 mol dm^{-3}, and the standard deviation is 0.0004 mol dm^{-3}. The author suggested the solubility value of 0.1340 mol dm^{-3}.

TABLE 50. Solubility of $TlClO_3$ and Tl_2SO_4 in water at 20 °C when present together (g dm^{-3} units)

Total salt	Tl_2SO_4 (by anal.)	Tl_2SO_4 (by diff.)	$TlClO_3$ (by anal.)	$TlClO_3$ (by diff.)
64.86	34.34	34.59[a]	30.27[a]	30.43
64.76	34.44	34.67[a]	30.09[a]	30.32
64.88	34.45	34.43	30.45	30.43
64.67[a]	lost	34.19[a]	30.48	lost
64.89	34.52	34.43	30.46	30.37
64.90	34.47	34.36	30.44	30.43
64.69	34.51	34.35	30.34	30.18
(av.) 64.83	(av.) 34.47	(av.) 34.39	(av.) 30.43	(av.) 30.36

[a] Omitted by the authors in calculation of the average values.

Auxiliary Information

Method/Apparatus/Procedure:

$TlClO_3$ crystals and water were placed in bottles. The bottles were rotated in a thermostat for 2–3 h. Saturation was approached from both the under- and supersaturated side. The contents of the bottles were allowed to settle and portions of the solution were drawn out through a filter equipped with a piece of cotton wool. When only one salt was present, a sample was evaporated in a platinum dish on a steam bath and dried. When both sulfate and chlorate were present, in order to determine the total salt content, the portions of the solutions were evaporated to dryness. The residue was dissolved in water and excess of $BaCl_2$ was added to the solution. The precipitated $BaSO_4$ was weighed. Chloride-free metallic zinc and sulfuric acid was added to the other portion. After zinc was filtered off, a slight excess of $AgNO_3$ was added to the filtrate. The silver chloride obtained was filtered off, dried and then weighed.

Source and Purity of Materials:

Thallium(I) sulfate was prepared from metallic thallium and sulfuric acid. The product was recrystallized form boiling water. Thallium chlorate was made by mixing hot, neutral solutions containing equivalent quantities of barium chlorate and thallium(I) sulfate. After a crystallization of the product its fine crystals were obtained; they contained 12.13–12.32 % Cl whereas the calculated content was 12.32 % Cl.

Estimated Error:

Solubility: precision ± 0.3 % (compilers).
Temperature: precision ± 0.03 K.

Components: (1) Lead chlorate; $Pb(ClO_3)_2$; [10294-47-0] (2) Water; H_2O; [7732-18-5]
Evaluators: Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. May 2002.

Critical Evaluation:

The solubility study of lead chlorate in water has been published in 2 publications (1, 2). Myllius and Funk (1) reported that 100 g H_2O dissolves 151.3 g $Pb(ClO_3)_2$ at 291 K. The density of the solution was found to be 1.947 g cm^{-3}. The solubility value of 4.04 mol kg^{-1} is calculated from this value by the evaluators. This value may be designated as a tentative solubility. Carlson (2) also studied the solubility and the density at 291 K. This results have been described in Linke's book (3) that 100 g H_2O dissolves 440 g $Pb(ClO_3)_2$ and the density of such solution is 1.63 g cm^{-3}. Because the original paper by Carlson (2) was not available to compilers, the compilation sheet is not prepared.

There is a great difference between two reported solubilities and densities. The additional studies are needed to decide the recommended solubility value, however saturating concentration ~ 12 mol kg^{-1} of (2) seems to be improbably high comparing to the similar salt solubilities.

References:

1. F. Myllius and R. Funk, Ber. Dtsch. Chem. Ges. **30**, 1716 (1897).
2. B. Carlson, *Klason-Festschrift* (Stockholm, 1910), p. 247; *Tabelles Annuelles* (1910) Vol. 1, p. 379.
3. W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1294.

Components:
(1) Lead chlorate; $\mathrm{Pb(ClO_3)_2}$; [10294-47-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. Myllius and R. Funk, Ber. Dtsch. Chem. Ges. **30**, 1716–25 (1897).

Variables:
One temperature: 291 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 51. Solubility of $\mathrm{Pb(ClO_3)_2}$ in water at 18 $^\circ\mathrm{C}$

Mass ratio[a]	$100w_1$	m_1 / mol kg^{-1} [b]	$100x_1$ [b]	ρ / g cm^{-3}
151.3	60.2	4.04	6.79	1.947

[a] The solubility was expressed as the amount in g of $\mathrm{Pb(ClO_3)_2}$ per 100 g of H_2O.
[b] Calculated by the compoler.

$\mathrm{Pb(ClO_3)_2 \cdot H_2O}$ was reported as the equilibrium solid.

Auxiliary Information

Method/Apparatus/Procedure:
The lead chlorate hydrate and water were placed in a bottle and agitated in a constant temperature bath for more than one hour. After the saturated solution settled, aliquots of the clear solution were withdrawn with a pipet, and weighed. $\mathrm{Pb(ClO_3)_2}$ was determined by evaporation to dryness.

Source and Purity of Materials:
The salt was stated to be of a ‘pure grade’ and trace impurities stated to be negligible.

Estimated Error:
Solubility: precision within 1 %.
Temperature: nothing specified.

Components:

(1) Ammonium chlorate; NH_4ClO_3; [10192-29-7]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. May 2002.

Critical Evaluation:

The Binary System

There is only one reported publication (1) on NH_4ClO_3 solubility. The ternary NH_4ClO_3–NH_4NO_3–H_2O and NH_4ClO_3–$NaNO_3$–H_2O systems are also reported in the publication (1). Numerical data of the solubility at 273, 298 and 313 K are reported and the phase diagram fragment for the binary system extended to 352 K was included. Therefore, the data points on the phase diagram were read by the evaluators. The solid phase equilibrated with the saturated solution is the anhydride. The phase diagram for the binary system is reproduced in Fig 8. The eutectic point was found at 264 K and 4.4 % NH_4ClO_3.

The solubility data given in Table 54 were used to fit to the smoothing equation given in the Preface in this volume; the freezing point of pure water (273.15 K) was also used. The detailed information on the calculation procedure for the smoothing equation has been described earlier.

(1) Smoothing equation for NH_4ClO_3 as the solid phase (264–352 K):

$$x = \frac{1933.73153}{(T/\mathrm{K})} + 11.8842375\ln(T/\mathrm{K}) - 69.1907156 - 0.0165056713(T/\mathrm{K}), \qquad (46)$$

where $\sigma_x = 0.0037$ was obtained.

(2) Smoothing equation for ice as the solid phase (264–273 K):

$$x = \frac{49589.2455}{(T/\mathrm{K})} + 336.477615\ln(T/\mathrm{K}) - 1913.47961 - 0.570062769(T/\mathrm{K}), \qquad (47)$$

where $\sigma_x = 0.00057$ was obtained.

The calculated smoothing curves are plotted in the Fig. 8. The solubilities calculated from the fitting equations are designated as tentative solubilities and at various temperature are given in Table 52. The eutectic point is determined to be 264.2 K at 4.3 mole % NH_4ClO_3.

TABLE 52. Tentative solubility of NH_4ClO_3 in water

T / K	$100x_1$
Solid phase: Ice	
271	0.71
269	1.53
267	2.52
265	3.72

Solid phase: NH_4ClO_3

265	4.32
273	5.1
283	6.3
293	7.7
298	8.5
303	9.3
313	11.0
323	12.8
333	14.5
343	16.3
348	17.1

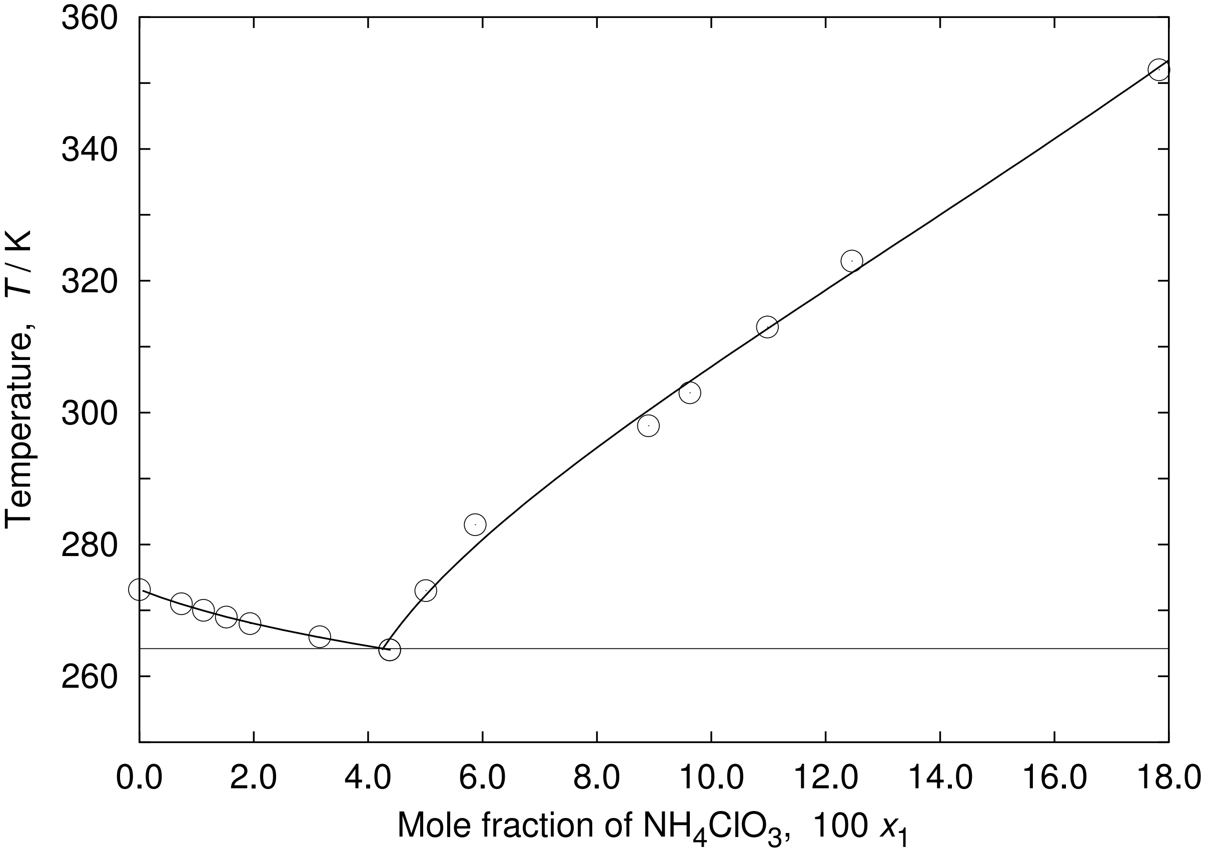

FIG. 8. Partial phase diagram of the NH_4ClO_3–H_2O system.

The Ternary System

Data for ternary systems have been reported in two publications(1, 2). A summaray of these studies is given in Table 53

TABLE 53. Summary for solubility studies of NH_4ClO_3 in ternary systems

Ternary system	T / K	Solid phases	Reference
NH_4ClO_3–NH_4NO_3–H_2O	258, 273, 298, 313	NH_4ClO_3; $NaNO_3$	(1)
NH_4ClO_3–$NaClO_3$–H_2O	273, 313	NH_4ClO_3; $NaClO_3$	(1)
NH_4ClO_3–NH_4NO_3–H_2O	273, 313	NH_4ClO_3; $NaNO_3$	(2)
NH_4ClO_3–$NaClO_3$–H_2O	273, 313	NH_4ClO_3; $NaClO_3$	(2)

Two systems in (2) are of simple eutectic types, and no double salts and their hydrates were formed.

The Multicomponent System

The solubility data for the NH_4ClO_3–$NaClO_3$–$NaNO_3$–H_2O system were reported by Hara et al. (2). The equilibrium NH_4NO_3 + $NaNO_3$ $\leftrightarrow$ NH_4ClO_3 + $NaNO_3$ occurs by double decomposition. Although the primary solid phases equibriated with the saturated solutions are NH_4NO_3, NH_4ClO_3, $NaClO_3$ and $NaNO_3$, two components (namely) $NaClO_3$ and $NaNO_3$ are not present in the system. No double salts and their hydrates were formed.

References:

1. Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 309 (1984).
2. Y. Hara, J. Iwao, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 267 (1984).

Components:
(1) Ammonium chlorate; NH_4ClO_3; [10192-29-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 309 (1984).

Variables:
Temperature: 264–352 K

Prepared By:
Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

The phase diagram for this binary system was shown in a figure. Numerical solubility data were read from the figure by the compilers.

TABLE 54. Experimental solubility of NH_4ClO_3 in water at various temperatures

t / °C	T / K	$100w_1$	$100x_1$ [c]	Solid phase[d]
−2	271	4	0.7	A
−3	270	6	1.1	A
−4	269	8	1.5	A
−5	268	10	1.9	A
−7	266	15.5	3.15	A
−9[a]	264	20.5[b]	4.37	A+B
0	273	22.9	5.01	B
10	283	26	5.90	B
25	298	35.5[b]	8.90	B
30	303	37.5	9.63	B
40	313	41.0[b]	11.0	B
50	323	44.5	12.5	B
79	352	55	17.8	B

[a] Eutectic point

[b] Numerical solubility value was reported in the original paper.

[c] Calculated by the compilers

[d] The solid phases are: A = ice; B = NH_4ClO_3.

Auxiliary Information

Method/Apparatus/Procedure:

Below 0 °C the solubilities for the binary system NH_4ClO_3–H_2O were determined by using the cooling curve method. Above 0 °C the NH_4ClO_3 content in the saturated solution was determined by conductometric titration.

Source and Purity of Materials:

Ammonium chlorate was prepared as described in (1). Ammonium chlorate was prepared from ammonium sulfate and barium chlorate. After barium sulfate was centrifuged off the clear solution was concentrated under reduced pressure below 50 °C. The crystals obtained were recrystallized from ethanol–water mixture.

Estimated Error:

Solubility: nothing specified; reading-out procedure ± 0.5 mass %.

Temperature: nothing specified; reading-out procedure about ± 1 K.

References:

[1] Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 87 (1984).

Components:

(1) Ammonium chlorate; NH_4ClO_3; [10192-29-7]
(2) Ammonium nitrate; NH_4NO_3; [6484-52-2]
(3) Sodium chlorate; $NaClO_3$; [7775-09-9]
(4)Water; H_2O; [7732-18-5]

Original Measurements:

Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 309 (1984).

Variables:

Composition of NH_4ClO_3 and NH_4NO_3
Temperature: 258–313 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 55. Composition of saturated solutions in the NH_4ClO_3–NH_4NO_3–H_2O and NH_4ClO_3–$NaClO_3$–H_2O systems

	$NaClO_3$		NH_4NO_3		NH_4ClO_3		
t / °C	$100w_3$	$100x_3$ [a]	$100w_2$	$100x_2$ [a]	$100w_1$	$100x_1$ [a]	Solid phase[b]
−15	—	—	38.0	12.1	0	0	A
	—	—	33.4	11.0	7.0	1.8	A
	—	—	33.0	10.9	8.0	2.1	A+B
	—	—	38.2	13.7	9.2	2.6	B+C
	—	—	38.9	13.6	6.7	1.8	C
	—	—	44.2	15.1	0	0	C
0	—	—	0	0	22.9[c]	5.01	B
	—	—	10.2	2.97	17.9	4.11	B
	—	—	12.1	3.58	17.6	4.10	B
	—	—	34.9	12.3	11.1	3.09	B
	—	—	36.1	13.1	12.3	3.53	B
	—	—	37.9	13.7	10.4	2.97	B
	—	—	49.0	20.6	10.4	3.45	B+C
	—	—	49.3	20.2	8.3	2.67	C
	—	—	54.2	21.0	0	0	C
0	10.1	2.20	—	—	16.9	3.86	B
	18.5	4.29	—	—	14.2	3.46	B
	26.0	6.39	—	—	11.6	2.99	B
	33.1	8.65	—	—	8.4	2.6	B
	45.4	13.9	—	—	8.6	2.8	B+D
	44.5	12.9	—	—	5.4	1.6	D
	44.1	11.8	—	—	0	0	D
25	—	—	0	0	35.5	8.90	B
	—	—	16.4	5.81	28.9	8.08	B
	—	—	45.6	20.6	18.1	6.45	B

	—	—	59.6	31.2	13.2	5.45	B+C
	—	—	60.0	30.7	11.6	4.68	C
	—	—	63.6	31.7	6.7	2.6	C
	—	—	67.6	32.0	0.	0	C
40	—	—	0	0	41.0[c]	11.0	B
	—	—	11.1	3.95	34.2	9.60	B
	—	—	19.7	7.44	30.6	9.12	B
	—	—	54.8	29.4	19.0	8.05	B
	—	—	64.0	38.6	15.9	7.56	B
	—	—	66.0	40.8	15.1	7.36	B+C
	—	—	69.3	39.5	8.3	3.7	C
	—	—	74.6	39.8	0	0	C
40	9.0	3.32	—	—	32.7	8.84	B
	10.9	2.83	—	—	31.3	8.52	B
	30.8	9.16	—	—	21.3	6.65	B
	42.2	13.9	—	—	16.3	5.61	B
	45.5	15.5	—	—	15.1	5.38	B+D
	46.8	15.7	—	—	12.8	4.49	D
	49.6	15.8	—	—	6.8	2.27	D
	55.7	17.5	—	—	0	0	D

[a] Calculated by the compilers.

[b] The solid phases are: A = ice; B = NH_4ClO_3; C = NH_4NO_3; D = $NaClO_3$

[c] For the binary system the compilers computed the following values:

$$\text{Solubility of } NH_4ClO_3 = 2.93 \text{ mol kg}^{-1} \text{ at } 0\ ^\circ\text{C}$$
$$= 5.42 \text{ mol kg}^{-1} \text{ at } 25\ ^\circ\text{C}$$
$$= 6.85 \text{ mol kg}^{-1} \text{ at } 40\ ^\circ\text{C}$$

Invariant point data for the ternary NH_4ClO_3–NH_4NO_3–H_2O system were determined: temperature −21 °C and composition NH_4NO_3–34, NH_4ClO_3–8, and H_2O–58 mass %.

Auxiliary Information

Method/Apparatus/Procedure:

Mixture of NH_4ClO_3, NH_4NO_3 and H_2O and that of $NaClO_3$, NH_4ClO_3 and H_2O were frequently shaken in a thermostat at the selected temperatures for 4 h. Analyses of the liquid and solid phases were performed by the "residues" method. In the NH_4ClO_3–NH_4NO_3–H_2O system total ammonium ion concentration was determined conductometrically and the nitrate ion concentration was determined specrophotometricaly. The amount of ammonium chlorate was calculated from the total ammonium and nitrate ion concentrations. In the case of $NaClO_3$–NH_4ClO_3–H_2O system the samples of the liquid and solid phases were analyzed for chlorate and ammonium ions. Chlorate concentration was determined by the Volhard method after the chlorate was reduced to chloride. The ammonium ion concentration was determined conductometrically. Sodium content was found by the difference.

Source and Purity of Materials:

Guarantee pure grade ammonium nitrate and sodium chlorate were recrystallized. Ammonium chlorate was prepared as described in (1). Aqueous solutions of ammonium sulfate and barium chlorate were mixed. Barium sulfate precipitate was centrifuged, and then the resulting solution was evaporated at temperatures below 50 °C under reduced pressure. The product was recrystallized from ethanol–water mixture.

Estimated Error:

Solubility: nothing specified.

Temperature: nothing specified.

References:

[1] Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 309 (1984).

Components:
(1) Ammonium chlorate; NH_4ClO_3; [10192-29-7]
(2) Sodium nitrate; $NaNO_3$; [7631-99-4]
(3) Ammonium nitrate; NH_4NO_3; [6484-52-2]
or (3) Sodium chlorate; $NaClO_3$; [7775-09-9]
(4) Water; H_2O; [7732-18-5]

Original Measurements:
Y. Hara, J. Iwao, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 267 (1984).

Variables:
Composition of NH_4ClO_3, NH_4NO_3, $NaNO_3$, and $NaClO_3$,
Temperature: 273 and 313 K

Prepared By:
Hiroshi Miyamoto and Ryo Miyamoto

Experimental Data

TABLE 56. Solubility in the NH_4ClO_3–$NaNO_3$–NH_4NO_3–$NaClO_3$–H_2O system

(1) NH_4ClO_3–$NaNO_3$–NH_4NO_3–H_2O system at 0 °C

NH_4NO_3		$NaNO_3$		NH_4ClO_3		H_2O	
$100w_3$	$100x_3$[a]	$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	w_{H_2O} / $w_{\text{total salts}}$ [b]	Solid phase[c]
82.5	85.7	0	0	17.5	14.3	0.682	A+C
63.2	66.2	21.3	21.0	15.5	12.8	0.555	A+C
51.0	53.8	34.2	33.9	14.8	12.3	0.500	A+C
62.8	64.1	37.4	35.9	0	0	0.549	B+C
39.7	42.4	41.8	42.0	18.5	15.6	0.518	A+B
5.8	6.5	59.4	62.8	34.7	30.7	0.571	A+B
48.4	51.1	37.0	36.8	14.6	14.6	0.485	A+B+C

(2) NH_4ClO_3–$NaNO_3$–$NaClO_3$–H_2O system at 0 °C

$NaClO_3$		$NaNO_3$		NH_4ClO_3		H_2O	
$100w_3$	$100x_3$[a]	$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	w_{H_2O} / $w_{\text{total salts}}$ [b]	Solid phase[c]
84.1	83.5	0	0	15.9	16.5	0.847	A+D
60.1	56.5	21.7	25.5	18.2	17.9	0.752	A+D
47.7	43.6	33.4	38.3	18.9	18.1	0.714	A+D
42.8	38.8	37.4	42.5	19.7	18.7	0.685	A+D
54.4	48.8	45.6	51.2	0	0	0.862	B+D
43.1	38.5	44.1	49.4	12.9	12.1	0.714	B+D
26.1	23.0	49.2	54.2	24.7	22.8	0.581	A+B
37.5	33.5	42.9	48.1	19.6	18.4	0.571	A+B+D

(3) NH_4ClO_3–$NaNO_3$–NH_4NO_3–H_2O system at 40 °C

NH_4NO_3		$NaNO_3$		NH_4ClO_3		H_2O	
$100w_3$	$100x_3$[a]	$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	w_{H_2O} / $w_{\text{total salts}}$ [b]	Solid phase[c]
85.1	87.9	0	0	14.9	12.1	0.307	A+C

75.6	78.4	11.0	10.7	13.3	10.9	0.296	A+C
67.0	70.2	19.2	18.8	13.2	10.9	0.259	A+C
74.3	75.4	25.7	24.6	0	0	0.260	B+C
66.3	69.0	21.2	20.8	12.5	10.3	0.261	B+C
61.5	64.4	23.5	23.2	15.1	12.5	0.275	A+B
45.8	49.0	31.9	32.2	22.3	18.8	0.346	A+B
38.0	41.1	37.2	37.9	24.7	21.1	0.373	A+B
15.3	17.1	49.2	51.7	35.5	31.2	0.427	A+B
63.8	66.7	21.7	21.4	14.5	12.0	0.251	A+B+C

(4) NH_4ClO_3–$NaNO_3$–$NaClO_3$–H_2O system at 40 °C

$NaClO_3$		$NaNO_3$		NH_4ClO_3		H_2O	
$100w_3$	$100x_3$[a]	$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	w_{H_2O} / $w_{\text{total salts}}$[b]	Solid phase[c]
75.2	74.3	0	0	24.8	25.7	0.650	A+D
58.0	55.4	13.0	15.6	29.0	29.1	0.663	A+D
42.8	39.5	27.2	31.4	30.0	29.0	0.571	A+D
55.4	49.8	44.6	50.2	0	0	0.565	B+D
41.9	37.3	46.2	51.5	11.9	11.1	0.549	B+D
31.2	27.7	46.1	51.5	22.7	21.1	0.485	B+D
26.1	23.1	46.1	51.1	27.8	25.8	0.475	B+D
0	0	56.3	60.6	43.7	39.4	0.424	A+B
21.7	19.2	44.2	49.1	34.1	31.7	0.410	A+B+D

[a] Calculated by the compilers.

[b] Mass ratio of water and total salts: w_{H_2O} / $w_{\text{total salts}}$.

[c] The solid phases are: A = NH_4ClO_3; B = $NaNO_3$; C = NH_4NO_3; D = $NaClO_3$.

w_i is a mass fraction of the ith species in the total amount of salts, and x_i is a mole fraction of the ith species in the total amount of salts.

Auxiliary Information

Method/Apparatus/Procedure:

Mixtures of $\mathrm{NH_4ClO_3}$, $\mathrm{NH_4NO_3}$, $NaNO_3$, and H_2O, and that of $\mathrm{NH_4ClO_3}$, $NaNO_3$, $NaClO_3$, and H_2O were frequently shaken in a thermostat at the selected temperatures for 4 h. The components in the saturated solutions and those in the solid phases were analyzed by the "residue method". Ammonium ion and nitrate ion concentrations were determined by conductometric and specrophotometric methods, respectively. The chlorate concentration was determined by the Volhard method after chlorate was reduced to chloride.

Source and Purity of Materials:

Ammonium chlorate was prepared as described in (1). Aqueous solutions of ammonium sulfate and barium chlorate were mixed. Barium sulfate precipitate was centrifuged, and then the resulting solution was evaporated at temperatures below 50 °C under reduced pressure. The product was recrystallized from ethanol–water mixture. Guarantee pure grade ammonium nitrate, sodium chlorate, and sodium nitrate were recrystallized.

Estimated Error:

Solubility: nothing specified.
Temperature: nothing specified.

References:

[1] Y. Hara, H. Nakamura, Y. Hirosaki, K. Hattori, and H. Osada, Kogyo Kayaku **45**, 87 (1984).

A. Cadmium Bromate

Components:

(1) Cadmium bromate; $\mathrm{Cd(BrO_3)_2}$; [14518-94-6]
(2) Sodium bromate; $NaBrO_3$; [7789-38-0]
(3) Potassium bromate; $KBrO_3$; [7758-01-2]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

I. M. Karataeva, S. V. Petrenko, and E. E. Vinogradov, Zh. Neorg. Khim. **33**, 786–90 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 442–4 (1988).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 57. Solubility in the $\mathrm{Cd(BrO_3)_2}$–$NaBrO_3$–$KBrO_3$–H_2O system at 25 °C

m_3 / mol kg^{-1}	m_2 / mol kg^{-1}	m_1 / mol kg^{-1}	Solid phase [a]
0.313	0.809	0	A+B
0.299	2.652	0.2	A+B
0.288	2.336	0.40	A+B
0.280	2.130	0.60	A+B
0.273	1.943	0.80	A+B
0.268	1.770	1.00	A+B
0.263	1.612	1.20	A+B
0.260	1.465	1.40	A+B
0.256	1.329	1.60	A+B
0.254	1.202	1.80	A+B
0.251	1.085	2.00	A+B
0.248	0.974	2.20	A+B
0.245	0.872	2.40	A+B
0.242	0.777	2.60	A+B
0.242	0.775	2.603	A+B+C
0.215	0	2.918	B+C
0.219	0.150	2.855	B+C
0.224	0.30	2.794	B+C
0.229	0.45	2.733	B+C
0.235	0.60	2.672	B+C
0.241	0.75	2.614	B+C
0.242	0.775	2.603	A+B+C
0	0.748	2.611	A+C

0.05	0.753	2.610	A+C
0.10	0.758	2.609	A+C
0.15	0.764	2.606	A+C
0.20	0.770	2.605	A+C
0.261[b]	0.773	2.624	A+B+C

[a] The solid phases are: A = $NaBrO_3$; B = $KBrO_3$; C = $Cd(BrO_3)_2 \cdot H_2O$

[b] Experimental result for the eutonic point.

The system is of a simple eutonic type.

TABLE 58. Solubility in the ternary $Cd(BrO_3)_2$–$NaBrO_3$–H_2O system at 25 °C

$100w_2$	$100x_2$[a]	m_2 / mol kg^{-1}	$100w_1$	$100x_1$[a]	m_1 / mol kg^{-1}	Solid phase [b]
28.95	4.64	2.70	0	0	0	A
22.82	3.87	2.25	9.91	0.688	0.40	A
17.91	3.25	1.87	18.67	1.374	0.80	A
14.00	2.67	1.56	26.35	2.06	1.20	A
10.87	2.20	1.28	33.04	2.74	1.60	A
8.35	1.79	1.05	38.86	3.41	2.00	A
16.24[c]	2.93	1.71	20.64	1.53	0.89	A
7.58	1.67	0.97	41.06	3.70	2.17	A
6.34	1.44	0.85	43.94	4.08	2.40	A
5.44	1.27	0.74	46.35	4.43	2.61	A+B
0	0	0	51.81	5.00	2.92	B
1.09	0.256	0.15	50.69	4.88	2.86	B
2.18[c]	0.511	0.30	49.58	4.76	2.79	B
2.18	0.511	0.30	49.60	4.77	2.79	B
3.27	0.766	0.45	48.50	4.65	2.73	B
4.36	1.02	0.60	47.42	4.54	2.67	B
5.44	1.27	0.74	46.35	4.43	2.61	A+B

[a] Calculated by the compilers.

[b] The solid phases: A = $NaBrO_3$; B = $Cd(BrO_3)_2 \cdot H_2O$.

[c] Experimental values determined by isothermal method.

TABLE 59. Solubility in the ternary $\mathrm{Cd(BrO_3)_2}$–$KBrO_3$–H_2O system at 25 °C

$100w_3$	$100x_3$[a]	m_3 / mol kg^{-1}	$100w_1$	$100x_1$[a]	m_1 / mol kg^{-1}	Solid phase [b]
7.42	0.857	0.48	0	0	0	A
4.22[c]	0.537	0.30	12.02	0.693	0.38	A
3.47	0.453	0.25	15.01	0.889	0.50	A
2.60	0.386	0.22	26.21	1.76	1.00	A
2.24	0.372	0.21	34.78	2.62	1.50	A
2.08[c]	0.377	0.22	40.62	3.34	1.93	A
2.02	0.371	0.21	41.55	3.46	2.00	A
1.84	0.370	0.22	47.05	4.29	2.50	A
1.70	0.366	0.21	50.91	4.97	2.92	A+B
1.72[c]	0.369	0.22	50.71	4.94	2.89	A+B
0	0	0	51.81	5.00	2.92	B
0.79	0.17	0.1	51.34	4.98	2.92	B
1.58	0.340	0.2	50.97	4.98	2.92	B
1.70	0.366	0.21	50.91	4.97	2.92	A+B

[a] Calculated by the compilers.

[b] The solid phases are: A = $KBrO_3$; B = $\mathrm{Cd(BrO_3)_2 \cdot H_2O}$.

[c] Experimental values determined by isothermal method.

A solubility of $\mathrm{Cd(BrO_3)_2}$ of 57 mass % at 50 °C was read from a figure by the compilers. All data have a tentative character.

Auxiliary Information

Method/Apparatus/Procedure:

The $KBrO_3$–$NaBrO_3$–$\mathrm{Cd(BrO_3)_2}$–H_2O and the partial ternary systems were mainly studied by the calculation–experimental method using Pitzer's equation (1). The experimental results were obtained by the method of isothermal saturation of the solution (2). The samples were equilibrated for 1.5–2 d. The bromate concentration was determined by volumetric titration with sodium thiosulfate and potassium by gravimetric method after its tetraphenylborate precipitation. Determination of sodium cadmium was not described.

Source and Purity of Materials:

Twice recrystallized "chemically pure" grade sodium and potassium bromates were used. Cadmium bromate was prepared from $HBrO_3$ and CdO, the product was recrystallized, and the resulting crystals of $\mathrm{Cd(BrO_3)_2 \cdot H_2O}$ were used in this work.

Estimated Error:

Solubility: precision of ± 0.3 mass %.

Temperature: nothing specified.

References:

[1] K. S. Pitzer and G. Mayorga, J. Phys. Chem. **77**, 2300 (1973).

[2] I. M. Karataeva, E. E. Vinogradov, and S. V. Petrenko, Zh. Neorg. Khim. **32**, 1230 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 747 (1987).

Components:

(1) Mercury(II) bromate; $\mathrm{Hg(BrO_3)_2}$; [15384-08-4]
(2) Nitric acid; HNO_3; [7697-37-2]
or (2) Perchloric acid; $HClO_4$; [7601-90-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

G. F. Smith, J. Am. Chem. Soc. **46**, 1577–83 (1924).

Variables:

Concentration of HNO_3 or $HClO_3$
One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 60. Solubility of $\mathrm{Hg(BrO_3)_2}$ in nitric acid and perchloric acid solutions at 25.0 °C

Acid	c_2 / mol dm^{-3} [a]	γ_1 / (g / 100 cm^3) [b]	10^2c_1 / mol dm^{-3} [c]
HNO_3	2.0	14.66	3.21
	3.0	15.00	3.29
	4.0	14.99	3.28
	5.0	14.75	3.23
$HClO_3$	2.0	6.58	1.44
	2.5	5.22	1.14
	3.0	4.13	0.905
	3.5	3.40	0.745
	4.0	2.58	0.565
	5.0	1.55	0.340

[a] Approximate concentration of acids.

[b] In the original paper the solubility was given as g of solute per 100 cc of the solution. γ is the IUPAC recommended symbol. Mass concentration $\gamma_1 = m_1/V$, where m_1 is mass of component 1 and V is volume of the solution.

[c] Calculated by the compilers.

Mercury(II) bromate was found to be slightly decomposed at 130–140 °C and deflagrated at 155–160 °C, thus forming mercury(II) bromide as the major product.

The solubility of mercury(II) bromate in pure water was not reported. The solubility of the bromate decreases with the increase of perchloric acid concentration. In the case of nitric acid such tendency is not described. The addition of acids reverses a hydrolysis of the bromate and probably preserves a formation of $HgBrO_3(OH)$. The solubility data may be designated as tentative.

Auxiliary Information

Method/Apparatus/Procedure:

An excess of the solute was added to the various acid solutions. Then the mixtures were sealed in solubility tubes and rotated during 6 to 15 h at the selected temperature. The bromate content was determined by adding nitric or perchloric acid, an excess of potassium iodide, and then iodine liberated was titrated using sodium thiosulfate with starch as an indicator. All determinations were carried out in duplicate.

Source and Purity of Materials:

A hot perchloric acid solution (3 mol dm^{-3}) nearly saturated with sodium bromate was treated with an equivalent amount of mercury(II) perchlorate dissolved in perchloric acid solution. After cooling to 0 °C, the crystals obtained were filtered off and washed with several portions of water, using centrifugal drainage. The resulting mercury(II) bromate dihydrate was dehydrated by storage over anhydrous calcium chloride. The anhydrous salt was used for the solubility measurements.

Estimated Error:

Solubility: precision no better than ± 0.2 % (compilers).
Temperature: precision ± 0.1 K.

Components:

(1) Mercury(II) bromate hydroxide; $HgBrO_3(OH)$; [11092-91-4]
(2) Nitric acid; HNO_3; [7697-37-2]
or (2) Perchloric acid; $HClO_4$; [7601-90-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

G. F. Smith, J. Am. Chem. Soc. **46**, 1577–83 (1924).

Variables:

Concentration of HNO_3 and $HClO_4$
One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

TABLE 61. Solubility of $HgBrO_3(OH)$ in nitric acid and perchloric acid solutions at 25.0 °C

Acid	c_2 / mol dm^{-3} [a]	γ_1 / (g / 100 cm^3) [b]	10^3c_1 / mol dm^{-3} [c]
None	—	0.081	0.023
HNO_3	1.0	12.52	3.63
	2.0	21.30	6.16
$HClO_4$	1.0	4.34	1.26
	2.0	5.94	1.72
	2.5	6.06	1.75
	3.0	6.06	1.75

[a] Approximate concentration of acids.

[b] In the original paper the solubility was given as g of solute per 100 cm^3 of the solution. γ is the IUPAC recommended symbol. Mass concentration $\gamma_1 = m_1/V$, where m_1 is mass of component 1. V is volume of the solution.

[c] Calculated by the compilers.

The solubility data may be designated as tentative.

The mixed salt was found to be completely volatile when heated with a very mild deflagration at 190–200 °C.

Auxiliary Information

Method/Apparatus/Procedure:

An excess of solute was added to the various acid solutions which were then sealed in solubility tubes and rotated during 6 to 15 h at the selected temperature. The bromate content was determined by adding nitric acid or perchloric acid, excess of potassium iodide, and then iodine liberated was titrated using sodium thiosulfate with starch as an indicator. Mercury content was determined by titration with potassium thiocyanate using ammonium iron(III) sulfate as an indicator. All determinations were carried out in duplicate.

Source and Purity of Materials:

Mercury(II) perchlorate was dissolved in a small volume of water and added slowly with constant stirring to a hot, half saturated solution of sodium bromate. The solution and crystals were cooled to room temperature and decanted. The crystals were filtered and washed with water using centrifugal drainage. The bromate hydroxide salt was recrystallized from 3 mol dm^{-3} perchloric acid solution. The recrystallized salt was anhydrous. Analysis of the product: 23.11 % Br and 57.12 %, Hg, calculated for $HgBrO_3(OH)$: 23.13 % Br and 58.06 % Hg.

Estimated Error:

Solubility: precision no better than ± 0.2 % (compilers).

Temperature: precision ± 0.1 K.

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. March 2002.

Critical Evaluation:

The Binary System

Data for the solubility of thallium bromate in water have been reported in 7 publications (1–7). Only one value based on mol kg^{-1} units was reported (2), and other solubility data were reported in mol dm^{-3} units (1, 3–7). Experimental data of thallium bromate solubility and techniques used are summarized in Table 62 and shown in Fig. 9. One may assume that the anhydrous salt is the equilibrium solid phase as it could be deduced from (3) but detailed investigation in this matter was not performed elsewhere.

TABLE 62. Summary for solubility studies of $TlBrO_3$ in water

T / K	$10^2 c_1$ / mol dm^{-3}	Analytical method	Reference
293	0.98	Densimetry	(6)
293	0.995	Iodometry	(3)
293.1	0.922	Conductometry	(1)
298	1.215[a]	Iodometry	(2)
303	1.489	Volumetry	(4)
303	1.489	Gravimetry	(5)
308	1.822	Volumetry	(4)
312.9	2.216	Gravimetry	(7)
313	2.206	Volumetry	(4)
318	2.624	Volumetry	(4)

[a] mol kg^{-1} units.

The solubility data given in Table 62 were used to fit to the smoothing equation (5) given in the Preface in this volume. The detailed information on the calculation procedure for the smoothing equation has been described therein. The obtained smoothing equation is shown below, and the calculated curves are plotted in the Fig. 9. The recommended solubility values at round temperature calculated from this equation are given in Table 63.

$$\ln\left(\frac{c_1}{\text{mol dm}^{-3}}\right) = \frac{241112.621}{(T/\text{K})} + 1624.30289\ln(T/\text{K}) - 9265.11136 - 2.6920175(T/\text{K}), \quad (48)$$

As the results of the calculation, $\sigma_c = 0.027 \times 10^{-2}$ was obtained.

TABLE 63. Recommended solubility of $TlBrO_3$ in water

T / K	$10^2 c_1$ / mol dm^{-3}
293	0.96
298	1.20
303	1.49
308	1.83
313	2.21
318	2.62

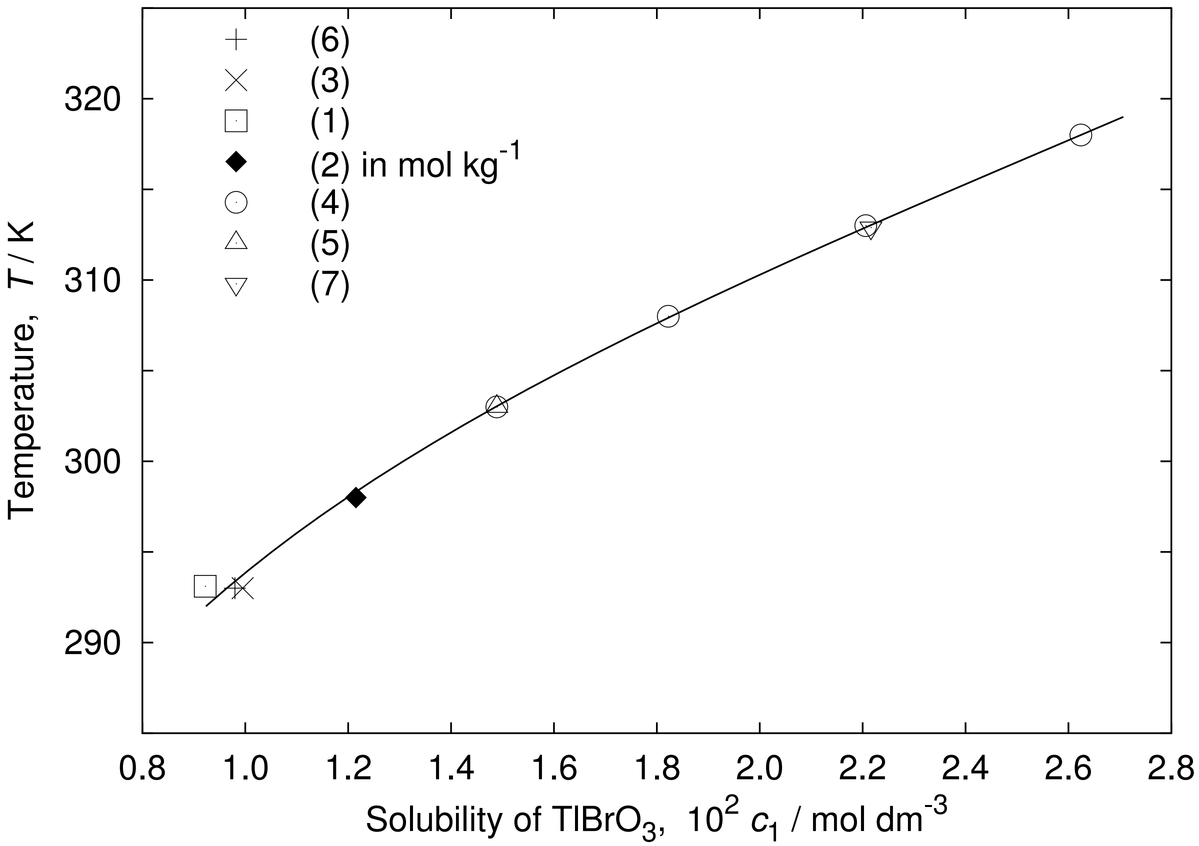

FIG. 9. Temperature dependence of solubility of $TlBrO_3$ in water

The Ternary System

Experimental data of thallium bromate in aqueous inorganic salt solutions are summarized in Table 64. The solubility of thallium bromate decreases with an increase of the added salt concentration in three systems, $TlBrO_3$–Tl_2SO_4–H_2O, $TlBrO_3$–$NaBrO_3$–H_2O, and $TlBrO_3$–$KBrO_3$–H_2O, as it is shown in Table 64. In other systems, the solubility increases with increasing the concentration of added salt. Khoo (4) stated that thallium bromate associates in aqueous solutions of sodium bromate or perchlorate and of magnesium perchlorate. Khoo (5) measured the solubilities of thallium bromate in various nitrate solutions, and then the association constant of thallium nitrate has been determined.

TABLE 64. Experimental data of $TlBrO_3$ solubility in aqueous inorganic salt solutions

Ternary system	T / K	c_2 / mol dm^{-3}	10^2c_1 / mol dm^{-3}	Reference
$TlBrO_3$–$LiNO_3$–H_2O	303	0–0.06000	1.489–1.746	(5)
$TlBrO_3$–$NaNO_3$–H_2O	303	0–0.06000	1.489–1.760	(5)
$TlBrO_3$–KNO_3–H_2O	298	0–1.0^a	1.215–2.778^a	(2)
$TlBrO_3$–KNO_3–H_2O	303	0–0.06000	1.489–1.773	(5)
$TlBrO_3$–$CsNO_3$–H_2O	303	0–0.06000	1.489–1.794	(5)
$TlBrO_3$–$Mg(NO_3)_2$–H_2O	303	0–0.0120	1.489–1.662	(5)
$TlBrO_3$–$Ba(NO_3)_2$–H_2O	298	0–0.2^a	1.215–2.140^a	(2)

$TlBrO_3$–$TlNO_3$–H_2O	298	0–0.2[a]	1.215–0.151[a]	(2)
$TlBrO_3$–K_2SO_4–H_2O	298	0–0.5[a]	1.215–2.955[a]	(2)
$TlBrO_3$–Tl_2SO_4–H_2O	298	0–0.1[a]	1.215–0.204[a]	(2)
$TlBrO_3$–$NaClO_4$–H_2O	303	0–0.06010	1.489–1.718	(4)
$TlBrO_3$–$NaClO_4$–H_2O	308	0–0.06016	1.822–2.072	(4)
$TlBrO_3$–$NaClO_4$–H_2O	313	0–0.06006	2.206–2.484	(4)
$TlBrO_3$–$NaClO_4$–H_2O	318	0–0.06027	2.624–2.953	(4)
$TlBrO_3$–$Mg(ClO_4)_2$–H_2O	303	0–0.02485	1.489–1.730	(4)
$TlBrO_3$–$Mg(ClO_4)_2$–H_2O	308	0–0.02336	1.822–2.072	(4)
$TlBrO_3$–$Mg(ClO_4)_2$–H_2O	313	0–0.02311	2.206–2.486	(4)
$TlBrO_3$–$Mg(ClO_4)_2$–H_2O	318	0–0.01867	2.624–2.915	(4)
$TlBrO_3$–$NaBrO_3$–H_2O	303	0–0.09013	1.489–0.345	(4)
$TlBrO_3$–$NaBrO_3$–H_2O	308	0–0.09000	1.822–0.494	(4)
$TlBrO_3$–$NaBrO_3$–H_2O	313	0–0.08996	2.206–0.679	(4)
$TlBrO_3$–$NaBrO_3$–H_2O	318	0–0.08996	2.624–0.930	(4)
$TlBrO_3$–$KBrO_3$–H_2O	298	0–0.2	1.215–0.138	(2)

[a] mol kg^{-1} units

Gross and Kahn (3) measured the solubilities in ethanol– and acetone–water mixed solvents at 293 K. In these systems, the solubility of thallium bromate decreases with increasing the concentration of the organic solvents. The solubilities of thallium bromate in aqueous solutions of urea have been reported by Gross and Kahn (3) and Gyunner and Fedorenko (6). In both studies, an increase of the urea concentration resulted in an increase of the solubility of thallium bromate. The tendency of the thallium bromate solubility changes in aqueous glycine solution (6) is similar to that observed for the urea–water system.

References:

1. W. Böttger, Z. Phys. Chem. **46**, 521 (1903).
2. S. Suzuki, Nippon Kagaku Zasshi **73**, 56 (1952).
3. P. Gross and F. Kahn, Monatsh. Chem. **86**, 371 (1955).
4. K. H. Khoo, J. Inorg. Nucl. Chem. **30**, 2425 (1968).
5. K. H. Khoo and J. D. Murray, J. Inorg. Nucl. Chem. **31**, 2437 (1969).
6. E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1797 (1974); Russ. J. Inorg. Chem. (Engl. Tramsl.) **19**, 979 (1974).
7. A. A. Noyes and C. G. Abbot, Z. Phys. Chem. **16**, 125 (1895).

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Thallium chloride; TlCl; [7791-12-0]
or (2) Thallium thiocyanide; TlSCN; [3535-84-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

A. A. Noyes and C. G. Abbot, Z. Phys. Chem. **16**, 125–38 (1895).

Variables:

Salt additives: TlCl or TlSCN
One temperature: 312.90 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 65. Solubility of $TlBrO_3$ in water and in presence of TlCl or TlSCN precipitates at 39.75 °C

Added salt	10^2c_1 / mol dm^{-3}
None	2.216 ± 0.0025
TlCl	1.567 ± 0.0015
TlSCN	1.496 ± 0.0018

Degree of $TlBrO_3$ dissociation was found to be 90.2 ± 0.4 % (in the solubility measurements) and 89.0 ± 0.1 % (in electric conductance measurements).

Auxiliary Information

Method/Apparatus/Procedure:

The saturated solutions were prepared by addition of $TlBrO_3$ to water. A bottle containing the mixture was rotated for 3–4 h in a bath at the selected temperature. The saturated solutions were filtered and the filtrate analyzed. Bromate was transferred to bromide by addition of H_2SO_4 and HNO_3. The resulting bromide content was determined by a titration with a standard $AgNO_3$ solution to form AgBr precipitate. Thiocyanides and chlorides in presence of bromates were determined by similar titration of hot and further diluted solutions with $AgNO_3$. Every determination was repeated 6 times.

Source and Purity of Materials:

Metallic Tl was dissolved in concentrated H_2SO_4. The resulting Tl_2SO_4 was recrystallized after filtration. Solutions of Tl_2SO_4 and $KBrO_3$ were mixed in the proper ratio. The product was freed from bromate excess by recrystallization in hot water. Purity of $TlBrO_3$ was analyzed. Calculated Br content was 24.7 % and found by analysis 24.10 % or 24.11 %.

Estimated Error:

Solubility: precision ± 0.1 % (given as the standard deviation).
Temperature: precision ± 0.03 K.

Components:
(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
W. Böttger, Z. Phys. Chem. **46**, 521–619 (1903).

Variables:
One temperature: 293.09 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $TlBrO_3$ in water at 19.94 °C was determined by the electrolytic conductivity method. The electrolytic conductivity of saturated solution of thallium bromate was given in Table 66.

TABLE 66. Solubility of $TlBrO_3$ in water at 19.94 °C

Equilibration time / h	$10^6\kappa$ / S cm^{-1}	10^3c_1 / mol dm^{-3}	10^5K_s / mol^2 dm^{-6}
7	1081		
7	1080		
12	1081		
14	1082		
16	1080		
17	1079		
	(av.) 1080	(av.) 9.22	(av.) 8.50

The molar conductances (λ^{∞}) of 68.8 and 48.3 S cm^2 mol^{-1} for Tl$^+$ and BrO$_3^-$, respectively, and the average (1080×10^{-6} S cm^{-1}) of the conductivity of κ were used to calculate the solubility from the following equations:

$$\kappa = \kappa_{\text{(solution)}} - \kappa_{\text{(water)}} \tag{49}$$

$$c_1 = \frac{1000\,\kappa}{\lambda^{\infty}_{\text{Tl}^+} + \lambda^{\infty}_{\text{BrO}_3^-}} \tag{50}$$

$\kappa_{\text{(water)}}$ was found to be 1.03×10^{-6} S cm^{-1}. The solubility value of 9.22×10^{-3} mol dm^{-3} and K_{s} = 8.50×10^{-5} mol^2 dm^{-6} were calculated. No equilibrium solid phase is specified.

Degree of the salt dissociation was found to be 92.7%. Effect of crystal dimensions on the solubility was found to be in the range of 1–2%.

(Erratum: In Table 43 on p. 602 of Böttger's paper, $10^6\kappa$ / S cm^{-1} = 108.0 should be read as $10^6\kappa$ / S cm^{-1} = 1080.)

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of TlBrO$_3$ in water was determined from measurements of electrolytic conductivities of the saturated solution and of pure water. The conductivity cell, which employed Pt electrodes, were designed to prevent entrapment of air bubbles. After sealing, the entire cell was immersed in a water bath and rotated in the dark for 7–17 h. Equilibrium was reached when the conductivity remained constant within the experimental error (± 0.1 %). The conductance bridge and the thermometer were calibrated. The cell constant was determined with 0.010 mol dm^{-3} KCl solution.

Source and Purity of Materials:

Salts (from Merck) were recrystallized several times. Water was twice distilled. Thallium bromate was prepared from potassium bromate and thallium nitrate.

Estimated Error:

Solubility: precision ± 0.2 %.

Temperature: stability ± 0.1 K; accuracy ± 0.02 K.

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Barium nitrate; $Ba(NO_3)_2$; [10022-31-8]
or (2) Thallium nitrate; $TlNO_3$; [10102-45-1]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
or (2) Thallium sulfate; Tl_2SO_4; [7446-18-6]
or (2) Potassium bromate; $KBrO_3$; [7758-01-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

S. Suzuki, Nippon Kagaku Zasshi **73**, 56–8 (1952).

Variables:

Concentration of added salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 67. Solubility of $TlBrO_3$ in various inorganic salt solutions at 25 °C

Added salt	m_2 / mol kg^{-1}	$10^2 m_1$ / mol kg^{-1}
None	—	1.215
KNO_3	0.01	1.270
	0.02	1.315
	0.05	1.410
	0.1	1.540
	0.2	1.738
	0.5	2.138
	1.0	2.778
$Ba(NO_3)_2$	0.01	1.368
	0.02	1.448
	0.05	1.658
	0.1	1.800
	0.2	2.140
$TlNO_3$	0.01	0.849_1
	0.02	0.641_0
	0.05	0.366_5
	0.1	0.232_7
	0.2	0.151_9
K_2SO_4	0.01	1.366
	0.02	1.472
	0.05	1.688
	0.1	1.948
	0.2	2.280

	0.5	2.955
Tl_2SO_4	0.01	0.685
	0.02	0.476
	0.05	0.276
	0.1	0.204
$KBrO_3$	0.01	0.845_4
	0.02	0.640_1
	0.05	0.364_1
	0.1	0.227_8
	0.2	0.138_5

The solid phase composition was not specified.

Auxiliary Information

Method/Apparatus/Procedure:

The details of equilibration were not reported. Analysis of thallium bromate in added salt solutions except $KBrO_3$ was performed iodometrically. An excess KI and HCl were added to thallium bromate solution. The liberated iodine was determined using sodium thiosulfate solution being preliminary standardized. The solubility of $TlBrO_3$ in $KBrO_3$ solution was determined by gravimetric analysis as TlI.

Source and Purity of Materials:

Thallium bromate was prepared by adding equimolar amounts of solutions of potassium bromate and thallium sulfate to large volume water at 60 °C. The solution was cooled, the precipitate filtered off and dried at 80 °C. All reagents were analytically pure (from Kahlbaum) and recrystallized.

Estimated Error:

Solubility: precision ± 0.1 % (compiler).
Temperature: nothing specified.

Components:
(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Ethanol; C_2H_6O; [64-17-5]
or (2) 2-Propanone (acetone); C_3H_6O; [67-64-1]
or (2) Urea; CH_4N_2O; [57-13-6]
or (2) Glycine (aminoacetic acid); $C_2H_5NO_2$; [56-40-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
P. Gross and F. Kahn, Monatsh. Chem. **86**, 371–8 (1955).

Variables:
Concentration of organic solvents
One temperature: 293 K

Prepared By:
Hiroshi Miyamoto and Ryo Miyamoto

Experimental Data

TABLE 68. Solubility of $TlBrO_3$ in water–organic solvent mixtures at 20 °C

Organic solvent	m_2 / mol kg^{-1}	$10^3 c_1$ / mol dm^{-3}
None	—	9.951
Ethanol	0.086	9.767
	0.173	9.588
	0.341	9.253
Acetone	0.146	9.681
	0.297	9.384
	0.838	8.388
Urea	0.100	10.10
	0.201	10.23
	0.301	10.38
Glycine	0.0322	10.07
	0.0642	10.22
	0.0963	10.38

The equilibrium solid phases were not analyzed. One may assume that it was anhydrous salt in every case as it was observed in case of water.

Auxiliary Information

Method/Apparatus/Procedure:

Thallium bromate and solvent mixtures were weighed, and placed in a thermostat. Equilibrium was established for about 3 h. The saturated solution was withdrawn with a pipet. The solubility of the bromate was determined iodometrically. The saturated solution containing acetone was heated to remove acetone from the solution before the analysis was performed.

Source and Purity of Materials:

Thallium bromate was prepared by mixing relatively concentrated solutions of thallium nitrate and potassium bromate (analytically pure salts from Kahlbaum or Merck). The purity of the product was 99.5 %. Ethanol was dehydrated over CaO, dried with aluminumethylate, and distilled. Acetone was purified over hydrogen sulfite compound, dried over calcium chloride, and then fractionally distilled. Urea (from Kahlbaum) was twice recrystallized from water. Glycine (from Kahlbaum) was dissolved in water, and by adding alcohol the crystals of glycine were precipitated from the solution.

Estimated Error:

Solubility: precision ± 0.02 %.
Temperature: precision ± 0.01 K.

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]
or (2) Magnesium perchlorate; $Mg(ClO_4)_2$; [10034-81-8]
or (2) Sodium bromate; $NaBrO_3$; [7789-38-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

K. H. Khoo, J. Inorg. Nucl. Chem. **30**, 2425–30 (1968).

Variables:

Concentration of $NaClO_4$, $Mg(ClO_4)_2$ and $NaBrO_3$
Temperature: 303–318 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 69. Solubility of $TlBrO_3$ in aqueous solutions of $NaClO_4$, $Mg(ClO_4)_2$ and $NaBrO_3$ at various temperatures

Added salt	t / °C	10^3c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
$NaClO_4$	30	0	14.89
		10.01	15.50
		20.03	15.92
		30.05	16.29
		40.06	16.62
		50.08	16.91
		60.10	17.18
	35	0	18.22
		10.03	18.83
		20.05	19.31
		30.08	19.74
		40.10	20.05
		50.14	20.41
		60.16	20.72
	40	0	22.06
		10.01	22.70
		20.02	23.20
		30.03	23.67
		40.04	24.08
		50.05	24.55
		60.06	24.84
	45	0	26.24
		10.05	27.09
		20.09	27.64
		30.14	28.22

		40.18	28.61
		50.24	29.15
		60.27	29.53
$Mg(ClO_4)_2$	30	4.14	15.55
		8.28	16.07
		12.41	16.38
		16.55	16.74
		20.71	17.02
		24.85	17.30
	35	3.89	18.88
		7.78	19.43
		11.67	19.75
		15.58	20.18
		19.45	20.54
		23.36	20.72
	40	3.85	22.72
		7.70	23.26
		11.54	23.76
		15.41	24.11
		19.24	24.56
		23.11	24.86
	45	3.73	27.04
		7.47	27.66
		11.20	28.24
		14.94	28.66
		18.67	29.15
$NaBrO_3$	30	10.01	11.06
		20.03	8.60
		30.05	6.96
		50.08	5.09
		70.11	4.08
		90.13	3.45
	35	10.00	14.24
		20.00	11.45
		30.00	9.55
		50.00	7.11
		70.00	5.77
		90.00	4.94
	40	10.00	17.99
		19.99	14.95
		30.00	12.69
		49.98	9.76
		69.99	8.01
		89.96	6.79
	45	10.00	22.24

	19.99	18.98
	29.99	16.55
	49.98	13.05
	69.99	10.84
	89.96	9.30

Auxiliary Information

Method/Apparatus/Procedure:

The method obtaining the saturated solution was the same as that used by Macaskill and Panckhurst (1). The saturated solution was analyzed for thallium. 4 cm^3 of concentrated HCl solution were added to 10 cm^3 of the saturated solution. The solution was warmed and sodium sulfite was added, and then boiled for about 2 min. After cooling 10 cm^3 of concentrated HCl solution and 1 cm^3 of carbon tetrachloride were added and the resulting solution was titrated with iodate solution using a microburet. The mean of 6 to 8 determinations was taken.

Source and Purity of Materials:

Thallium bromate was precipitated by adding solutions of thallium sulfate (Analar) and sodium bromate (B. D. H. laboratory grade) to twice the total volume of distilled water at 70 °C. The precipitate was washed with water several times and dried in a vacuum desiccator at room temperature. Sodium perchlorate (from Hopkin and William G. P. R.) was used without further purification. Magnesium perchlorate was prepared by neutralizing 60 % perchloric acid (Analar) with magnesium carbonate (Merck, for analysis). Excess carbonate was removed by filtration and the solution was evaporated to near dryness. The product was recrystallized twice from distilled water and dried in a vacuum desiccator. A stock solution of magnesium perchlorate was prepared from approximately known weight of the undehydrated salt and then standardized by complexometric titration. Sodium bromate (B.D.H. laboratory grade) was used without further purification. Distilled water was used.

Estimated Error:

Solubility: precision ± 0.25 %.
Temperature: stability ± 0.02 K.

References:

[1] J. B. Macaskill and M. H. Panckhurst, Aust. J. Chem. **17**, 522 (1964).

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Lithium nitrate; $LiNO_3$; [7790-69-4]
or (2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Cesium nitrate; $CsNO_3$; [7789-18-6]
or (2) Magnesium nitrate; $Mg(NO_3)_2$; [10377-60-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

K. H. Khoo and J. D. Murray, J. Inorg. Nucl. Chem. **31**, 2437–43 (1969).

Variables:

Concentration of salts
One temperature: 303 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 70. Solubility of $TlBrO_3$ in aqueous solutions of $LiNO_3$, $NaNO_3$, KNO_3, $CsNO_3$, and $Mg(NO_3)_2$ at 30 °C

Added salt	10^3c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
None	—	14.89
$LiNO_3$	10.00	15.57
	20.00	16.06
	30.00	16.48
	40.00	16.83
	50.00	17.18
	60.00	17.46
$NaNO_3$	10.00	15.60
	20.00	16.11
	30.00	16.55
	40.00	16.92
	50.00	17.28
	60.00	17.60
KNO_3	10.00	15.63
	20.00	16.17
	30.00	16.63
	40.00	17.03
	50.00	17.38
	60.00	17.73
$CsNO_3$	10.00	15.68
	20.00	16.26
	30.00	16.77
	40.00	17.22

	50.00	17.58
	60.00	17.94
$Mg(NO_3)_2$	2.00	15.36
	4.00	15.69
	6.00	15.95
	8.00	16.20
	10.00	16.42
	12.00	16.62

Auxiliary Information

Method/Apparatus/Procedure:

The method of obtaining the saturated solution was the same as used by Macaskill and Panckhurst (1). The analysis of the saturated solution for thallium content was performed gravimetrically. 8 cm^3 of concentrated HCl and 5 cm^3 of water were added to 20 cm^3 of the saturated solution. The solution was warmed to about 50 °C and a slight excess of the saturated solution of recrystallized $[Co(NH_3)_6]Cl_3$ was added. The suspension was digested for 5 min, cooled in ice, and precipitate of $[Co(NH_3)_6]TlCl_6$ was transferred to a weighed sintered-glass crucible, thoroughly washed with 2 % HCl solution and dried to constant weight at 105 °C. Minimum 8 determinations were carried out.

Source and Purity of Materials:

Thallium bromate was prepared by adding solutions of thallium sulfate (Analar) and sodium bromate (B. D. H. Laboratory grade) to twice the total volume of distilled water at 70 °C. The precipitate was washed with water several times and dried in a vacuum desiccator at room temperature. Lithium nitrate (Hopkin and William G. P. R.) was used without further purification, but dried at 180 °C before preparation of the stock solution. Sodium and potassium nitrates were of Analar grade; they were dried at 100 °C before preparation of stock solutions. Cesium nitrate solution was prepared by mixing stoichiometric amount of cesium chloride and silver nitrate. The filtrate was evaporated, the product recrystallized twice from water and dried at 100 °C. A stock solution of magnesium nitrate (Analar) was prepared from an approximately known weight of hydrated salt and then standardized by a complexometric titration.

Estimated Error:

Solubility: precision ± 0.25 %; standard deviation of determinations $<$ 0.2 %.
Temperature: stability ± 0.02 K.

References:

[1] J. B. Macaskill and M. H. Panckhurst, Aust. J. Chem. **17**, 522 (1964).

Components:

(1) Thallium bromate; $TlBrO_3$; [14550-84-6]
(2) Urea; CH_4N_2O; [57-13-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1797–9 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 979–80 (1974).

Variables:

Concentration of urea: 0–3.0 mol dm^{-3}
One temperature: 293 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 71. Solubility of $TlBrO_3$ in aqueous urea solutions at 20 °C

c_2 / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}
0	9.8
0.300	10.35
0.600	10.96
0.900	11.20
1.200	11.77
1.500	12.29
1.800	12.75
2.100	13.24
2.400	13.62
2.700	14.00
3.000	14.95

The solubility product of $TlBrO_3$ was 9.51×10^{-5} mol^2 dm^{-6}.

Equilibrium constant

$$K = \frac{[\mathrm{Tl(NH_2)_2CO^+}]}{[\mathrm{Tl^+}][\mathrm{(NH_2)_2CO}]}$$

was calculated to be 0.410 ± 0.035 dm^3 mol^{-1}.

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:

The initial urea solutions were saturated with $TlBrO_3$ at the selected temperature. The solubility of $TlBrO_3$ was estimated by pycnometric method (1), but any details in the quoted paper were also not specified.

Source and Purity of Materials:

No information was reported.

Estimated Error:

Solubility: the error did not exceed $\pm 4 \times 10^{-5}$ mol dm^{-3}.

Temperature: precision ± 0.05 K.

References:

[1] E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **15**, 1430 (1970); Russ. J. Inorg. Chem. (Engl. Transl.) **15**, 734 (1970).

Components:
(1) Lead bromate; $\mathrm{Pb(BrO_3)_2}$; [34018-28-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. April 2002.

Critical Evaluation:

The Binary System

Data for the solubilities of lead bromate in water have been reported in 3 publications (1–3). Böttger (1) determined the solubility by the conductivity measurement at 293.1 K, MacDougall and Hoffman (2) measured the solubility by iodometric titration at 298.15 K, and Fedorov et al. (3) measured the solubility by amperometric titration and potentiometry at 278–338 K. The $\mathrm{Pb(BrO_3)_2}$ solubility values in water in (1) and (2) were 2.05×10^{-2} mol dm^{-3} at 293.1 K and 3.44×10^{-2} mol dm^{-3} at 298.15 K, respectively. Fedorov et al. (3) observed a smaller increase of the solubility with temperature than in a comparison of the results of (1) and (2). Moreover, the solubility value of 3.49×10^{-2} mol dm^{-3} in 3×10^{-2} mol dm^{-3} $LiClO_3$ solution by Fedorov et al. (3) at 298 K is very similar to that of (2). Therefore, the result reported by MacDougall and Hoffman (2) seem to be more reliable and may be treated as a tentative one.

The Ternary System

MacDougall and Hoffman (2) measured the bromate solubilities in aqueous solutions of 6 nitrates. The solubility of lead bromate in aqueous solutions of alkali and alkaline earth metal nitrates increases with increasing the nitrate concentration. In the case of aqueous lead nitrate solution, a dependence of the solubility *vs.* the concentration of the lead nitrate have a minimum at about 0.1 mol dm^{-3} $\mathrm{Pb(NO_3)_2}$ what is probably connected with complexation of lead by nitrate ions.

The Multicomponent System

Data for the solubilities in the quarternary $\mathrm{Pb(BrO_3)_2}$–$LiNO_3$–$LiClO_3$–H_2O system have been reported by Fedorov et al. (3). Lithium nitrate and perchlorate were used to adjust the constant ionic strength at 3.0 mol dm^{-3}.

References:

1. W. Böttger, Z. Phys. Chem. **46**, 521 (1903).
2. F. H. MacDougall and E. J. Hoffman, J. Phys. Chem. **40**, 317 (1936).

3. V. A. Fedorov, A. M. Robov, I. D. Isaev, and V. E. Mironov, Uch. Zap. Leningr. Gos. Pedagog. Inst. im. A. I. Gertsena **385**, 101 (1969).

Components:

(1) Lead bromate; $\mathrm{Pb(BrO_3)_2}$; [34018-28-5]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

W. Böttger, Z. Phys. Chem. **46**, 521–619 (1903).

Variables:

One temperature: 293.09 K

Prepared By:

Hiroshi Miyamoto and Ceary Guminski

Experimental Data

The solubility of $\mathrm{Pb(BrO_3)_2}$ in water at 19.94 $^\circ\mathrm{C}$ was determined by electric conductivity method. The electrolytic conductivity of the saturated lead bromate solution was reported in Table 72.

TABLE 72. Solubility of $\mathrm{Pb(BrO_3)_2}$ in water at 19.94 $^\circ\mathrm{C}$

Equilibration time / h	$10^6\kappa_{\text{(satd. soln.)}}$ / S cm^{-1}	10^2c_1 / mol dm^{-3}	$10^{-5}K_s$ / mol^3 dm^{-9}
44	4599		
60	4620		
128	4603		
164	4699		
85	4689		
	(av.) 4635	(av.) 2.05	(av.) 3.47

The author reported $\kappa_{\text{(water)}} = 0.95 \times 10^{-6}$ S cm^{-1}, as in the case of the conductivity study of $\mathrm{Pb(IO_3)_2}$ solution in this paper. Therefore, the compilers assumed that the $\kappa_{\text{(water)}}$ value was negligibly small in comparison with the $\kappa_{\text{(satd. soln.)}}$ value. The molar conductances (λ^{∞}) of 64.5 and 48.3 S cm^2 mol^{-1} for $\frac{1}{2}$Pb^{2+} and BrO$_3^-$, respectively, and the average value (4635×10^{-6} S cm^{-1}) of $\kappa_{\text{(Pb(BrO}_3)_2)}$ were used to calculate the solubility from the following equation:

$$\kappa = \kappa_{\text{(satd. soln.)}} - \kappa_{\text{(water)}} \tag{51}$$

$$c_1 = \frac{1000\,\kappa}{2\left\{\lambda^{\infty}_{\frac{1}{2}\text{Pb}^{2+}} + \lambda^{\infty}_{\text{BrO}_3^-}\right\}} \tag{52}$$

The solubility value of 2.05×10^{-2} mol dm^{-3} was calculated by the compilers what is equivalent to the author's reported solubility of 4.11×10^{-2} g equiv. dm^{-3} and $K_{\text{s}} = 3.47 \times 10^{-5}$ mol^3 dm^{-9}. No equilibrium solid phase was specified. Degree of the salt dissociation was found to be 72 %. Effect of crystal dimension on the solubility was found to be 1–2 %.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of $\mathrm{Pb(BrO_3)_2}$ in water was determined from measurements of electrolytic conductivities of saturated solution and of pure water. The conductivity cells which employed Pt electrodes were designed to prevent entrapment of air bubbles. After sealing, the entire cell was immersed in a water bath and rotated in the dark for 44 to 164 min. Equilibrium was reached when the conductivity remained constant within experimental error (± 0.8 %). The conductance bridge and thermometer were calibrated. The cell constant was determined with 0.010 mol dm^{-3} KCl solution. The result of the solubility was confirmed by bromate determination: addition of KI, acidification and titration with sodium thiosulfate.

Source and Purity of Materials:

All salts (from Merck) were recrystallized several times. Water was twice distilled. Lead bromate was prepared from potassium bromate and lead nitrate.

Estimated Error:

Solubility: precision ± 1 %.

Temperature: stability ± 0.1 K; accuracy ± 0.02 K.

Components:
(1) Lead bromate; $Pb(BrO_3)_2$; [34018-28-5]
(2) Lithium nitrate; $LiNO_3$; [7790-69-4]
or (2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Calcium nitrate; $Ca(NO_3)_2$; [10124-37-5]
or (2) Strontium nitrate; $Sr(NO_3)_2$; [10042-76-9]
or (2) Lead nitrate; $Pb(NO_3)_2$; [10099-74-8]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. MacDougall and E. J. Hoffman, J. Phys. Chem. **40**, 317–31 (1936).

Variables:
Concentration of nitrates
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 73. Solubility of $Pb(BrO_3)_2$ in aqueous solutions of $LiNO_3$, $NaNO_3$, KNO_3, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Pb(NO_3)_2$ and their densities at 25.00 °C

Nitrate	c_2 / mol dm^{-3}	c_1 / mol dm^{-3}	ρ / kg dm^{-3}
None	—	0.03437	1.0112
$LiNO_3$	0.04925	0.03849	1.0135
	0.09873	0.04244	1.0158
	0.2049	0.04874	1.0244
	0.3012	0.05365	1.0293
	0.3933	0.05800	1.0348
	0.4929	0.06206	1.0399
	0.5815	0.06517	1.0446
	0.7751	0.07225	1.0549
	0.9644	0.07867	1.0649
	1.4259	0.09337	1.0880
	1.8680	0.1061	1.1084
	2.3032	0.1190	1.1302
	2.7213	0.1310	1.1507
	3.5192	0.1534	1.1887
	4.9703	0.1930	1.2562
$NaNO_3$	0.04833	0.03884	1.0141
	0.1013	0.04317	1.0184
	0.1991	0.04984	1.0266
	0.2963	0.05580	1.0334
	0.3938	0.06144	1.0424
	0.4892	0.06430	1.0465

	0.5874	0.07112	1.0564
	0.7776	0.08024	1.0697
	0.9655	0.08899	1.0837
	1.1970	0.09967	1.1000
	1.4241	0.1098	1.1158
	1.8655	0.1295	1.1460
	2.2921	0.1487	1.1754
	2.6860	0.1651	1.2010
	3.4672	0.1990	1.2530
	4.8546	0.2536	1.3378
	6.0912	0.3026	1.4172
	7.1770	0.3416	1.4824
KNO_3	0.04883	0.03924	1.0156
	0.09934	0.04378	1.0181
	0.1956	0.05104	1.0286
	0.2950	0.05798	1.0378
	0.3901	0.06396	1.0456
	0.4857	0.07003	1.0547
	0.5784	0.07577	1.0608
	0.7671	0.08734	1.0776
	0.9339	0.09818	1.0906
	1.1850	0.1134	1.1127
	1.4419	0.1293	1.1327
	1.8314	0.1550	1.1664
	2.0352	0.1685	1.1833
	2.2469	0.1782	1.2011
	2.4449	0.1799	1.2161
	2.6423	0.1732	1.2220
$Ca(NO_3)_2$	0.006271	0.03565	1.0099
	0.01116	0.03640	1.0117
	0.02156	0.03921	1.0135
	0.05162	0.04298	1.0195
	0.1215	0.05387	1.0313
	0.1975	0.06202	1.0425
	0.3352	0.07497	1.0643
	0.4920	0.08769	1.0869
	0.5764	0.09473	1.0997
	0.7780	0.1105	1.1283
	0.9524	0.1194	1.1457
	1.3805	0.1435	1.2090
	1.7872	0.1647	1.2607
	2.1719	0.1813	1.3074
	2.5361	0.1944	1.3489
	3.1950	0.2118	1.4215
$Sr(NO_3)_2$	0.005566	0.03581	1.0117

	0.01031	0.03702	1.0129
	0.01958	0.03893	1.0144
	0.05020	0.04455	1.0226
	0.09944	0.05180	1.0332
	0.1975	0.06339	1.0548
	0.2949	0.07301	1.0741
	0.3903	0.08177	1.0933
	0.4856	0.08939	1.1107
	0.5804	0.09739	1.1301
	0.7614	0.1110	1.1627
	0.9446	0.1249	1.1971
	1.3742	0.1535	1.2748
	1.7789	0.1765	1.3438
	2.1599	0.1955	1.4084
	2.5197	0.2105	1.4668
	2.8704	0.2228	1.5261
$Pb(NO_3)_2$	0.004947	0.03371	1.0104
	0.009921	0.03332	1.0118
	0.1990	0.03258	1.0132
	0.04981	0.03161	1.0219
	0.09940	0.03145	1.0356
	0.1978	0.03264	1.0643
	0.2955	0.03457	1.0936
	0.3882	0.03657	1.1120
	0.4871	0.03876	1.1480
	0.5804	0.04102	1.1735
	0.7672	0.04572	1.2290
	0.9470	0.05046	1.2786
	1.1663	0.05613	1.3402
	1.3807	0.06183	1.4011

The equilibrium solid phase was not analyzed, but may be assumed to be the anhydrous salt.

Auxiliary Information

Method/Apparatus/Procedure:

Water was added by means of an accurately calibrated pipet to glass-stoppered bottles containing previously weighed quantities of the nitrate and an excess of lead bromate. The bottles were mechanically rotated in a water-bath at the selected temperature for several hours until equilibrium was established. After equilibration, the bottles were settled. The clear solution was drawn off by means of vacuum into an Erlenmeyer flask. The delivery tube equipped with a small plug of absorbent cotton was used to transfer the solution from bottles to the flask. The sample solutions were withdrawn with calibrated pipet and these sample were accurately weighed to determine the densities of the solutions. The bromate content was determined by iodometric titration by using starch indicator containing salicylic acid as preservative. The titration was performed in quadruplicate.

Source and Purity of Materials:

Lead bromate was prepared by adding sodium bromate solution to lead nitrate solution at 30°C with constant stirring. The product was allowed to settle, and the solution was cooled. The supernatant liquid was decantated, and the lead bromate was washed several times by decantation, filtered, and washed with 95 % alcohol and absolute ether and dried in a vacuum desiccator over magnesium perchlorate at room temperature. Conductivity water was prepared by distillation water over sodium hydroxide and potassium permanganate. The nitrates were of the best quality, recrystallized once and some times twice from water, and then dehydrated thoroughly.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

Components:

(1) Lead bromate; $\mathrm{Pb(BrO_3)_2}$; [34018-28-5]
(2) Lithium nitrate; $LiNO_3$; [7790-69-4]
(3) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Fedorov, A. M. Robov, I. D. Isaev, and V. E. Mironov, Uch. Zap. Leningr. Gos. Pedagog. Inst. im. A. I. Gertsena **385**, 101–9 (1969).

Variables:

Concentration of $LiNO_3$ and $LiClO_3$: 0–3.0 mol dm^{-3}
One temperature: 278–338 K

Prepared By:

Cezary Guminski, E. V. Zagnit'ko, and Hiroshi Miyamoto

Experimental Data

TABLE 74. Solubility of $\mathrm{Pb(BrO_3)_2}$ in ($LiNO_3$ + $LiClO_4$) solution with the total salt concentration of 3.0 mol dm^{-3} and pH = 3

t / °C	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	c_1 / mol dm^{-3}	K_s / mol^3 dm^{-9} [a]	$10^5 K_\mathrm{s}^\circ$ / mol^3 dm^{-9} [b]
5	3.0	0	0.0209	3.65×10^{-5}	3.50
	2.7	0.3	0.0282		2.64
	2.4	0.6	0.0352		3.54
	2.1	0.9	0.0422		3.26
	1.9	1.1	0.0486		3.16
	1.5	1.5	0.0560		2.90
	1.2	1.8	0.0615		2.68
	0.9	2.1	0.0685		2.50
	0.6	2.4	0.0770		2.40
	0.3	2.7	0.0885		2.36
	0	3.0	0.0960		2.78
25	3.0	0	0.0349	1.70×10^{-4} [c]	15.3
	2.7	0.3	0.0436		12.1
	2.4	0.6	0.0525		15.0
	2.1	0.9	0.0625		15.5
	1.9	1.1	0.0715		13.1
	1.5	1.5	0.0825		12.2
	1.2	1.8	0.0915		11.5
	0.9	2.1	0.104		11.8
	0.6	2.4	0.115		11.4
	0.3	2.7	0.127		11.6
	0	3.0	0.139		11.5
45	3.0	0	0.0520	5.62×10^{-4}	42.3
	2.7	0.3	0.0650		54.8
	2.4	0.6	0.0800		41.3

	2.1	0.9	0.0925		37.6
	1.9	1.1	0.106		40.9
	1.5	1.5	0.119		36.3
	1.2	1.8	0.132		32.0
	0.9	2.1	0.148		33.8
	0.6	2.4	0.163		35.0
	0.3	2.7	0.178		38.7
	0	3.0	0.194		35.4
65	3.0	0	0.0765	1.79×10^{-3}	[d]
	2.7	0.3	0.0917		[d]
	2.4	0.6	0.111		[d]
	2.1	0.9	0.128		[d]
	1.9	1.1	0.147		[d]
	1.5	1.5	0.165		[d]
	1.2	1.8	0.187		[d]
	0.9	2.1	0.209		[d]
	0.6	2.4	0.225		[d]
	0.3	2.7	0.248		[d]
	0	3.0	0.269		[d]

[a] Determined from amperometric titration (Pb concentration). $K_\mathrm{s} = 4c_1^3$.

[b] Determined from potentiometric titration (Pb activity).

[c] Recalculated by the compilers. The reported value was 1.80×10^{-4} mol^3 dm^{-9}.

[d] No reliable results were obtained at this temperature.

The authors determined equilibrium constants of complexation of lead ions by nitrate ions; all constants of four step complexation decrease with temperature increase. The equilibrium solid phase was not investigated.

Auxiliary Information

Method/Apparatus/Procedure:

The total lead concentration in the saturated solutions was determined by amperometric titration with 0.05 mol dm^{-3} $Na_2Cr_2O_7$ solution in acetate buffer with pH = 4.2 as the background electrolyte. The mercury dropping electrode was used for lead ion detection. Equilibrium established was controlled analytically. Every experiment was repeated 3–4 times. Due to complexation of Pb^{2+} by NO_3^- the solubility product of $Pb(BrO_3)_2$ was determined also by potentiometry of a cell:

Pb(Hg) | 0.01 mol dm^{-3} $Pb(ClO_4)_2$, 0.001 mol dm^{-3} $HClO_4$, 3.0 mol dm^{-3} $LiClO_4$ | 3.0 mol dm^{-3} $LiClO_4$ | 3.0 mol dm^{-3} ($LiClO_4$ + $LiNO_3$), $Pb(BrO_3)_2$ satd. | Pb(Hg);

Pb(Hg) was the homogeneous amalgam dropping electrode. The solutions were freed from oxygen by stream of nitrogen.

Source and Purity of Materials:

All salts were analytically or chemically pure. They were twice or triple recrystallized from water. Content of lithium in the solutions was tested by gravimetric sulfate method. Purified nitrogen was used (1).

Estimated Error:

Solubility: precision ± 1 %.
Temperature: precision at best ± 0.1 K (authors of (1)).

References:

[1] V. A. Fedorov, A. M. Robov, and V. E. Mironov, Zh. Neorg. Khim. **12**, 3307 (1967).

A. Aluminum Iodate

Components:

(1) Aluminum iodate; $Al(IO_3)_3$; [15123-75-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Cezary Guminski, University of Warsaw, Warsaw, Poland, and Ryo Miyamoto, Hirosaki University, Hirosaki, Japan. September 2001.

Critical Evaluation:

The Binary System

The solubility of aluminum iodate in pure water at 298 K have been reported in 12 publications (1–12). The studies were performed by two investigator's groups of Shklovskaya et al. (1, 2, 4, 6–12) and Vinogradov and Tarasova (3, 5). All these studies deal with the ternary systems, and the binary system is regularly given as one point on a phase diagram. The value reported by the group of Shklovskaya et al., who made independent analyses, is 0.110 mol kg^{-1} at 298 K. This result is identical with that of Vinogradov and Tarasova, thus the value of 0.110 mol kg^{-1} reported by the two groups is designated as a recommended value at 298 K. The semi-quantitive estimation of von Endreedy (16) that the aluminum iodate is miscible with water in 1:1 ratio at 293 K seems to be erroneous is rejected. The stable solid in equilibrium with the saturated solution is $Al(IO_3)_3 \cdot 6H_2O$ [61355-88-2].

The Ternary System

Solubility studies for ternary aqueous systems with alkali metal iodates and iodic acid have been reported in five publications (1–4, 6), and are summarized in Table 75.

TABLE 75. Summary for solubility studies of $Al(IO_3)_3$ in the ternary systems with alkali metal halates or iodic acid at 298 K

Ternary system	Solid phase	Reference
$Al(IO_3)_3$–HIO_3–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; HIO_3; $Al(IO_3)_3 \cdot 2HIO_3 \cdot 6H_2O$	(2) compiled in (14) p. 483
$Al(IO_3)_3$–$LiIO_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $LiIO_3$	(1) compiled in (14) p. 298
$Al(IO_3)_3$–$NaIO_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $NaIO_3 \cdot H_2O$	(4) compiled in (14) p. 369
$Al(IO_3)_3$–KIO_3–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; KIO_3	(3) compiled in (14) p. 398
$Al(IO_3)_3$–$RbIO_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $RbIO_3$	(3) compiled in (14) p. 440
$Al(IO_3)_3$–$CsIO_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $CsIO_3$	(6) compiled in (14) p. 455

The dominant feature of the ternary system with iodic acid is the existence of double salt, $Al(IO_3)_3 \cdot 2HIO_3 \cdot 6H_2O$. The ternary systems with alkali metal iodates are of simple eutonic type, and no double salts were found. The reader finds the detailed compilations for these systems in (14).

Solubility studies for ternary aqueous systems with alkaline earth metal and other metal iodates have been reported in five publications (5, 7, 10–12). They are summarized in Table 76.

TABLE 76. Summary for solubility studies of $Al(IO_3)_3$ in the ternary systems with alkaline earth and other metal iodates at 298 K

Ternary system	Solid phase	Reference
$Al(IO_3)_3$–$Mg(IO_3)_2$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $Mg(IO_3)_2 \cdot 4H_2O$	(5) compiled in (15)
$Al(IO_3)_3$–$Ca(IO_3)_2$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $Ca(IO_3)_2 \cdot 6H_2O$	(10)
$Al(IO_3)_3$–$Ga(IO_3)_2$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $Ga(IO_3)_2 \cdot 2H_2O$	(7)
$Al(IO_3)_3$–$In(IO_3)_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; solid solution based on $In(IO_3)_3 \cdot H_2O$; $In(IO_3)_3 \cdot H_2O$	(11)
$Al(IO_3)_3$–$Nd(IO_3)_3$–H_2O	$Al(IO_3)_3 \cdot 6H_2O$; $Nd(IO_3)_3 \cdot 2H_2O$	(12)

The ternary systems given in Table 76 except the ternary system with indium iodate are of simple eutonic type and no double salts were found. The reader finds the detailed compilation for the ternary $Al(IO_3)_3$–$Mg(IO_3)_2$–H_2O system in (15), the rest of them is enclosed in this volume.

Solubility studies with aluminum nitrate and sulfate have been measured at 298 K by group of Shklovskaya et al. (8, 9). The dominant feature of the ternary systems with $Al(NO_3)_3$ or $Al_2(SO_4)_3$ is the existence of double salts: $Al(IO_3)_3 \cdot 2Al(NO_3)_3 \cdot 18H_2O$ or $Al(IO_3)_3 \cdot 2Al_2(SO_4)_3 \cdot 42H_2O$, respectively.

The Multicomponent System

Equilibria in the quaternary $Al(IO_3)_3$–$LiIO_3$–HIO_3–H_2O system at 298 K has been reported by Shklovskaya et al. (13, 14). The corresponding data sheet was prepared in (14). The following solid phases were found: α-$LiIO_3$, HIO_3, $Al(IO_3)_3 \cdot 2HIO_3 \cdot 6H_2O$ and solid solution of (H, Li)IO_3.

References:

1. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A. Kuzina, and G. V. Poleva, Zh. Neorg. Khim. **21**, 3116 (1976); Russ. J. Inorg. Chem. (Engl. Transl.) **21**, 1718 (1976).
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9. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, T. V. Daminova, and V. A. Kuzina, Zh. Neorg. Khim. **31**, 270 (1986); Russ. J. Inorg. Chem. (Engl. Transl.) **31**, 153 (1986).
10. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **32**, 246 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 140 (1987).

11. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, B. I. Kidyarov, V. A. Kuzina, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **33**, 219 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 122 (1988).

12. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, L. A. Kolobkova, and N. G. Dubovenko, Zh. Neorg. Khim. **33**, 3217 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 1859 (1988).

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14. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 298, 323, 369, 398, 455, 483.

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Components:
(1) Aluminum iodate; $\mathrm{Al(IO_3)_3}$; [15123-75-8]
(2) Gallium iodate; $\mathrm{Ga(IO_3)_3}$; [70504-12-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 219–21 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 122–3 (1985).

Variables:
Composition of the salts
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 77. Composition of saturated solutions in the ternary $\mathrm{Al(IO_3)_3}$–$\mathrm{Ga(IO_3)_3}$–H_2O system at 25 °C

$\mathrm{Ga(IO_3)_3}$		$\mathrm{Al(IO_3)_3}$		Solid phase[b]
$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	
0.85[c]	0.026	–	–	A
0.66	0.020	0.70	0.023	A
0.43	0.013	1.35	0.045	A
0.36	0.011	1.75	0.058	A
0.31	0.010	2.45	0.082	A
0.24	0.0075	2.93	0.099	A
0.22	0.0069	3.25	0.110	A
0.16	0.0051	4.05	0.138	A
0.14	0.0044	4.37	0.149	A
0.10	0.0032	5.01	0.172	A
0.052[d]	0.0017	5.47[d]	0.189	A+B
–	–	5.70[c]	0.197	B

[a] Calculated by the compiler.

[b] The solid phases are: A = $\mathrm{Ga(IO_3)_3 \cdot 2H_2O}$; B = $\mathrm{Al(IO_3)_3 \cdot 6H_2O}$.

[c] For binary systems the compiler computes the following values:

$$\text{Solubility of } \mathrm{Al(IO_3)_3} = 0.110 \text{ mol kg}^{-1},$$
$$\text{Solubility of } \mathrm{Ga(IO_3)_3} = 0.014 \text{ mol kg}^{-1}.$$

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 25–30 d. Samples were analyzed for the total gallium and aluminum content by a complexometric titration; for aluminum by back titration. Analysis of samples with less than 0.1 mass % aluminum and gallium was determined by spectrography and by atomic absorption spectroscopy, respectively. The solid phases were identified by the "residues" method with X-ray diffraction used as an additional check.

Source and Purity of Materials:

Aluminum iodate was prepared at 80–90 °C by neutralization of a saturated solution of iodic acid with an equivalent amount of freshly prepared aluminum hydroxide (1). Gallium iodate was prepared as follows (2): metallic gallium was dissolved in nitric acid and the theoretical amount of iodic acid added to the solution. The nitric acid was driven off by evaporation on a water-bath. The product was washed with water and carefully dried in a desiccator over anhydrous calcium chloride. Found: Ga–11.06 and I–60.84 mass %. Calcd. for $Ga(IO_3)_3 \cdot 2H_2O$: Ga–11.09 and I–60.38 mass %.

Estimated Error:

Solubility: nothing specified, but probably few percent.

Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A. Kuzina, and G. V. Poleva, Zh. Neorg. Khim. **21**, 3116 (1976); Russ. J. Inorg. Chem. (Engl. Transl.) **21**, 1718 (1976).

[2] P. Neogi and S. K. Nandi, J. Indian Chem. Soc. **14**, 492 (1937).

Components:

(1) Aluminum iodate; $\mathrm{Al(IO_3)_3}$; [15123-75-8]
(2) Aluminum nitrate; $\mathrm{Al(NO_3)_3}$; [13473-90-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, T. V. Daminova, and V. A. Kuzina, Zh. Neorg. Khim. **30**, 1857–9 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 1055–9 (1985).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 78. Composition of saturated solutions in the ternary $\mathrm{Al(IO_3)_3}$–$\mathrm{Al(NO_3)_3}$–H_2O system at 25 °C

$\mathrm{Al(IO_3)_3}$		$\mathrm{Al(NO_3)_3}$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
5.70[c]	0.197	–	–	A
7.20	0.262	3.51	0.331	A
7.38	0.288	9.92	1.001	A
7.51	0.306	13.84	1.46	A
7.42[d]	0.310	16.00[d]	1.73	A+B
5.95	0.247	17.18	1.85	B
4.55	0.191	19.61	2.14	B
3.82	0.164	22.05	2.45	B
3.42	0.151	24.84	2.84	B
3.27	0.148	27.19	3.20	B
2.25	0.111	34.29	4.37	B
1.28	0.064	36.80	4.78	B
0.83[e]	0.042	38.07[e]	5.00	B+C
–	–	39.25[c]	5.18	C

[a] Calculated by the compiler.

[b] The solid phases are: A = $\mathrm{Al(IO_3)_3 \cdot 6H_2O}$; B = $\mathrm{2Al(IO_3)_3 \cdot Al(NO_3)_3 \cdot 18H_2O}$; C = $\mathrm{Al(NO_3)_3 \cdot 9H_2O}$.

[c] For binary systems the compiler computes the following values:

$$\text{Solubility of } \mathrm{Al(IO_3)_3} = 0.110 \text{ mol kg}^{-1}$$
$$\text{Solubility of } \mathrm{Al(NO_3)_3} = 3.03 \text{ mol kg}^{-1}.$$

[d] Peritonic composition.

[e] Eutonic composition.

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 20–30 d. Samples of coexisting phases were analyzed for aluminum by titration with EDTA using xylenol orange as an indicator and for the iodate ion by an iodometric titration. The nitrate content was calculated by the difference. The solid phases were identified by the "residues" method and verified by X-ray diffraction.

Source and Purity of Materials:

Aluminum iodate hexahydrate was prepared from iodic acid and freshly prepared aluminum hydroxide (1). "Pure grade" $\mathrm{Al(NO_3)_3 \cdot 9H_2O}$ was recrystallized from water.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A. Kuzina, and G. V. Poleva, Zh. Neorg. Khim. **21**, 3116 (1976); Russ. J. Inorg. Chem. (Engl. Transl.) **21**, 1718 (1976).

Components:
(1) Aluminum iodate; $Al(IO_3)_3$; [15123-75-8]
(2) Aluminum sulfate; $Al_2(SO_4)_3$; [10043-01-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, T. V. Daminova and V. A. Kuzina, Zh. Neorg. Khim. **31**, 270–2 (1986); Russ. J. Inorg. Chem. (Engl. Transl.) **31**, 153–5 (1986).

Variables:
Composition of the salts
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 79. Composition of saturated solutions in the ternary $Al(IO_3)_3$–$Al_2(SO_4)_3$–H_2O system at 25 °C

$Al(IO_3)_3$		$Al_2(SO_4)_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
5.70[c]	0.197	—	—	A
8.04	0.299	4.56	0.273	A
8.69	0.339	8.37	0.527	A
8.58	0.348	11.79	0.771	A
8.54	0.359	14.92	1.012	A
8.52	0.377	18.92	1.35	A
8.46[d]	0.380	20.20[d]	1.46	A+B
5.78	0.253	21.03	1.49	B
3.47	0.153	23.65	1.68	B
3.15	0.140	25.02	1.80	B
2.72	0.123	26.61	1.94	B
2.22[e]	0.100	26.88[e]	1.96	B+C
1.32	0.059	27.31	1.97	C
—	—	27.65[c]	1.97	C

[a] Calculated by the compiler.

[b] The solid phases are: A = $Al(IO_3)_3 \cdot 6H_2O$; B = $Al(IO_3)_3 \cdot Al_2(SO_4)_3 \cdot 42H_2O$; C = $Al_2(SO_4)_3 \cdot 18H_2O$.

[c] For the binary system the compiler computes the following value:

$$\text{Solubility of } Al(IO_3)_3 = 0.110 \text{ mol kg}^{-1}.$$

[d] Peritonic composition.

[e] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was established in 20–30 d. Samples of coexisting phases were analyzed for aluminum content by titrating with EDTA and for iodate ion iodometrically. The quantity of sulfate ion was found by the difference. The solid phases were identified by the "residues" method, and the results were confirmed by X-ray diffraction.

Source and Purity of Materials:

Aluminum iodate was prepared at 80–90 $^\circ\mathrm{C}$ by neutralization of a saturated solution of iodic acid with an equivalent amount of freshly prepared aluminum hydroxide (1). The solution was cooled to room temperature and the solid dried. The hexahydrate salt was obtained. A "very pure" aluminum sulfate $\mathrm{Al_2(SO_4)_3 \cdot 18H_2O}$ was used.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.1 K.

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, V. A. Kuzina, and G. V. Poleva, Zh. Neorg. Khim. **21**, 3116 (1976); Russ. J. Inorg. Chem. (Engl. Transl.) **21**, 1718 (1976).

Components:

(1) Aluminum iodate; $Al(IO_3)_3$; [15123-75-8]

(2) Calcium iodate; $Ca(IO_3)_2$; [7789-80-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **32**, 246–7 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 140–1 (1987).

Variables:

Composition of the salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 80. Composition of saturated solutions in the ternary $Al(IO_3)_3$–$Ca(IO_3)_2$–H_2O system at 25 °C

$Al(IO_3)_3$		$Ca(IO_3)_2$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
–	–	0.33[c]	0.015	A
0.19	0.0062	0.21	0.0097	A
0.39	0.013	0.14	0.0065	A
0.68	0.022	0.05	0.0023	A
1.32	0.044	0.025	0.0012	A
2.40	0.080	0.016	0.00076	A
3.21	0.108	0.010	0.00048	A
4.04	0.136	0.0090	0.00043	A
4.77	0.163	0.0085	0.00041	A
5.17	0.178	0.0083	0.00040	A
5.59[d]	0.193	0.012[d]	0.00059	A+B
5.7[c]	0.197	–	–	B

[a] Calculated by the compiler.

[b] The solid phases are: A = $Ca(IO_3)_2 \cdot 6H_2O$; B = $Al(IO_3)_3 \cdot 6H_2O$.

[c] For binary systems the compiler computes the following values:

$$\text{Solubility of } Al(IO_3)_3 = 0.110 \text{ mol kg}^{-1}$$
$$\text{Solubility of } Ca(IO_3)_2 = 8.49 \times 10^{-3} \text{ mol kg}^{-1}.$$

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 20–25 d. Samples of the liquid and solid phases were analyzed for iodate ion by iodometric titration. The calcium concentration in the liquid phase was determined by flame emission spectroscopy; the solutions were buffered by an excess of lanthanum chloride to eliminate the influence of aluminum on the intensity of the calcium radiation. The calcium content in the solid phases was determined by spectroscopy. The quantity of aluminum was found by the difference. The solid phases were identified by the "residues" method, and verified by X-ray diffraction.

Source and Purity of Materials:

Aluminum iodate hexahydrate was prepared from freshly precipitated aluminum hydroxide and iodic acid. Calcium iodate hexahydrate was synthesized from calcium nitrate and iodic acid.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.1 K.

Components:

(1) Aluminum iodate; $\mathrm{Al(IO_3)_3}$; [15123-75-8]
(2) Indium iodate; $\mathrm{In(IO_3)_3}$; [13510-34-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, B. I. Kidyarov, V. A. Kuzina, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **33**, 219–21 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 122–3 (1988).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 81. Composition of saturated solutions in the ternary $\mathrm{Al(IO_3)_3}$–$\mathrm{In(IO_3)_3}$–H_2O system at 25 °C

$\mathrm{Al(IO_3)_3}$		$\mathrm{In(IO_3)_3}$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
–	–	0.049[c]	1.4×10^{-3}	A
0.61	0.020	0.0036	1.0×10^{-4}	B
1.41	0.047	0.0027	7.7×10^{-5}	B
2.04	0.068	0.0016	4.6×10^{-5}	B
2.45	0.082	0.0015	4.3×10^{-5}	B
3.07	0.103	0.0014	4.1×10^{-5}	B
3.78	0.128	0.0015	4.4×10^{-5}	B
4.09	0.139	0.0026	7.6×10^{-5}	B
4.50	0.154	0.0035	1.0×10^{-4}	B
5.11[d]	0.176	0.0038[d]	1.1×10^{-4}	B+C
5.70[c]	0.197	–	–	C

[a] Calculated by the compiler.

[b] The solid phases are: A = $\mathrm{In(IO_3)_3 \cdot H_2O}$; B = solid solution based on $\mathrm{In(IO_3)_3 \cdot H_2O}$; C = $\mathrm{Al(IO_3)_3 \cdot 6H_2O}$.

[c] For binary systems the compiler computes the following values:

Solubility of $\mathrm{Al(IO_3)_3}$ = 0.110 mol kg^{-1}
Solubility of $\mathrm{In(IO_3)_3}$ = 7.7×10^{-4} mol kg^{-1}.

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied by the isothermal method. Equilibrium was reached in 20–30 d. Samples of the coexisting phases were analyzed for iodate ion by an iodometric titration. Aluminum content in the liquid and in the solid phase containing more than 1 mass % Al was determined by a complexometric back titration, and in other cases by spectrography. Indium content in the liquid phase was determined by atomic absorption method with the solution previously buffered with lanthanum chloride: a control determination was carried out simultaneously by a spectroscopic method. The indium in the solid phases was calculated by the difference and checked by a complexometric titration. The solid phases were identified by the "residues" method and checked by X-ray diffraction.

Source and Purity of Materials:

$\mathrm{Al(IO_3)_3 \cdot 6H_2O}$ was obtained by the reaction of freshly precipitated aluminum hydroxide with iodic acid. $\mathrm{In(IO_3)_3 \cdot H_2O}$ was prepared from indium nitrate and iodic acid.

Estimated Error:

Solubility: precision no better than ± 1 % (compiler).
Temperature: precision ± 0.1 K.

Components:

(1) Aluminum iodate; $Al(IO_3)_3$; [15123-75-8]
(2) Neodymium iodate; $Nd(IO_3)_3$; [14732-16-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, L. A. Kolobkova, and N. G. Dubovenko Zh. Neorg. Khim. **33**, 3217–8 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 1859–60 (1988).

Variables:

Composition of the the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 82. Composition of saturated solutions in the ternary $Al(IO_3)_3$–$Nd(IO_3)_3$–H_2O system at 25 °C

$Al(IO_3)_3$		$Nd(IO_3)_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
–	–	0.070[c]	1.9×10^{-3}	A
0.32	0.010	0.0088	2.4×10^{-4}	A
0.70	0.023	0.00062	1.7×10^{-5}	A
1.00	0.033	0.00050	1.4×10^{-5}	A
1.46	0.048	0.00025	0.68×10^{-5}	A
1.64	0.054	0.00012	0.33×10^{-5}	A
1.84	0.061	< 0.00010	$< 0.28 \times 10^{-5}$	A
2.45	0.081	< 0.00010	$< 0.28 \times 10^{-5}$	A
3.27	0.110	< 0.00010	$< 0.28 \times 10^{-5}$	A
4.42	0.151	< 0.00010	$< 0.28 \times 10^{-5}$	A
5.06	0.174	< 0.00010	$< 0.28 \times 10^{-5}$	A
5.47[d]	0.189	< 0.00010[d]	$< 0.28 \times 10^{-5}$	A+B
5.70[c]	0.197	–	–	B

[a] Calculated by the compiler.

[b] The solid phases are: A = $Nd(IO_3)_3 \cdot 2H_2O$; B = $Al(IO_3)_3 \cdot 6H_2O$.

[c] For binary systems the compiler computes the following values:

Solubility of $Al(IO_3)_3$ = 0.110 mol kg^{-1}
Solubility of $Nd(IO_3)_3$ = 1.05×10^{-3} mol kg^{-1}.

[d] Eutonic composition.

Auxiliary Information

Method/Apparatus/Procedure:

The aluminum iodate–neodymium iodate–water system was studied isothermally. The equilibrium was established within 20–25 d. The iodate ion in the sample solution was determined iodometrically. The aluminum content, in the liquid phase and in the sample of the solid “residues” with Al content of > 0.3 mass $\%$, was determined by back titration using ammonium fluoride. In the solid phase specimens and the neodymium iodate crystallization region, aluminum was determined spectrometrically. Neodymium in the specimens of the solid “residues” was determined by complexometric titration in the presence of sulphosalicylic acid with methyl thymol blue, and it was determined spectrometrically in specimens of the liquid phases. The solid phases were identified by the the method of “residues” and the identification was checked by X-ray diffraction.

Source and Purity of Materials:

$\mathrm{Al(IO_3)_3 \cdot 6H_2O}$ was obtained from freshly precipitated aluminum hydroxide and iodic acid. $\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$ was synthesized by the reaction of neodymium oxide and iodic acid.

Estimated Error:

Solubility: nothing specified.

Temperature: precision within ± 0.1 K.

Components:

(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of zinc iodate in water have been reported in 10 publications (1–10). Three solubility studies (6–8) deal with ternary systems and the solubility in the binary system was treated as one point in those ternary systems. The solubility values in water were also reported in studies concerning the solubility in mixtures of aqueous–organic solvents (1, 3, 9) and aqueous solutions containing other electrolyte (2, 4, 5). The elder work of Rammelsberg (10) was not compiled because its copy was not available; Linke (11) have reported his results that 100 g of cold H_2O dissolve 0.87 g of $\mathrm{Zn(IO_3)_2}$ and 1.31 g of $\mathrm{Zn(IO_3)_2}$ in the same amount of hot water. These results are of proper order magnitude. Lepeshkov et al. (6–8) have stated that the composition of solid phase equilibrated with the saturated solution was $\mathrm{Zn(IO_3)_2 \cdot 2H_2O}$. No information on equilibrium solid phases was reported in the other papers. A summary of solubility studies of $\mathrm{Zn(IO_3)_2}$ in water is given in Table 83 and in Fig. 10.

TABLE 83. Summary for solubility studies of $\mathrm{Zn(IO_3)_2}$ in water at various temperatures

T / K	$10^2 c_1$ / mol dm^{-3}	$10^2 m_1$ / mol kg^{-1}	Analytical method	Reference
293.4	1.421	—	Radiotracer analysis	(4)
298	1.457	1.461	Iodometry	(2)
298	1.548	1.554	Volumetry, Gravimetry	(1)
298.15	1.556	—	Iodometry	(3)
298	1.63	—	Iodometry (Amperometry)	(5)
301.6	1.654	—	Radiotracer analysis	(4)
303	—	0.5141	Complexometry (Zn^{2+})	(9)
313.8	1.893	—	Radiotracer analysis	(4)
323	—	1.6	Complexometry (Zn^{2+}), Iodometry (IO_3^-)	(6)
323	—	1.6	Complexomety (Zn^{2+}), Iodometry (IO_3^-)	(7)
323	—	1.6	Complexometry (Zn^{2+})	(8)

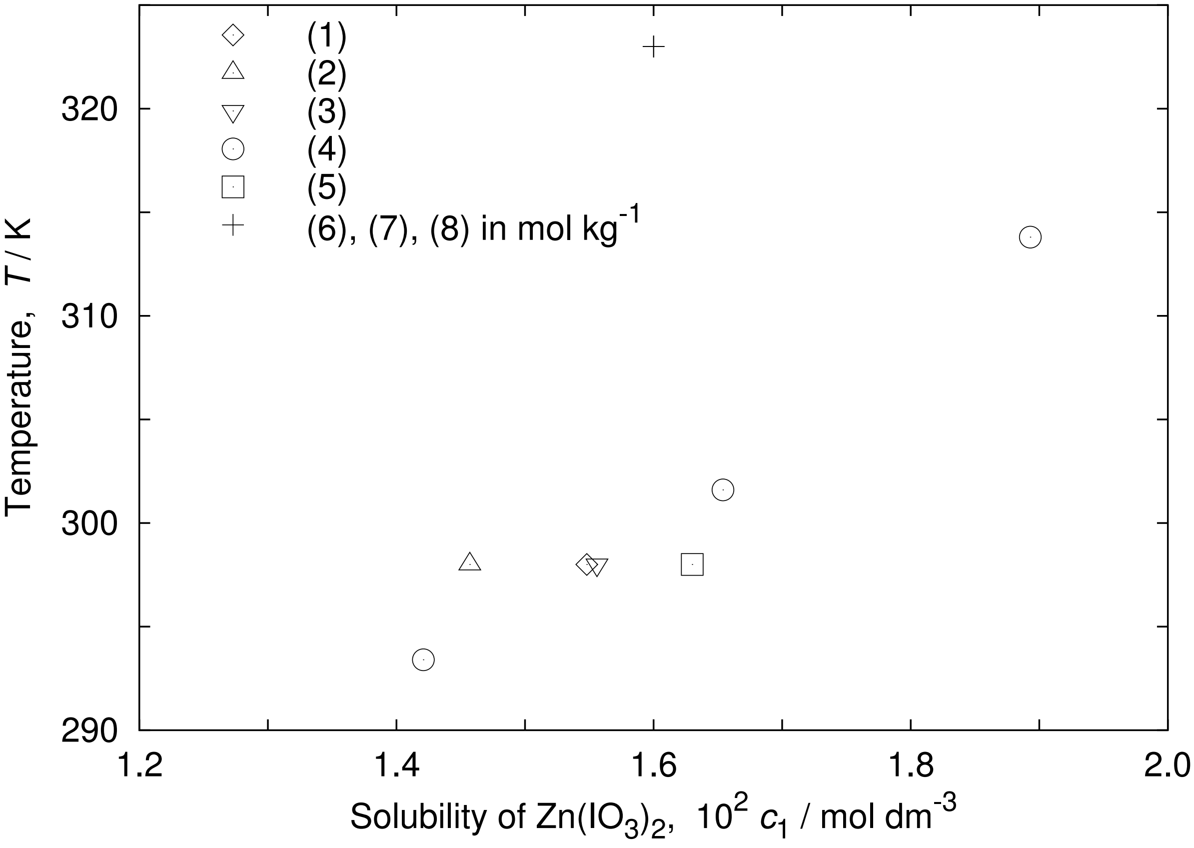

FIG. 10. Temperature dependence of solubility of $\mathrm{Zn(IO_3)_2}$ in water

Four data (1–3, 5) are available for the evaluation of the solubility of zinc iodate at 298 K. The arithmetic mean of 4 results is 1.55×10^{-2} mol dm^{-3}, and the standard deviation is $\sigma = 0.07 \times 10^{-2}$ mol dm^{-3}. The mean value, which is in agreement with data of Spencer et al. (4) at other temperature, is designated as a recommended value.

The solubilities of 1.42×10^{-2}, 1.65×10^{-2} and 1.85×10^{-2} mol dm^{-3} at 293.4, 301.6 and 313.8 K, respectively, are designated as tentative values. They are in agreement with the recommended value at 298 K.

The solubility of zinc iodate at 298 K based on mol kg^{-1} units has been reported in 2 publications (1, 2). The arithmetic mean of 2 results is 1.51×10^{-2} mol kg^{-1} with the standard deviation $\sigma = 0.04$. However, the mean value is more doubtful than the recommended solubility in mol dm^{-3} units, because it should be slightly higher than the solubility expressed in mol dm^{-3}.

The solubility at 323 K has been reported in 3 publications (6–8) by Lepeshkov et al. (6, 8) and Vinogradov (7). The reported solubility of 1.6×10^{-2} mol kg^{-1} is lower than the tentative values in mol dm^{-3} at 301.6 and 313.8 K and this fact needs additional verification. The solubility at 303.2 K reported by Varghese and Kalidas (9) is extremely doubtful, and is rejected.

The Ternary System

The solubilities of zinc iodate in aqueous solutions of potassium nitrate have been reported in 2 publications (2, 4). Saegusa (2) measured the solubility at 298 K, and mean activity coefficients were calculated from the solubility. The reported solubility increases with an increase of the KNO_3 concentration up to 0.5 mol dm^{-3}, and then solubility decreases in the range 0.5–0.95 mol dm^{-3}. Spencer et al. (4) studied the solubility at 293.4, 301.6 and 313.8 K. The solubility increases monotonically with increase of the KNO_3 concentration up to 0.1 mol dm^{-3}, and any maximum value for the solubility was not observed. Fedorov et al. (5) have reported the solubilities in aqueous solutions of lithium nitrate and perchlorate at 298 K. Maxima of the solubility are observed in both systems: at 1.0 mol dm^{-3} $LiClO_4$ and 2.0 mol dm^{-3} $LiNO_3$. These maxima on the curve might be due to the effect of ion–ion interaction.

Ricci and Nesse (1), Miyamoto (2), and Varghese and Kalidas (9) have reported the solubilities of zinc iodate in mixtures of organic solvents and water. 1,4-Dioxane (1), tetrahydrofuran (3) and pyridine (9) were used as organic solvents. The results of Ricci and Nesse (1), and Miyamoto (3) were used to test the application of the Born equation or its modification. Verghese and Kalidas (9) discussed the ion–solvent interactions in pyridine–water mixtures from the solubility measurements.

The solubility studies of the ternary system with HIO_3, KIO_3, and $RbIO_3$ have been reported in 3 publications (6–8). All systems were studied by the isothermal method. The summary of the solubility studies are listed in Table 84 and the detailed compilations were given in (12).

TABLE 84. Summary for solubility studies of $Zn(IO_3)_2$ in the ternary systems with iodic acid and alkali metal iodates at 323 K

Ternary system	Solid phase	Reference
$Zn(IO_3)_2$–HIO_3-H_2O	HIO_3, $Zn(IO_3)_2 \cdot 2H_2O$	(8), compiled in (12)
$Zn(IO_3)_2$–KIO_3–H_2O	KIO_3, $2KIO_3 \cdot Zn(IO_3)_2 \cdot 2H_2O$, $Zn(IO_3)_2 \cdot 2H_2O$	(6), compiled in (12)
$Zn(IO_3)_2$–$RbIO_3$–H_2O	$RbIO_3$, $Zn(IO_3)_2 \cdot 2H_2O$	(7), compiled in (12)

References:

1. J. E. Ricci and G. J. Nesse, J. Am. Chem. Soc. **64**, 2305 (1942).
2. F. Saegusa, Nippon Kagaku Kaishi **71**, 223 (1950).
3. H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659.
4. J. N. Spencer, E. A. Unger, and D. N. Bailey, J. Chem. Eng. Data **19**, 140 (1974).
5. V. A. Fedorov, A. M. Robov, I. I. Shmyd'ko, T. N. Koneva, L. S. Simaeva, and V. A. Kukhtina, Zu. Fiz. Khim. **50**, 2213 (1976); Russ. J. Phys. Chem. (Engl. Transl.) **50**, 1330 (1976).
6. I. N. Lepeshkov, E. E. Vinogradov, and I. M. Karataeva, Zh. Neorg. Khim. **22**, 2277 (1977); Russ. J. Inorg. Chem. (Engl. Transl.) **22**, 1232 (1977).
7. E. E. Vinogradov and I. M. Karataeva, Zh. Neorg. Khim. **24**, 2529 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 1406 (1979).
8. I. N. Lepeshkov, E. E. Vinogradov and I. M. Karataeva, Zh. Neorg. Khim. **24**, 2540 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 1412 (1979).
9. A. V. Varghese and C. Kalidas, Z. Naturforsch. **46a**, 703 (1991).
10. Rammelsberg, Pogg. Ann. **43**, 665 (1838); ibid **44**, 573 (1838).
11. W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1668.
12. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 402, p. 439, p. 482.

Components:
(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]
(2) 1,4-Dioxane; $\mathrm{C_4H_8O_2}$; [123-91-1]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
J. E. Ricci and G. J. Nesse, J. Am. Chem. Soc. **64**, 2305–11 (1942).

Variables:
Concentration of dioxane up to 100 %
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 85. Solubility of $\mathrm{Zn(IO_3)_2}$ in dioxane–water mixtures at 25 °C

$100w_2$	$100x_2{}^{\mathrm{a}}$	$100w_1$	10^3c_1 / mol dm^{-3}
0	0	0.6410	15.48
10	2.2	0.3746	9.10
20	4.9	0.2301	5.62
30	8.1	0.1158	2.85
40	12.0	0.0572	1.42
50	17.0	0.0244	0.605
60	23.5	0.0090	0.22
70	32.3	0.0030	0.075
80	45.0	0.0017	0.042
90	64.8	0.0008	0.019
100	100	< 0.0001	< 0.002

[a] Calculated by the compilers.

Anhydrous zinc iodate (99.3 % pure) was found as the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:

Mixtures of dioxane and water, prepared in the desired proportions by direct weighing, were stirred with excess of zinc iodate crystals in glass-stoppered bottles. Equilibrium was established after stirring for 2–7 d. Method involving reduction of iodate to iodide by sodium bisulfite was used for analysis of the saturated solutions. The excess of the reagent was removed by boiling with diluted sulfuric acid. In high dioxane solvents ($>$ 40 mass %), the resulting iodide solution was analyzed volumetrically, at pH of 9–10 by titration with standard silver nitrate using eosin indicator. The iodide formed was determined gravimetrically as silver iodide at the very low solubility. The densities of the saturated solutions were determined separately to calculate the molarities.

Source and Purity of Materials:

Zinc iodate was prepared by mixing dilute solutions of sodium iodate and zinc nitrate. The solutions were poured slowly into a large beaker with constant mechanical stirring. Zinc iodate was obtained by evaporating some of the liquid, filtering, washing and drying its crystals at 100 °C. Volumetric analysis of the product gave a value of 99.3 % $\mathrm{Zn(IO_3)_2}$; the additional 0.7 % was probably represented by water not readily driven off by heat without decomposition of the salt. A purified dioxane was used and its purity was verified.

Estimated Error:

Solubility: precision within 0.6–0.7 %; gravimetric analysis was less precise; detection limit of 0.0001 mass %.

Temperature: nothing specified.

Components:

(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]
(2) Potassium nitrate; KNO_3; [7757-79-1]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

F. Saegusa, Nippon Kagaku Zasshi **71**, 223–6 (1950).

Variables:

Concentration of KNO_3: 0–0.94 mol dm^{-3}
Temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 86. Solubility of $\mathrm{Zn(IO_3)_2}$ in aqueous potassium nitrate solutions at 25 °C

10^2c_2 / mol dm^{-3}	10^2m_2 / mol kg^{-1}	10^2c_1 / mol dm^{-3}	10^2m_1 / mol kg^{-1}	10^4x_1	ρ / g cm^{-3}
0	0	1.457	1.461	2.630	1.0034
0.2523	0.2530	1.472	1.476	2.657	1.0038
1.001	1.004	1.513	1.518	2.731	1.0042
5.035	5.058	1.686	1.694	3.043	1.0075
10.07	10.14	1.826	1.839	3.298	1.0109
23.67	23.96	2.063	2.008	3.725	1.0203
47.34	48.41	2.433	2.488	4.399	1.0358
62.87	64.66	2.353	2.420	4.255	1.0456
74.81	77.29	1.897	1.960	3.432	1.0514
94.68	98.72	1.730	1.804	3.135	1.0619

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:

Excess of zinc iodate crystals and aqueous potassium nitrate solutions were placed in glass-stoppered bottles coated with paraffin. The bottles were rotated in a thermostat at the selected temperature for 15 h. Aliquots of the saturated solutions were filtered through cotton wool, and the iodate content was determined iodometrically.

Source and Purity of Materials:

Zinc iodate was prepared by adding aqueous zinc chloride solution to potassium iodate solution. The precipitate was washed and dried. Guaranteed reagent potassium nitrate was recrystallized twice from water. Conductivity water was used.

Estimated Error:

Solubility: precision within 0.3 %.
Temperature: nothing specified.

Components:
(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]
(2) Tetrahydrofuran; $\mathrm{C_4H_8O}$; [109-99-9]
(3) Water; $\mathrm{H_2O}$; [7732-18-5]

Original Measurements:
H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659–61.

Variables:
Concentration of tetrahydrofuran: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 87. Solubility of $\mathrm{Zn(IO_3)_2}$ in tetrahydrofuran–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	15.56
5	1.3	11.03
10	2.7	7.71
15	4.2	5.23
20	5.9	3.57
25	7.7	2.39
30	9.7	1.52
40	14.3	0.79

The equilibrium solid phase was not analysed.

Auxiliary Information

Method/Apparatus/Procedure:
An excess of zinc iodate crystals and the solvent mixtures were placed into glass-stoppered bottles. The bottles were rotated in a thermostat at the selected temperature for 48 h. Then the saturated solutions were withdrawn through a siphon tube equipped with a sintered-glass filter. The iodate content was determined iodometrically.

Source and Purity of Materials:
Anhydrous zinc iodate was prepared by dropping solutions of equivalent quantities of zinc nitrate (Wako guaranteed reagent) and potassium iodate (Wako guaranteed reagent) in large volume of aqueous potassium nitrate solution. The precipitate was filtered, washed and dried under reduced pressure. Tetrahydrofuran was distilled over sodium hydroxide and then redistilled over sodium metal.

Estimated Error:
Solubility: precision between 0.1 % and 1 %.
Temperature: precision ± 0.02 K.

Components:

(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]
(2) Potassium nitrate; KNO_3; [7757-79-1]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

J. N. Spencer, E. A. Unger, and D. N. Bailey, J. Chem. Eng. Data **19**, 140–1 (1974).

Variables:

Concentration of KNO_3: 0–0.1 mol dm^{-3}
Temperature: 293.4–313.8 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 88. Solubility of $\mathrm{Zn(IO_3)_2}$ in aqueous KNO_3 solutions at various temperatures

t / °C	c_2 / mol dm^{-3}	$10^2 c_1$ / mol dm^{-3}	$10^2 s_0$ / mol dm^{-3} [a]	$10^6 K_{\mathrm{so}}^{\mathrm{o}}$ / mol^3 dm^{-9} [b]
20.2	0	1.421	0.9632	3.57 ± 0.21
	0.02500	1.567	—	
	0.05000	1.643	—	
	0.07500	1.756	—	
	0.1000	1.814	—	
28.4	0	1.654	1.105	5.40 ± 0.20
	0.02500	1.766	—	
	0.05000	1.880	—	
	0.07500	1.961	—	
	0.1000	2.025	—	
40.6	0	1.893	1.227	7.39 ± 0.36
	0.02500	2.050	—	
	0.05000	2.171	—	
	0.07500	2.243	—	
	0.1000	2.327	—	

[a] s_0 is the solubility at zero ionic strength calculated from the Debye–Hückel relation.

[b] Error reported is the maximum error based on the uncertainty in the least-squares extrapolated values of $\log s_0$.

According to the authors, a complex formation between Zn^{2+} and IO_3^- seems to be unimportant.

Auxiliary Information

Method/Apparatus/Procedure:

For the solubility measurements, 3 samples of the radioactive precipitate were prepared at each ionic strength. The ionic strength was adjusted with potassium nitrate. Samples were equilibrated by shaking in constant-temperature baths. Equilibration was generally attained after 2 d. The activity of the sample solutions was determined by counting 5 cm^3 aliquots of each sample on a NaI(Tl) scintillation counter over a period of days to ensure equilibration conditions. The samples were prepared by centrifuging 10 cm^3 of each sample and pipeting 5 cm^3 of the supernatant solution into test tubes. A count rate of 5000 cpm was attained for each sample. All samples were counted for a time sufficient to minimize counting errors with respect to other experimental errors. The sample activity was compared to that of the standard. A minimum of 6 determinations was made for each solubility. The standard solution of $^{65}\mathrm{Zn}(\mathrm{IO}_3)_2$ was prepared by quantitatively transferring weighed amounts of the tagged precipitate to volumetric flasks and dissolving the precipitate in 6 mol dm^{-3} HCl.

Source and Purity of Materials:

Solutions of potassium iodate (J. T. Baker AR) and zinc nitrate (J. T. Baker AR) were heated to boiling. The iodate solution was added slowly to the zinc nitrate solution with continuous stirring. The precipitate was allowed to digest and after filtration was dried over phosphorous pentaoxide. The precipitate was dissolved, and the dilute solution was titrated with a solution of arsenous oxide and was found to be 98.6 % pure. The radioactive precipitate was prepared in same fashion with tracer amounts of ^{65}Zn.

Estimated Error:

Solubility: accuracy ± 0.4 %.
Temperature: precision ± 0.2 K.

Components:
(1) Zinc iodate; $\mathrm{Zn(IO_3)_2}$; [7790-37-6]
(2) Lithium nitrate; $LiNO_3$; [7790-69-4]
or (2) Lithium perchlorate; $LiClO_4$; [7791-03-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
V. A. Fedorov, A. M. Robov, I. I. Shmyd'ko, T. N. Koneva, L. S. Simaeva, and V. A. Kukhtina, Zh. Fiz. Khim. **50**, 2213–5 (1976); Russ. J. Phys. Chem. (Engl. Transl.) **50**, 1330–2 (1976).

Variables:
Concentration of lithium salts: 0–4.0 mol dm^{-3}
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 89. Solubility of $\mathrm{Zn(IO_3)_2}$ in aqueous $LiNO_3$ or $LiClO_3$ solutions at 25 °C

Lithium salt	c_2 / mol dm^{-3}	10^2c_1 / mol dm^{-3}
None	—	1.63
$LiNO_3$	0.01	1.74
	0.05	1.89
	0.10	2.00
	0.25	2.22
	0.40	2.33
	0.50	2.39
	1.00	2.58
	2.00	2.70
	3.00	2.65
	4.00	2.50
$LiClO_4$	0.01	1.67
	0.05	1.79
	0.10	1.89
	0.25	2.03
	0.40	2.12
	0.50	2.15
	1.00	2.20
	2.00	2.00
	3.00	1.64
	4.00	1.14

The authors stated that the solid phase was $\mathrm{Zn(IO_3)_2 \cdot 2H_2O}$, independently of the salt composition. pK_s° values found after extrapolation to the zero ionic strength are: 5.40 (in $LiClO_4$), 5.38 (in $LiNO_3$) and 5.39 (in pure H_2O).

Auxiliary Information

Method/Apparatus/Procedure:

The experimental method used was similar to that of Fedorov et al. (1). Equilibrium required 4–6 h of intensive mixing of the solutions and salts. The solubilities were determined by iodometric titration of the iodate with amperometric end point detection. Each solubility value given in the data table above is the mean result of 3–4 determinations.

Source and Purity of Materials:

The source and purities of materials were not given.

Estimated Error:

Solubility: reproducibility within 2–3 %.

Temperature: nothing specified.

References:

[1] V. A. Fedorov, I. I. Shmyd'ko, A. M. Robov, L. S. Simaeva, V. A. Kukhtina, and V. E. Mironov, Zh. Neorg. Khim. **18**, 1274 (1973): Russ. J. Inorg. Chem. (Engl. Transl.) **18**, 673 (1973).

Components:

(1) Zinc iodate; $Zn(IO_3)_2$; [7790-37-6]

(2) Pyridine; C_5H_5N; [110-86-1]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

A. V. Varghese and C. Kalidas, Z. Naturforsch. **46a**, 703–6 (1991).

Variables:

Concentration of pyridine

One temperature: 303 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 90. Solubility of $Zn(IO_3)_2$ in pyridine–water mixtures at 30 °C

x_2	$10^4 m_1$ / mol kg^{-1}	pK_s°	$10^7 K_s^\circ$ / mol^3 kg^{-9} [a]
0	51.41	6.537	2.90
0.1	69.90	6.156	6.98
0.2	36.88	7.128	0.745
0.3	23.32	7.719	0.191
0.4	7.07	9.260	0.00550
0.5	2.54	10.50	0.00032
0.6	1.80	11.04	0.000091
0.7	1.76	11.19	0.000065
0.8	1.58	11.47	0.000034
0.9	1.51	11.60	0.000025
1.0	1.34	11.84	0.000014

[a] Calculated by the compilers.

Method/Apparatus/Procedure:

The details of preparation of the $\mathrm{Zn(IO_3)_2}$ saturated solutions were given in (1). Sufficient amounts of the salts were stirred with the solvent mixtures in a thermostated vessel. Saturation was achieved after 12 h of stirring where no detectable changes of the salt concentration were noted. The liquid was then filtered off in a G-4 glass filter. The solubilities in the resulting solutions were determined by estimating the zinc content by complexometric titration with EDTA. All solubility measurements were carried out in duplicate.

Source and Purity of Materials:

Zn(II) iodate was prepared by the metathesis of zinc(II) sulfate and potassium iodate. The anhydrous salt was obtained by heating this sample to 240 $^\circ\mathrm{C}$, and it was stored over $CaCl_2$ under vacuum. To test the salt purity, the zinc content was determined by EDTA titration using eriochrome black-T indicator. Pyridine (GR, Merck) was refluxed over KOH pellets and then distilled with the exclusion of moisture. The middle fraction, boiling at 115 $^\circ\mathrm{C}$ and 0.1 MPa, was collected and stored over KOH pellets.

Estimated Error:

Solubility: accuracy ± 0.2 %.

Temperature: precision ± 0.1 K.

References:

[1] C. Kalidas and H. Schneider, Z. Phys. Chem. N. F. **120**, 145 (1980).

Components:

(1) Gallium iodate; $Ga(IO_3)_3$; [70504-12-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Ryo Miyamoto, Hirosaki University, Hirosaki, Japan. October 2001.

Critical Evaluation:

The Binary System

Solubility data for gallium iodate in pure water have been reported in 4 publications (1–4). These studies deal with ternary systems, and the solubility in the binary system was given as one point on relative phase diagrams. The same value was reported in successive papers of Shklovskaya et al. (1–4). The value of 0.014 mol kg^{-1} is designated as a tentative solubility at 298 K. The stable solid phase in equilibrium with the saturated solution at 298 K is $Ga(IO_3)_3 \cdot 2H_2O$ [70504-76-3].

The Ternary System

Solubility studies for aqueous ternary systems with some inorganic compounds have been reported in 4 publications (1-4). All systems were studies by isothermal method. The summary of these studies is given in Table 91.

TABLE 91. Summary for solubility studies of $Ga(IO_3)_3$ in the ternary systems at 298 K

Ternary system	Solid phase	Reference
$Ga(IO_3)_3$–HIO_3–H_2O	HIO_3; $Ga(IO_3)_3 \cdot 2H_2O$; $Ga(IO_3)_3 \cdot 2HIO_3 \cdot 2H_2O$; solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$	(3)
$Ga(IO_3)_3$–$LiIO_3$–H_2O	α-$LiIO_3$; $Ga(IO_3)_3 \cdot 2H_2O$; $2Ga(IO_3)_3 \cdot LiIO_3 \cdot 4H_2O$; $Ga(IO_3)_3 \cdot LiIO_3 \cdot 2H_2O$; solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$	(1), compiled in (5)
$Ga(IO_3)_3$–$Mg(IO_3)_2$–H_2O	$Ga(IO_3)_3 \cdot 2H_2O$; $Mg(IO_3)_2 \cdot 4H_2O$; solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$; solid solution based on $Mg(IO_3)_2 \cdot 4H_2O$	(2)
$Ga(IO_3)_3$–$Al(IO_3)_3$–H_2O	$Ga(IO_3)_3 \cdot 2H_2O$; $Al(IO_3)_3 \cdot 6H_2O$	(2)
$Ga(IO_3)_3$–$Ga(NO_3)_3$–H_2O	$Ga(IO_3)_3 \cdot 2H_2O$; $Ga(NO_3)_3 \cdot 8H_2O$	(4)
$Ga(IO_3)_3$–$Ga_2(SO_4)_3$–H_2O	$Ga(IO_3)_3 \cdot 2H_2O$; $Ga_2(SO_2)_4 \cdot 10H_2O$; solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$	(4)

In the system with HIO_3 (3), the solid solution based on $Ga(IO_3)_3{\cdot}2H_2O$ and the incongruently soluble compound $Ga(IO_3)_3 \cdot 2H_2O$ are formed. In the system with $Mg(IO_3)_2$ (2), two types of limiting solid solutions based on $Ga(IO_3)_3{\cdot}2H_2O$ and on $Mg(IO_3)_2{\cdot}4H_2O$ are formed. The ternary systems with $Al(IO_3)_3$ (2) and $Ga(NO_3)_3 \cdot 4H_2O$ (4) are of simple eutonic type and no double salts were formed. The system formed by gallium iodate and sulfate is eutonic, and restricted to solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$. The detailed compilation of the ternary $Ga(IO_3)_3$–$LiIO_3$–H_2O system is given in (5). Note that the compilation for the system with $Al(IO_3)_3$ is given in the data sheets concerned with $Al(IO_3)_3$ in this volume.

References:

1. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, A. G. Tokareva, and V. A. Kuzina, Zh. Neorg. Khim. **25**, 1112 (1980); Russ. J. Inorg. Chem. (Engl. Transl.) **25**, 618 (1980).
2. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 219 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 122 (1985).
3. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, T. E. Vdovkina, V. A. Kuzina, and A. G. Tokareva, Zh. Neorg. Khim. **31**, 272 (1986); Russ. J. Inorg. Chem. (Engl. Transl.) **31**, 155 (1986).
4. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and T. E. Vdovkina, Zh. Neorg. Khim. **32**, 2334 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 1365 (1987).
5. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 300.

Components:

(1) Gallium iodate; $Ga(IO_3)_3$; [70504-12-0]

(2) Magnesium iodate; $Mg(IO_3)_2$; [7790-32-1]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. V. Poleva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 219–21 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 122–3 (1985).

Variables:

Composition of the salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 92. Composition of saturated solutions in the ternary $Ga(IO_3)_3$–$Mg(IO_3)_2$–H_2O system at 25 °C

$Ga(IO_3)_3$		$Mg(IO_3)_2$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.85[c]	0.026	—	—	A
0.67	0.021	0.97	0.047	B
0.42	0.013	1.85	0.091	B
0.34	0.011	2.74	0.136	B
0.30	0.0094	3.49	0.174	B
0.26	0.0083	4.51	0.228	B
0.25	0.0081	5.87	0.300	B
0.26	0.0085	6.97	0.360	B
0.28	0.0092	7.76	0.405	B
0.29	0.0096	8.63	0.454	B
0.30[d]	0.010	9.06[d]	0.479	B+C
0.27	0.0090	9.04	0.478	C
0.21	0.0070	9.02	0.476	C
0.16	0.0053	8.96	0.472	C
0.09	0.003	8.68	0.456	C
—	—	8.48[c]	0.444	D

[a] Calculated by the compiler.

[b] The solid phases are: A = $Ga(IO_3)_3 \cdot 2H_2O$; B = solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$; C = solid solution based on $Mg(IO_3)_2 \cdot 4H_2O$; D = $Mg(IO_3)_3 \cdot 4H_2O$.

[c] For binary systems the compiler computes the following values:

$$\text{Solubility of } Ga(IO_3)_3 = 0.246 \text{ mol kg}^{-1}$$
$$\text{Solubility of } Mg(IO_3)_2 = 0.014 \text{ mol kg}^{-1}.$$

[d] Eutonic point.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 25–30 d. Samples of the liquid phases and solid residues were analyzed for iodate ion by iodometric titration and for gallium by complexometric titration using xylenol orange as indicator. The magnesium content was calculated by the difference. The solid phases were identified by the "residues" method with X-ray diffraction used as a check.

Source and Purity of Materials:

Magnesium iodate was prepared from magnesium carbonate and iodic acid. Gallium iodate was prepared (1) from metallic gallium which was dissolved in nitric acid and the theoretical amount of iodic acid was added to the solution. The nitric acid was driven off by evaporation on a water-bath. The product was washed with water and carefully dried in a desiccator over anhydrous calcium chloride. Analysis of the product gives: Ga–11.06; I–60.84 mass %. Calculated content for $\mathrm{Ga(IO_3)_3 \cdot 2H_2O}$: Ga–11.09; I–60.38 mass %.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.1 K.

References:

[1] P. Neogi and S. K. Nandi, J. Indian Chem. Soc. **14**, 492 (1937).

Components:

(1) Gallium iodate; $Ga(IO_3)_3$; [70504-12-0]
(2) Iodic acid; HIO_3; [7782-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, T. E. Vdovkina, V. A. Kuzina, and A. G. Tokareva, Zh. Neorg. Khim. **31**, 272–4 (1986); Russ. J. Inorg. Chem. (Engl. Transl.) **31**, 155–6 (1986).

Variables:

Composition of salt and acid
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 93. Composition of saturated solutions in the ternary $Ga(IO_3)_3$–HIO_3–H_2O system at 25 °C

$Ga(IO_3)_3$		HIO_3		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.85[c]	0.026	—	—	A
0.20	0.0063	3.65	0.387	B
0.35	0.012	11.40	1.306	B
0.37	0.013	17.30	2.106	B
0.45	0.017	20.54	2.595	B
0.51	0.020	24.97	3.317	B
1.25	0.0533	30.90	4.454	B
1.66	0.0776	37.41	5.911	B
2.43	0.128	44.88	8.013	B
2.47	0.139	48.61	9.224	B
2.53	0.153	52.67	10.73	B
2.56[d]	0.158	54.03[d]	11.29	B+C
2.25	0.144	56.06	12.09	C
2.27	0.164	62.35	15.26	C
2.28	0.188	67.91	18.88	C
2.37	0.209	70.58	21.04	C
2.23[e]	0.203	71.86[e]	22.08	C+D
1.17	0.106	72.84	22.28	D
—	—	75.1[c]	23.60	D

[a] Calculated by the compiler.

[b] The solid phases are: A = $Ga(IO_3)_3 \cdot 2H_2O$; B = solid solution based on $Ga(IO_3)_2 \cdot 2H_2O$; C = $Ga(IO_3)_3 \cdot 2HIO_3 \cdot 2H_2O$; D = HIO_3.

[c] For the binary system the compiler computes the following value:

$$\text{Solubility of } Ga(IO_3)_3 = 0.014 \text{ mol kg}^{-1}.$$

[d] Peritonic point.
[e] Eutonic point.

Auxiliary Information

Method/Apparatus/Procedure:
The isothermal method was used. Equilibrium was reached in 25–30 d. The iodate content in the saturated solution was determined iodometrically. Samples were analyzed for gallium by flame absorption spectrometry in the case of the liquid phase and by complexometric titration in the case of the residues. The concentration of iodic acid in the samples was found by the difference. The solid phases were identified by the “residues” method, and the results confirmed by X-ray diffraction.

Source and Purity of Materials:
Gallium iodate dihydrate was prepared from gallium nitrate and iodic acid. “Chemically pure” grade iodic acid was recrystallized from its aqueous solution.

Estimated Error:
Solubility: nothing specified.
Temperature: precision ± 0.1K.

Components:

(1) Gallium iodate; $Ga(IO_3)_3$; [70504-12-0]
(2) Gallium nitrate; $Ga(NO_3)_3$; [13494-90-1]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and T. E. Vdovkina, Zh. Neorg. Khim. **32**, 2334–6 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 1365–7 (1987).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 94. Composition of saturated solutions in the ternary $Ga(IO_3)_3$–$Ga(NO_3)_3$–H_2O system at 25 °C

$Ga(IO_3)_3$		$Ga(NO_3)_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.85[c]	0.026	–	–	A
1.36	0.0432	3.45	0.225	A
1.64	0.0540	6.86	0.525	A
1.71	0.0577	9.13	0.716	A
1.77	0.0633	14.60	1.214	A
1.93	0.0712	17.14	1.469	A
2.15	0.0841	22.00	2.001	A
2.04	0.0836	25.90	2.467	A
1.95	0.0818	27.81	2.711	A
1.77	0.0774	31.18	3.169	A
1.59	0.0735	35.34	3.794	A
1.36	0.0668	39.76	4.538	A
1.29	0.0673	43.72	5.300	A
1.45[d]	0.0782	45.57[d]	5.709	A+B
0.15	0.0078	44.31	5.321	B
–	–	45.84[c]	5.627	B

[a] Calculated by the compiler.

[b] The solid phases are: A = $Ga(IO_3)_3 \cdot 2H_2O$; B = $Ga(NO_3)_3 \cdot 8H_2O$.

[c] For binary systems the compiler computes the following values:

$$\text{Solubility of } Ga(IO_3)_3 = 0.014 \text{ mol kg}^{-1}$$
$$\text{Solubility of } Ga(NO_3)_3 = 3.310 \text{ mol kg}^{-1}.$$

[d] Eutonic point.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 20–30 d. In the liquid and solid phases, the iodate content was determined iodometrically, and gallium was determined by complexometric titration with PAN indicator. The quantity of nitrate ion was calculated by the difference. The solid phases were identified by the “residues” method, and the results were confirmed by X-ray diffraction patterns.

Source and Purity of Materials:

Gallium iodate dihydrate was prepared by the reaction of gallium nitrate with iodic acid. Gallium nitrate octahydrate of unspecified purity was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.1 K.

Components:

(1) Gallium iodate; $Ga(IO_3)_3$; [70504-12-0]
(2) Gallium sulfate; $Ga_2(SO_4)_3$; [13494-91-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and T. E. Vdovkina, Zh. Neorg. Khim. **32**, 2334–6 (1987); Russ. J. Inorg. Chem. (Engl. Transl.) **32**, 1365–7 (1987).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 95. Composition of saturated solutions in the ternary $Ga(IO_3)_3$–$Ga_2(SO_4)_3$–H_2O system at 25 °C

$Ga(IO_3)_3$		$Ga_2(SO_4)_3$		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	
0.85[c]	0.026	—	—	A
1.19	0.0368	0.82	0.035	B
1.34	0.0419	1.76	0.076	B
1.72	0.0555	4.63	0.208	B
1.80	0.0591	6.15	0.281	B
2.25	0.0746	8.80	0.406	B
2.49	0.0881	12.41	0.610	B
2.57	0.0963	17.36	0.904	B
2.70	0.107	21.71	1.194	B
2.49	0.101	24.04	1.358	B
2.27	0.0955	26.92	1.575	B
2.20	0.0956	29.38	1.775	B
2.16[d]	0.0982	32.60[d]	2.060	B+C
0.84	0.0395	36.18	2.362	C
—	—	36.80[c]	2.394	C

[a] Calculated by the compiler.

[b] The solid phases are: A = $Ga(IO_3)_3 \cdot 2H_2O$; B = solid solution based on $Ga(IO_3)_3 \cdot 2H_2O$; C = $Ga_2(SO_4)_3 \cdot 18H_2O$.

[c] For binary systems the compiler computes the following values:

Solubility of $Ga(IO_3)_3$ = 0.014 mol kg^{-1}
Solubility of $Ga_2(SO_4)_3$ = 1.362 mol kg^{-1}.

[d] Eutonic point at 0.19 mass % $Gd(IO_3)_3$ and 35.65 mass % $Ga_2(SO_4)_3$.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached in 20–30 d. In the liquid and solid phases, the iodate content was determined iodometrically, and gallium was determined by complexometric titration with PAN indicator. The quantity of sulfate ion was calculated by the difference. When the sample of solid phase contained less than 2 mass % sulfate ion, its concentration was also determined gravimetrically. The solid phases were identified by the "residues" method, and the results were confirmed form the X-ray diffraction patterns recorded on a diffractometer.

Source and Purity of Materials:

Gallium iodate dihydrate was prepared by the reaction of gallium sulfate with iodic acid. Anhydrous gallium sulfate of high purity was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.1 K.

Components:
(1) Cadmium iodate; $\mathrm{Cd(IO_3)_2}$; [7790-81-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. November 2001.

Critical Evaluation:

The Binary System

Solubility data for $\mathrm{Cd(IO_3)_2}$ in water have been reported in 4 publications (1–3, 6). These studies deal with ternary systems, and the solubility in the binary $\mathrm{Cd(IO_3)_2}$–H_2O system is given as one data point in the ternary systems.

Saegusa (1) and Miyamoto (2) determined the solubility at 298 K by iodometric titration. The result of Saegusa is 10 % larger than that of Miyamoto. The arithmetic mean of the 2 investigators is 2.0×10^{-3} mol dm^{-3} and this result is designated as a tentative solubility value.

The value of 1.5×10^{-3} mol kg^{-1} by Oelke and Wagner (6) differs from 2.109×10^{-3} mol kg^{-1} by Saegusa (1), both at 298 K. The value of 1.5×10^{-3} mol kg^{-1} at 323 K by Lepeshkov et al. (3) is smaller than that at 298 K of (1), therefore the result of (3) should be treated as doubtful. It seems by analogy to the molar scale that the $\mathrm{Cd(IO_3)_2}$ solubility at 298 K is lower than in (1) and higher than in (6). Composition of the equilibrium solid phase is known as anhydrous $\mathrm{Cd(IO_3)_2}$ at 323 K (3).

The Multicomponent System

Saegusa (1) measured the solubilities of $\mathrm{Cd(IO_3)_2}$ in aqueous potassium nitrate solutions at 298 K. The solubility increases with an increase of the potassium nitrate concentration. From the solubility data, the mean activity coefficients of $\mathrm{Cd(IO_3)_2}$ were calculated. Miyamoto (2) measured the solubilities of $\mathrm{Cd(IO_3)_2}$ in tetrahydrofuran–water mixtures at 298 K. The logarithm of the solubility of the iodate decreases almost linearly with the reciprocal dielectric constant of the solvent mixture. Ramette (4, 5) studied the solubilities of $\mathrm{Cd(IO_3)_2}$ in cadmium perchlorate solution containing the appropriate amount of sodium perchlorate. The ionic strength of the solution was adjusted at 1.00 (4) or 3.00 (5). The solubility product at 298 K and the $\mathrm{CdIO_3^+}$ formation constant were reported (4). The author of (5) also studied the solubilities of $\mathrm{Cd(IO_3)_2}$ in sodium bromide and sodium perchlorate solutions, both at an ionic strength 3.00. In each system, sodium perchlorate was used to adjust the ionic strength. The solubility products at 298 and 303 K reported in (5) showed distinct increase of the solubility with temperature thus questioning the result of (3) obtained in water. Oelke and Wagner (6) have reported the solubilities of $\mathrm{Cd(IO_3)_2}$ in solutions of potassium chloride and magnesium sulfate. The data were graphically presented. The values read out by the compilers show qualitative agreement with the results of (1), (4) and (5). Generally, the solubility of $Cd(IO_3)_2$ increases in a presence of inert salts (more weakly in divalent–divalent salts) but decreases in a presence of cadmium ions in the solution.

Lepeshkov et al. (3) have reported the solubility study for the ternary $Cd(IO_3)_2$–HIO_3–H_2O system at 323 K. The system is of simple eutonic type and a solid double compound $2HIO_3 \cdot Cd(IO_3)_2$ is formed. The compilation of (3) was inserted in (7).

References:

1. F. Saegusa, Nippon Kagaku Zasshi **71**, 223 (1950).
2. H. Miyamoto, Nippon Kagaku Kaishi **1972**, 659.
3. I. N. Lepeshkov, E. E. Vinogradov, and I. M. Karataeva, Zh. Neorg. Khim. **24**, 2540 (1979); Russ. J. Inorg. Chem. (Engl. Transl.) **24**, 1412 (1979).
4. R. W. Ramette, Anal. Chem. **53**, 2244 (1981),
5. R. W. Ramette, Anal. Chem. **55**, 1232 (1983).
6. W. C. Oelke and C. Wagner, Proc. Iowa Acad. Sci. **46**, 187 (1939).
7. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 481.

Components:

(1) Cadmium iodate; $\mathrm{Cd(IO_3)_2}$; [7790-81-0]
(2) Potassium chloride; KCl; [7447-40-7]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

W. C. Oelke and C. Wagner, Proc. Iowa Acad. Sci. **46**, 187–8 (1939).

Variables:

Concentration of KCl or $MgSO_4$
Temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

Numerical data were not reported in the original paper, so they were read out from the figure by the compilers. The solubility curves at 298 K were shown in the original figure by Oelke and Wagner. The solubility data has also been given in a book edited by Linke (1). Both numerical values data were cited in Table 96 and the solubility values cited in (1) slightly differed from our findings. The compilers think that there is no reason to doubt the results reported by Linke (1) for the difference in our findings

TABLE 96. Solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous KCl and $MgSO_4$ solutions at 298 K

Added salt	m_2 / mol kg^{-1}	m_1 / mol kg^{-1} [a]	m_1 / mol kg^{-1} [b]
None	—	0.0012	0.0015
KCl	0.001	—	0.0016
	0.01	—	0.0019
	0.02	0.0022	—
	0.05	—	0.0029
	0.06	0.0031	—
	0.1	—	0.0040
	0.5	0.009	0.0093
	1.0	0.016	0.00162
	2.5	0.046	0.045
$MgSO_4$	0.004	—	0.0020
	0.01	—	0.0023
	0.02	0.0026	—
	0.06	0.0035	—
	0.5	0.006	0.0061
	1.0	0.0075	0.0074
	2.0	—	0.0086
	2.5	0.009	—

[a] Estimated from the curves given by Oelke and Wagner, and cited in (1).
[b] Reading out from the figure in the original paper by the compilers.

The equilibrium solid phase was not specified.

Auxiliary Information

Method/Apparatus/Procedure:
Saturation of the solutions was obtained by shaking of the salts with the solutions for a long time. Analyses of the saturated solutions were made by using volumetric method (iodometry) with carefully calibrated burets.

Source and Purity of Materials:
The materials were prepared by methods similar to those used by Pearce and Oelke (2); the salts were purified by multiple recrystallization.

Estimated Error:
Solubility: reading-out procedure ± 0.0003 mol kg^{-1} (compilers).
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1958) Vol. 1, p. 742.
[2] J. N. Pearce and W. C. Oelke, J. Phys. Chem. **42**, 95 (1938).

Components:
(1) Cadmium iodate; $\mathrm{Cd(IO_3)_2}$; [7790-81-0]
(2) Potassium nitrate; KNO_3; [7757-79-1]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
F. Saegusa, Nippon Kagaku Zasshi **71**, 223–6 (1950).

Variables:
Concentration of KNO_3: 0–1.0 mol dm^{-3}
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 97. Solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous KNO_3 solutions at 25 °C

c_2 / mol dm^{-3}	m_2 / mol kg^{-1}	$10^3 c_1$ / mol dm^{-3}	$10^3 m_1$ / mol kg^{-1}	$10^5 x_1$	ρ / g cm^{-3}
0	0	2.104	2.109	3.799	0.9985
0.002523	0.002529	2.166	2.171	3.910	0.9987
0.005045	0.005058	2.217	2.223	4.003	0.9989
0.01001	0.01004	2.310	2.317	4.159	0.9991
0.05057	0.05080	2.718	2.730	4.908	1.0019
0.1012	0.1019	3.055	3.075	5.518	1.0052
0.2496	0.2527	3.662	3.708	6.618	1.0145
0.4790	0.4896	4.252	4.346	7.692	1.0287
0.7487	0.7739	4.841	5.005	8.768	1.0454
0.9581	0.9995	5.161	5.384	9.360	1.0578

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:
Excess of cadmium iodate crystals and aqueous potassium nitrate solutions were placed in glass-stoppered bottles coated with paraffin. The bottles were rotated in a thermostat at the selected temperature for 15 h. Aliquots of the saturated solutions were filtered through cotton wool, and the iodate content was determined iodometrically.

Source and Purity of Materials:
Cadmium iodate was prepared by adding aqueous cadmium chloride solution to aqueous potassium iodate solution. The precipitate was washed and dried. Guaranteed reagent potassium nitrate was recrystallized twice from water. The conductivity water was used.

Estimated Error:
Nothing specified.

Components:
(1) Cadmium iodate; $Cd(IO_3)_2$; [7790-81-0]
(2) Tetrahydrofuran; C_4H_8O; [109-99-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, Nippon Kagaku Kaishi **1971**, 659–61.

Variables:
Concentration of tetrahydrofuran: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 98. Solubility of $Cd(IO_3)_2$ in tetrahydrofuran–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^4c_1 / mol dm^{-3}
0	0	18.40
5	1.3	13.75
10	2.7	9.87
15	4.2	7.11
20	5.9	5.17
25	7.7	3.84
30	9.7	2.85
40	14.3	1.43

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:
An excess of cadmium iodate crystals and the solvent mixtures were placed in glass-stoppered bottles. The bottles were rotated in a thermostat at constant temperature for 48 h. After the saturated solutions were allowed to settle, the solutions were withdrawn through a siphon tube equipped with a sintered-glass filter. The iodate content in the saturated solutions was determined iodometrically.

Source and Purity of Materials:
Anhydrous cadmium iodate was prepared by dropping solutions of equivalent quantities of cadmium nitrate (Wako guaranteed reagent) and potassium iodate (Wako guaranteed reagent) in a large volume of aqueous potassium nitrate solution. The precipitate was filtered, washed, and dried under reduced pressure. Tetrahydrofuran was distilled over sodium hydroxide and then redistilled over sodium metal.

Estimated Error:
Solubility: precision between 0.2 and 3.4 % (compiler).
Temperature: precision ± 0.02 K.

Components:

(1) Cadmium iodate; $\mathrm{Cd(IO_3)_2}$; [7790-81-0]
(2) Cadmium perchlorate; $\mathrm{Cd(ClO_4)_2}$; [13760-37-7]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

R. W. Ramette, Anal. Chem. **53**, 2244–6 (1981).

Variables:

Concentration of $\mathrm{Cd(ClO_4)_2}$ and $NaClO_4$ to give ionic strength of 1.00
One temperature: 298.21 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 99. Solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous solutions containing cadmium and sodium perchlorates at ionic strength of 1.00 at 25.06 °C

10^3c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3} [a]	10^3c_1 / mol dm^{-3} [b]
9.814	1.3031	1.3016
	1.3022	
	1.2995	
	(av.) 1.3016	
19.839	0.9776	0.9796
	0.9794	
	0.9782	
	(av.) 0.9784	
29.587	0.8378	0.8340
	0.8367	
	0.8368	
	(av.) 0.8371	
39.382	0.7461	0.7471
	0.7452	
	0.7450	
	(av.) 0.7454	
46.165	0.6897	0.6891
	0.6882	
	0.6889	
	(av.) 0.6889	

[a] The first value in each group is the result of a single pass of solution through the cadmium iodate bed, while the second and third results were obtained by using solution which had been recycled through the bed once and twice, respectively. The values indicated by (av.) were calculated by the compiler.

[b] Calculated by the author. Details of the treatment may be found in the original paper.

The author fitted the solubility data to a smoothing equation assuming that the species in solution were Cd^{2+}, $CdIO_3^+$ and $\mathrm{Cd(IO_3)_3}$. Neglecting activity coefficients in these solutions of the final ionic strength $I = 1.0$, excellent fits were obtained based on the agreement between the observed and calculated solubilities.

The solubility product of $\mathrm{Ca(IO_3)_2}$ in water at 25.06 $^\circ\mathrm{C}$ and ionic strength at 1.00 is $(6.94 \pm 0.02) \times 10^{-8}$ mol^3 dm^{-9}.

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility vessel consisted a fine porosity sealing tube, the upper half of which was packed with $\mathrm{Cd(IO_3)_2}$. Small volumes of solutions of $\mathrm{Cd(ClO_4)_2}$ and $NaClO_4$ were added to the top of the tube, and the fitted tube placed in a screw-capped test tube. The sealed assembly was placed in a water bath of controlled temperature. The initial output of solution was discarded, and the inner fritted tubes refilled with fresh solvent. Aliquots of 0.0005 cm^3 were withdrawn for analysis with a calibrated transfer pipet. Saturated solutions were coulometrically analyzed for iodate. Calibration of method was performed using recrystallized KIO_3. The densities of the saturated solutions were also determined but not published.

Source and Purity of Materials:

Cadmium iodate was precipitated by dropwise and simultaneous addition of equal volumes of 0.4 mol dm^{-3} cadmium nitrate and warm 0.8 mol dm^{-3} sodium iodate solution to gently boiling 1 mol dm^{-3} nitric acid. During the precipitation the mixture was stirred vigorously with a magnetic stirrer. The product was washed 4 times by decantation with water before being placed in a Soxhlet extractor. Over a period of 3 weeks, the hot water Soxhlet extraction transferred the cadmium iodate to the boiling flash. Reagent grade cadmium perchlorate was used. $\mathrm{Cd(ClO_4)_2}$ stock solution was standardized on a mass basis by controlled potential coulometry using a mercury cathode. Redistilled water from alkaline permanganate was used.

Estimated Error:

Solubility: Standard deviation in the determination of iodate was lower than 0.05 %; accuracy of the analysis ± 0.02 %.

Temperature: stability ± 0.005 K.

Components:

(1) Cadmium iodate; $\mathrm{Cd(IO_3)_2}$; [7790-81-0]
(2) Cadmium perchlorate; $\mathrm{Cd(ClO_4)_2}$; [13760-37-7]
or (2) Sodium bromide; NaBr: [7647-15-6]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

R. W. Ramette, Anal. Chem. **55**, 1232–6 (1983).

Variables:

Concentration of $\mathrm{Cd(ClO_4)_2}$ or NaBr and $NaClO_4$ to give ionic strength of 3.00
Temperature: 298 and 308 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 100. Solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous cadmium perchlorate solutions at ionic strength of 3.00 with sodium perchlorate at 25 and 35 °C

t / °C	$10^2 c_2$ / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3} [a]	$10^3 c_1$ / mol dm^{-3} [b]
25	1.0836	0.9352	0.9363
	2.1816	0.7010	0.6992
	3.2373	0.5961	0.5958
	4.3448	0.5304	0.5324
	5.4196	0.4929	0.4919
35	1.0836	1.020	1.022
	2.1816	0.7675	0.7657
	3.2373	0.6550	0.6534
	4.3448	0.5830	0.5845
	5.4196	0.5406	0.5405

[a] Observed.

[b] Calculated by the author from the thermodynamical consideration.

From the solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous cadmium perchlorate solutions at ion strength of 3.00 the solubility products of $\mathrm{Cd(IO_3)_2}$ are given as follows: $K_\mathrm{s} = (3.81 \pm 0.03) \times 10^{-8}\mathrm{mol^3dm^{-9}}$ at 25 °C and $K_\mathrm{s} = (4.55 \pm 0.03) \times 10^{-8}\mathrm{mol^3dm^{-9}}$ at 35 °C.

TABLE 101. Solubility of $\mathrm{Cd(IO_3)_2}$ in aqueous sodium bromide solutions at ionic strength of 3.00 with sodium perchlorate at 25 and 35 °C

t / °C	c_2 / mol dm^{-3}	[Br$^-$]$_\mathrm{eqil}$ / mol dm^{-3}	10^3c_1 / mol dm^{-3} [a]	10^3c_1 / mol dm^{-3} [b]
25	0.02046	0.01870	2.810	2.808
	0.03027	0.02786	3.076	3.081
	0.04462	0.04129	3.459	3.453
	0.06633	0.06158	3.973	3.983
	0.09891	0.09180	4.760	4.757
	0.14864	0.13723	5.976	5.967
	0.22387	0.20452	7.946	7.958
	0.33357	0.30054	11.25	11.24
	0.48820	0.43333	16.50	16.49
	0.73138	0.63832	25.77	25.83
	1.1008	0.94342	41.84	41.78
35	0.02038	0.01858	2.964	2.960
	0.03018	0.02767	3.236	3.244
	0.04448	0.04100	3.637	3.631
	0.06613	0.6113	4.180	4.187
	0.09861	0.9106	5.004	5.003
	0.14819	0.13599	6.302	6.289
	0.22319	0.20241	8.406	8.417
	0.33255	0.29707	11.934	11.929
	0.48669	0.42787	17.57	17.55
	0.7291	0.62958	27.43	27.52
	1.0973	0.92937	44.59	44.50

[a] Observed.

[b] Calculated by the author from the thermodynamical consideration.

The author also computed Gibbs energies, enthalpies and entropies of the solution based on this least squares smoothing equation applied to two temperatures: the reader is referred to the original literature in event that these calculations are of interest.

The equilibrium solid phase was not analyzed.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of cadmium iodate was determined using high accuracy controlled potential coulometer in solution of cadmium perchlorate or sodium bromide, all at ionic strength 3.00 with sodium perchlorate as inert electrolyte. The details of the techniques for sampling, the equipment and the procedure for coulometric analysis have been described in (1). The author fitted the solubility data to a smoothing equation assuming the species in solution were Cd^{2+}, $CdIO_3^+$, $\mathrm{Cd(IO_3)_2}$ and $\mathrm{Cd(IO_3)_n^{2-n}}$ for n = 1 and 4. Good fits were obtained based on the agreement between observed and calculated solubilities. Densities of the solutions were determined by weighing of precise volumes delivered by standardized and siliconized pipet.

Source and Purity of Materials:

Cadmium iodate was prepared by dropwise and simultaneous addition of dilute solutions of cadmium nitrate and sodium iodate to gently boiling dilute nitric acid. The product was recrystallized from water. Reagent grade cadmium perchlorate, sodium bromide and sodium perchlorate were dissolved in water. Water was redistilled from alkaline permanganate.

Estimated Error:

Solubility: standard deviation 0.2 %; reproducibility ± 0.1 %.
Solubility product: precision ± 2 %.
Temperature: precision ± 0.005 K.

References:

[1] R. W. Ramette, Anal. Chem. **53**, 2244 (1981).

Components:

(1) Indium iodate; $In(IO_3)_3$; [13510-34-4]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Solubility data for $In(IO_3)_3$ in water have been reported in 6 publications (1–6). Ensslin (1) has measured the solubility of $In(IO_3)_3$ at 293 K, and obtained the value of 8.3×10^{-4} mol dm^{-3}. This value is 15 % lower than one obtained earlier by Mathers and Schluederberg (6). Solubility data at 298 K have been reported in 4 publications (2–5). Campbell (2) studied the solubility by conductometry, and the value of 1.027×10^{-3} mol dm^{-3} was reported. A group of Shklovskaya (3–5) studied the solubility by chemical analysis. These investigations deal with the ternary systems and the binary system is given as one point on phase diagrams; the solubility of 7.7×10^{-4} mol kg^{-1} was reported by this group. The result of Campbell is larger than that of a group of Shklovskaya, and moreover Campbell pointed out that the hydrolysis of $In(IO_3)_3$ occurs. This would suggest a higher reliability of the result of Shklovskaya et al., however then we should accept a decrease of the solubility with temperature increase taking into account the higher solubilities results of (1) and (6) at 293 K. Thus acceptance of the result of Campbell (2) as tentative seems to be more rational. The stable solid in equilibrium with the saturated solution at 298 K was found to be $In(IO_3)_3 \cdot H_2O$ according to (3–5) whereas (1) reported a solid containing much more water. Anhydrous $In(IO_3)_3$ is formed over 398 K according to (1).

The Ternary System

Solubility studies for the ternary aqueous systems have been reported in 3 publications (3–5), which are summarized in Table 102.

TABLE 102. Summary of solubility studies of $In(IO_3)_3$ in the aqueous systems at 298 K

Ternary system	Solid phases	Reference
$In(IO_3)_3$–HIO_3–H_2O	$In(IO_3)_3 \cdot H_2O$; HIO_3	(4)
$In(IO_3)_3$–$LiIO_3$–H_2O	$In(IO_3)_3 \cdot H_2O$; $LiIO_3 \cdot In(IO_3)_3 \cdot H_2O$; $2LiIO_3 \cdot In(IO_3)_3 \cdot H_2O$; $LiIO_3$	(3), compiled in (7)
$In(IO_3)_3$–$Al(IO_3)_3$–H_2O	$In(IO_3)_3 \cdot H_2O$; solid solution based on $In(IO_3)_3 \cdot H_2O$; $Al(IO_3)_3 \cdot 6H_2O$	(5), see the ternary $Al(IO_3)_3$–$In(IO_3)_3$–H_2O system in this volume

References:

1. F. Ensslin, Anorg. Allg. Chem. **250**, 199 (1942).
2. A. N. Campbell, Can. J. Chem. **52**, 3769 (1974).
3. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, G. A. Poleva, and V. A. Kuzina, Zh. Neorg. Khim. **26**, 791 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 425 (1981).
4. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, A. G. Tokareva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 2181 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 1240 (1985).
5. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, B. I. Kidyarov, V. A. Kuzina, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **33**, 219 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 122 (1988).
6. F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc. **30**, 211 (1908).
7. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 302.

Components:
(1) Indium iodate; $\mathrm{In(IO_3)_3}$; [13510-34-4]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc. **30**, 211–5 (1908).

Variables:
Addition of 16.7 mass % HNO_3
Two temperatures: 293 and 353 K

Prepared By:
Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Indium iodate is soluble in water in mass ratio of $1 : 1500$ at 20 °C. The compilers recalculated this result and obtained 1.05×10^{-3} mol kg^{-1}. Indium iodate is soluble in $1 : 5$ (16.7 mass %) nitric acid solution in mass ratio of $1 : 150$ at 80 °C. The compilers recalculated this value and obtained 0.67 mass % or about 0.01 mol dm^{-3} $\mathrm{In(IO_3)_3}$ in about 3 mol dm^{-3} HNO_3. The authors stated that indium iodate is soluble in dilute sulfuric acid (no numerical result reported) and in hydrochloric acid solution but the latter causes a decomposition of the salt with liberation of chlorine. No equilibrium solid phases were analyzed.

Auxiliary Information

Method/Apparatus/Procedure:
Method of the solubility determination was not exactly described but it is sure that indium content was analyzed by precipitation of its hydroxide with ammonia; finally indium oxide was weighed. Iodate was determined by titration of iodine (that was liberated when a sample was treated with potassium iodide and dilute sulfuric acid) with sodium thiosulfate.

Source and Purity of Materials:
Equivalent quantities of anhydrous indium trichloride (self made) and potassium iodate (recrystallized twice from water) were dissolved separately and mixed. The entire solution was evaporated to dryness and the residue collected was washed with cold water. The precipitate was dried over sulfuric acid. The substance was then dissolved in a boiling nitric acid. On evaporation of this solution the crystals formed were washed with water and dried in a vacuum desiccator. Theoretical content of the product: 21.71 % In_2O_3 and 59.53 % I; analysis of the product: 21.76, 21.93 % In_2O_3 and 59.19, 59.30 % I.

Estimated Error:
Nothing specified.

Components:
(1) Indium iodate; $\mathrm{In(IO_3)_3}$; [13510-34-4]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
F. Ensslin, Z. Anorg. Allg. Chem. **250**, 199–201 (1942)

Variables:
Concentration of HNO_3; 0–14.3 mol dm^{-3}
Temperature: 293 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 103. Solubility of $\mathrm{In(IO_3)_3}$ in solutions of nitric acid at 20 °C

γ_2 / g dm^{-3}	c_2 / mol dm^{-3} [a]	γ_1 / g dm^{-3}	10^3c_1 / mol dm^{-3} [a]	ρ / g cm^{-3}
0	0	0.53	0.83	1.000
84	1.3	7.80	12.20	1.048
142	2.25	10.90	17.04	1.080
278	4.41	15.99	25.00	1.158
300	4.76	16.89	26.41	1.167
376	5.97	14.69	22.97	1.200
515	8.17	11.10	17.36	1.259
704	11.2	6.02	9.41	1.329
900	14.3	0.72	1.13	1.396

[a] Calculated by the compiler.

The phase precipitated from the saturated solution contained: 11.58 % In and 52.91 mass % IO_3. This gives $\mathrm{In} : \mathrm{IO_3} : \mathrm{H_2O}$ molar ratio $1 : 3.3 : 19.5$. Anhydrous $\mathrm{In(IO_3)_3}$ is formed over 125 °C.

Auxiliary Information

Method/Apparatus/Procedure:
No information about the experimental method was reported.

Source and Purity of Materials:
Indium iodate was precipitated from aqueous solutions of indium sulfate with equivalent amount of potassium iodate. The product was filtered and dried by heating at 125 °C. The anhydrous iodate was obtained by crystallization from a dilute nitric acid solution. Theoretical composition: 17.94 % In and 82.06 mass % IO_3. An analysis of the salt gave 17.75 % In and 82.22 mass % IO_3 what corresponds to In:IO_3 = 1:3. The salt was stored over P_2O_5.

Estimated Error:
Nothing specified.

Components:

(1) Indium iodate; $\mathrm{In(IO_3)_3}$; [13510-34-4]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

A. N. Campbell, Can. J. Chem. **52**, 3769–72 (1974).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{In(IO_3)_3}$ in water at 25 $^\circ\mathrm{C}$ is: 0.6568 g dm^{-3} (author) and 1.027×10^{-3} mol dm^{-3} (compilers); the corresponding K_s was reported as 3.007×10^{-4} mol^4 dm^{-12} but should be 3.004×10^{-11} mol^4 dm^{-12} according to calculation of the compilers. The solubility value for $\mathrm{In(IO_3)_3}$ is a maximum value because of the hydrolysis of salt. Composition of the equilibrium solid phase was not specified.

Auxiliary Information

Method/Apparatus/Procedure:

Excess of powdered $\mathrm{In(IO_3)_3}$ was placed in a conductance cell filled with water. At intervals of 24 h, measurements of conductance of the solutions were taken until a maximum value occurred. The liquid was then poured off, fresh water added, and the procedure repeated. Finally a state of affairs was reached when the conductance did not increase on standing and this was accepted as the true conductance of a saturated solution.

Source and Purity of Materials:

Indium iodate was prepared by its precipitation from indium trichloride with potassium iodate. The precipitate was washed thoroughly with hot water but this procedure is insufficient to remove all traces of soluble electrolytes. Conductivity water was used in the experiments.

Estimated Error:

Nothing specified.

Components:

(1) Indium iodate; $\mathrm{In(IO_3)_3}$; [13510-34-4]

(2) Iodic acid; HIO_3; [7782-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, A. G. Tokareva, and T. E. Vdovkina, Zh. Neorg. Khim. **30**, 2181–2 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 1240 (1985).

Variables:

Composition of the salt and acid

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 104. Composition of saturated solutions in the ternary $\mathrm{In(IO_3)_3}$–HIO_3–H_2O system at 25 °C

$\mathrm{In(IO_3)_3}$		HIO_3		Solid phase[b]
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	
0.049 [c]	1.4×10^{-3}	—	—	A
0.0052	1.6×10^{-4}	9.05	1.01	A
0.0087	2.9×10^{-4}	16.49	1.982	A
0.022	7.9×10^{-4}	23.72	3.087	A
0.081	3.5×10^{-3}	38.25	5.972	A
0.15	7.5×10^{-3}	48.26	8.748	A
0.18	9.9×10^{-3}	54.04	10.78	A
0.25	1.7×10^{-2}	65.54	16.40	A
0.28	2.1×10^{-2}	69.39	18.98	A
0.30 [d]	2.6×10^{-2}	74.25 [d]	23.00	A+B
—	—	75.10 [c]	23.60	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{In(IO_3)_3 \cdot H_2O}$; B = HIO_3

[c] For binary systems the compilers compute the following values:

Solubility of $\mathrm{In(IO_3)_3}$ = 7.7×10^{-4} mol kg^{-1}
Solubility of HIO_3 = 17.17 mol kg^{-1}.

[d] Eutonic point

Auxiliary Information

Method/Apparatus/Procedure:

The ternary $In(IO_3)_3$–HIO_3–H_2O system was studied by isothermal method. Reaching of the equilibrium took 20–30 d. Iodate ion was determined iodometrically in sample of the coexisting phases of the system. The indium content was determined by complexometric titration with xylenol orange indicator in the solid residues and by absorption flame spectrometry in the liquid phase. The concentrations of iodic solid in the sample were found by the difference. The solid phases were identified by the "residues" method, and the results were confirmed by X-ray diffraction.

Source and Purity of Materials:

Indium iodate monohydrate was obtained by the reaction of indium nitrate with iodic acid. Chemically pure grade iodic acid was recrystallized.

Estimated Error:

Solubility: precision no better than few % (compilers).
Temperature: precision ± 0.1 K (authors (1)).

References:

[1] R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and T. V. Daminova, Zh. Neorg. Khim. **30**, 2180 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 1239 (1985).

F. Mercury(I) Iodate

Components:

(1) Mercury(I) iodate; $Hg_2(IO_3)_2$; [13465-35-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. November 2001.

Critical Evaluation:

The solubility studies of mercury(I) iodate in water have been reported in 4 original papers (1–4). The solubility of some sparingly soluble mercury salts containing mercury halates have also been compiled and evaluated by Clever et al. (5).

The solubility value of Spacu and Spacu (2) was 4.4×10^{-5} mol dm^{-3} at 288.7 K. The result reported by Kryukova (3) was 6.0×10^{-7} (with no units given) at 298 K. This value is cited by Linke (6) using units of g dm^{-3}. If these units are correct, the evaluators obtain the solubility value of 8.0×10^{-10} mol dm^{-3} by converting the experimental solubility data units. While, Clever et al. (5) stated that the value reported by Kryukova was in mol dm^{-3} units. The evaluators assume that the value of 6.0×10^{-7} mol dm^{-3} is more probable because in a table inserted in the paper (3) the solubility result quoted from Brodsky (1) was expressed in mol dm^{-3} units (6.9×10^{-7}). The reported value of Spacu and Spacu (2) as well as of Takacs (4), 1.7×10^{-5} mol dm^{-3}, are two orders of magnitude higher at 288.7 K than the value of Brodsky (1) and Kryukova (3) at 298 K. Taking into account the ambiguity of the Kryukova's (3) result we may suggest the result of Brodsky (1) 7×10^{-7} mol dm^{-3} as doubtful value of the $Hg_2(IO_3)_2$ solubility at 298 K.

The solubility products of mercury(I) iodate in water have been reported in 3 original papers (1, 2, 4). The reported solubility products were 3.4×10^{-13} at 288.7 K (2) and 1.29×10^{-18} (1), and 1.94×10^{-14} mol^3 dm^{-9} (4) both at 298 K. No arithmetic mean of the values (1) and (4) may be done for designation of even tentative solubility product at 298 K. Two solubility products of 3.0×10^{-14} mol^3 dm^{-9} and 3.0×10^{-20} mol^3 dm^{-9} were also reported by Clever et al. (5). They stated that the former value was calculated from a recommended and probably the most reliable E^0 values reported by Charlot et al. (7), and the original source of the latter value is not clear. These values are not taken into account because the corresponding sheets were not prepared.

Anhydrous $Hg_2(IO_3)_2$ is the equilibrium solid phase.

References:

1. A. E. Brodsky, Z. Elektrochem. **35**, 833 (1929); J. F. Spencer, Z. Phys. Chem. **80**, 701 (1912).
2. G. Spacu and P. Spacu, Z. Anal. Chem. **96**, 30 (1934).
3. T. A. Kryukova, Zh. Fiz. Khim. **13**, 693 (1939).
4. I. Takacs, Magy. Chem. Foly. **49**, 33, 100 (1943).
5. H. L. Clever, S. A. Johnson, M. E. Derrick, J. Phys. Chem. Ref. Data **14**, 631 (1985).

6. W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1958) Vol. 1, p. 1243.

7. G. Charlot, A. Collumeau, and M. J. C. Marchon, *Selected Constants, Oxidation–Reduction Potentials of Inorganic Substances in Aqueous Solution* (Butterworths, London, 1971).

Components:
(1) Mercury(I) iodate; $Hg_2(IO_3)_2$; [13465-35-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
A. E. Brodsky, Z. Elektrochem. **35**, 833–7 (1929).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The author calculated the solubility product of $Hg_2(IO_3)_2$ from the experimental data reported by Spencer (1). The solubility product of $Hg_2(IO_3)_2$ in water at 25 °C is given:

$$K_s = 1.29 \times 10^{-18}\ \text{mol}^3\ \text{dm}^{-9}$$

and

$$\text{Solubility} = 6.9 \times 10^{-7}\ \text{mol dm}^{-3}\ \text{(calculated by the compilers).}$$

Auxiliary Information

Method/Apparatus/Procedure:
The solubility product was determined from electromotive force measurements of (1) at constant temperature. The following cell was used:

$$\text{Hg} \,||\, Hg_2(IO_3)_2, \text{KIO}_3 \,|\, NH_4NO_3 \text{ (satd.)} \,|\, 0.1 \text{ mol dm}^{-3} \text{ KCl}, Hg_2Cl_2 \,||\, \text{Hg}.$$

Concentration of KIO_3 was changed between 9.7×10^{-4} and 6.25×10^{-2} mol dm^{-3}. The potentials were measured for 24 h to observe their stability. This author calculated the solubility product from a difference of the self-measured Hg/Hg_2^{2+} potential and that found for the cell described in the paper (1).

Source and Purity of Materials:
No information was reported in this paper as well as in (1).

Estimated Error:
Solubility: precision no better than ± 1 % (compilers).
Temperature: nothing specified.

References:
[1] J. F. Spencer, Z. Phys. Chem. **80**, 701 (1912).

Components:
(1) Mercury(I) iodate; $\mathrm{Hg_2(IO_3)_2}$; [13465-35-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. Spacu and P. Spacu, Z. Anal. Chem. **96**, 30–4 (1934).

Variables:
One temperature: 288.7 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 105. Solubility and solubility product of $\mathrm{Hg_2(IO_3)_2}$ in water at 15.5 °C

γ_1 / mg dm^{-3}	$10^5 c_1$ / mol dm^{-3}	$10^{13} K_\mathrm{s}$ / mol^3 dm^{-9}
32.80	4.37	3.33
33.30	4.44	3.50
(av.) 33.05[a]	(av.) 4.41[a]	(av.) 3.42[a]

[a] Calculated by the compilers.

Results obtained at 16 °C during 0.5 h of the solution saturation have larger scatter and were further neglected by the authors.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of 0.15–0.17 g of mercury(I) iodate was shaken with 20 cm^3 of water at room temperature for 2 h. 10 cm^3 aliquots of the saturated solution after addition of 0.1 g KI and 0.5 cm^3 of 1 mol dm^{-3} H_2SO_4, were titrated with 0.005 mol dm^{-3} sodium thiosulfate solution using starch as an indicator.

Source and Purity of Materials:
Mercury(I) iodate was prepared by adding potassium iodate to mercury nitrate solution. The sediment was filtrated on a porcelain filter, washed with water, 96 % pure alcohol, ether and finally dried in vacuum.

Estimated Error:
Solubility: precision ± 0.2 %.
Temperature: nothing specified.

Components:

(1) Mercury(I) iodate; $\mathrm{Hg_2(IO_3)_2}$; [13465-35-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

T. A. Kryukova, Zn. Fiz. Kim. **13**, 693–700 (1939).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, E. V. Zagnit'ko, and Cezary Guminski

Experimental Data

Solubility of mercury(I) iodate in water 25 °C is given: 6.0×10^{-7} probably in mol $\mathrm{dm^{-3}}$. Information on concentration units is absent in a table of the original paper. By analogy with the results of $PbSO_4$ solubility determinations, the compilers would consider the concentration to be expressed in g $\mathrm{dm^{-3}}$.

On the other hand a solubility result quoted from Brodsky (2) agrees with the original value if mol $\mathrm{dm^{-3}}$ concentration name is assumed. Therefore mol $\mathrm{dm^{-3}}$ concentration name seems to be more probable.

Auxiliary Information

Method/Apparatus/Procedure:

Solubility was studied by polarographic method by means of a Russian made polarograph with a drop time of electrode of 5.7 s. Mercury dropping electrode was electrooxidized in solution of 0.001 mol $\mathrm{dm^{-3}}$ KIO_3 and 0.2 mol $\mathrm{dm^{-3}}$ KNO_3. The resulting polarographic curve showed 2 waves, corresponding to formation of soluble $\mathrm{Hg_2(NO_3)_2}$ and sparingly soluble $\mathrm{Hg_2(IO_3)_2}$, respectively. The half-wave potential differences formed a straight line relationship in relation to various Hg(I) salt solubilities. Based on the potential difference for $\mathrm{Hg_2(IO_3)_2}$, its solubility was read from this plot. The straight line relationship was based on literature data from (1) and (2) for $\mathrm{Hg_2Cl_2}$, $\mathrm{Hg_2Br_2}$ and $\mathrm{Hg_2I_2}$.

Source and Purity of Materials:

No information is reported.

Estimated Error:

Nothing specified. The determination has a relative character because the author used literature data for the plot: solubility – potential difference.

References:

[1] M. S. Sherrill, Z. Phys. Chem. **43**, 705 (1903).
[2] A. E. Brodsky, Z. Elektrochem. **35**, 833 (1929).

Components:

(1) Mercury(I) iodate; $\mathrm{Hg_2(IO_3)_2}$; [13465-35-5]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

I. Takacs, Magy. Chem. Foly. **49**, 33-51, 100-8 (1943).

Variables:

Concentration of iodate: 5×10^{-4}–0.3 mol dm^{-3}

One temperature: 298 K

Prepared By:

Jitka Eysseltova, Hiroshi Miyamoto, and Cezary Guminski

Experimental Data

TABLE 106. Solubility product of $\mathrm{Hg_2(IO_3)_2}$ in water at 25 °C

$10^2 c$ / mol dm^{-3} (concentration of IO_3^-)	γ (activity coefficient of IO_3^-)	E / V	$10^{14} K_s^\circ$ / mol^3 dm^{-9}
0.0495	1.000	0.2511	1.39[a]
0.1009	1.000	0.2328	1.39[a]
0.2080	0.932	0.2205	1.97
0.4982	0.897	0.1990	1.97
1.027	0.859	0.1804	1.80
2.003	0.814	0.1658	1.97
4.990	0.732	0.1451	1.98
10.11	0.667	0.1285	1.85
20.03	0.584	0.1152	1.98
29.96	0.520	0.1079	1.99
			(av.) 1.94

[a] The value has been rejected by the author owing to calculated average value.

The solubility calculated from the average K_s° value is 1.7×10^{-5} mol dm^{-3} (by the compiles).

Auxiliary Information

Method/Apparatus/Procedure:

The values of solubility product were derived from the electromotive forces of the following cell:

$$\text{Hg} \mid\mid \text{Hg}_2(\text{IO}_3)_2, \text{KIO}_3 \mid \text{KCl}, \text{Hg}_2\text{Cl}_2 \mid\mid \text{Hg}.$$

The solubility products were calculated from the equation,

$$E = \frac{0.05915}{2}\left(\log K_s - \log \gamma^2 c^2 - \log 1.84 \times 10^{-16}\right)$$

where E is the potential measured (corrected for the diffusion potential), and γ and c are the activity coefficient and the concentration of KIO_3, respectively. All electrodes were prepared with a special care to avoid a presence of oxygen and/or air moisture. Emf measurements were performed by means of compensation method. The mercury content in $Hg_2(IO_3)_2$ solution was gravimetrically determined as HgS, and the iodate content was determined iodometrically.

Source and Purity of Materials:

All starting materials were analytically pure from Merck or Kahlbaum, some of them (not specified) were recrystallized. Metallic mercury was stirred with HNO_3 for 24 h, washed with water, dried, and then distilled. Mercury iodate was prepared by adding potassium iodate solution to mercury(I) nitrate solution. The precipitate was washed with hot water and dried in a box at 50–60 °C. An analysis give: Hg–53.25, IO_3–46.50 mass %; calculated content for $Hg_2(IO_3)_2$: Hg–53.41, IO_3–46.59 mass %.

Estimated Error:

Solubility product: standard deviation 0.4 %.
Temperature: precision ± 0.05 K.

Components:
(1) Mercury(II) iodate; $Hg(IO_3)_2$; [7783-32-6]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. A. Piperaki and T. P. Hadjiioannou, Chim. Chron. New Series **6**, 375–84 (1977).

Variables:
One temperature: 303 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

The solubility of $Hg(IO_3)_2$ at 30 °C (temperature reported in (1)) was determined to be:

$$1.00 \times 10^{-4} \text{ mol dm}^{-3}.$$

This value is designated as a tentative solubility. These authors quoted also a value of 4.15×10^{-5} mol dm^{-3} but no reference followed this information. Probably, anhydrous $Hg(IO_3)_2$ is the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:
No information on equilibrium and sampling was reported. A semiautomatic titration method was used to analyze the concentration of mercury(II) in 10 cm^3 sample of the saturated $Hg(IO_3)_2$ solution. An excess of standard EDTA (0.01000 mol dm^{-3}) solution was added and the EDTA excess was back titrated with Mn(II) standard solution (0.0600 mol dm^{-3}) using calcein as an indicator.

Source and Purity of Materials:
All reagents were of reagent grade. The solutions were prepared from deionized, double distilled water.

Estimated Error:
Solubility: relative standard deviation 1–2 %.
Temperature: precision ± 0.05 K (authors (1)).

References:
[1] T. P. Hadjiioannou and T. A. Kephalas, Mikrochim. Acta (Wien) **6**, 1215 (1969).

Components:
(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. March 2002.

Critical Evaluation:

The Binary System

Data for the solubility of $TlIO_3$ in water have been reported in 14 publications (1–14). Except the works of Böttger (1) and Spencer (2), these studies deal with ternary (3–5, 7, 8, 10–14) and multi-component (6, 9) systems. The solubility was determined by chemical analyses of saturated solutions with exception of two works by Böttger (1) and Spencer (2) where the authors used instrumental methods. Böttger (1) determined the solubility of thallium iodate in water at 293 K from a conductivity measurement, and the solubility product was also calculated. Spencer (2) determined the activity solubility product of $TlIO_3$ in water at 298 K by a potentiometric measurement, and the solubility value of 2.12×10^{-3} mol dm^{-3} was obtained from the solubility product. The reported value is fairly larger than the results of other investigators (3–7, 9–11) because it was determined in presence of an unknown amount of $TlNO_3$. This solubility value was neglected in further formulation of the solubility equation.

A summary of the experimental solubility data for the binary $TlIO_3$–H_2O system is given in Table 107 and in the Fig. 11. Korenman et al. (12) have reported the solubility of $TlIO_3$ in water at not strictly defined temperature. The result of Ramette and Palmer (16) was obtained at pH = 4.

TABLE 107. Summary for solubility studies of $TlIO_3$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method^{d}	Reference
273	0.650	Iodometry ($Na_2S_2O_3$)	(10)
273	0.656	Iodometry ($Na_2S_2O_3$)	(11)
291.00	1.410	Iodometry ($Na_2S_2O_3$)	(8)
291.15	1.417	Iodometry ($Na_2S_2O_3$)	(8)
291–293	1.56^{a}	Iodometry ($Na_2S_2O_3$)	(12)
293.10	1.47	Conductometry	(1)
293	1.56	Iodometry ($Na_2S_2O_3$)	(13)
298.2	1.7^{b}	Iodometry (iodate content), Flame emission photometry and gravimetry (Tl content)	(14)
298	1.819	Iodometry ($Na_2S_2O_3$)	(4)
298.2	1.834$^{b\ c}$	Coulometry	(16)
298.15	1.834	Iodometry ($NaAsO_2$)	(9)
298	1.838	Iodometry ($Na_2S_2O_3$)	(10)

298	1.841	Iodometry ($NaAsO_2$)	(5)
298	1.842	Iodometry ($Na_2S_2O_3$)	(11)
298.15	1.843	Iodometry ($NaAsO_2$)	(6)
298	1.844	Iodometry ($NaAsO_2$)	(3)
298.15	1.850	Iodometry ($NaAsO_2$)	(7)
298	2.12[a]	Potentiometry	(2)
313	3.043	Iodometry ($Na_2S_2O_3$)	(10)
313	3.048	Iodometry ($Na_2S_2O_3$)	(11)

[a] Rejected values, see text

[b] mol kg^{-1} units

[c] at pH = 4

[d] The standard solution is indicated in the parentheses, except analytical method reported in reference 14.

The experimental solubilities based on mol dm^{-3} units except of one from (2) were fitted to the following simple smoothing equation:

$$\ln(c_1/\text{mol dm}^{-3}) = \frac{170726.029}{(T/\text{K})} + 1201.26625\ \ln(T/\text{K}) - 6806.12158 - 2.06978586(T/\text{K}) \quad (53)$$

where $\sigma_c = 7.7 \times 10^{-5}$.

From the smoothing equation, the recommended solubilities at round temperatures were calculated and the values are given in Table 108.

The solid phase equilibrated with the saturated solution is the anhydrous $TlIO_3$ salt (14) and no other informations were reported.

TABLE 108. Recommended solubility of $TlIO_3$ in water

T / K	$10^3 c_1$ / mol dm^{-3}
273	0.65
278	0.80
283	0.99
288	1.23
293	1.52
298	1.86
303	2.24
308	2.65
313	3.05

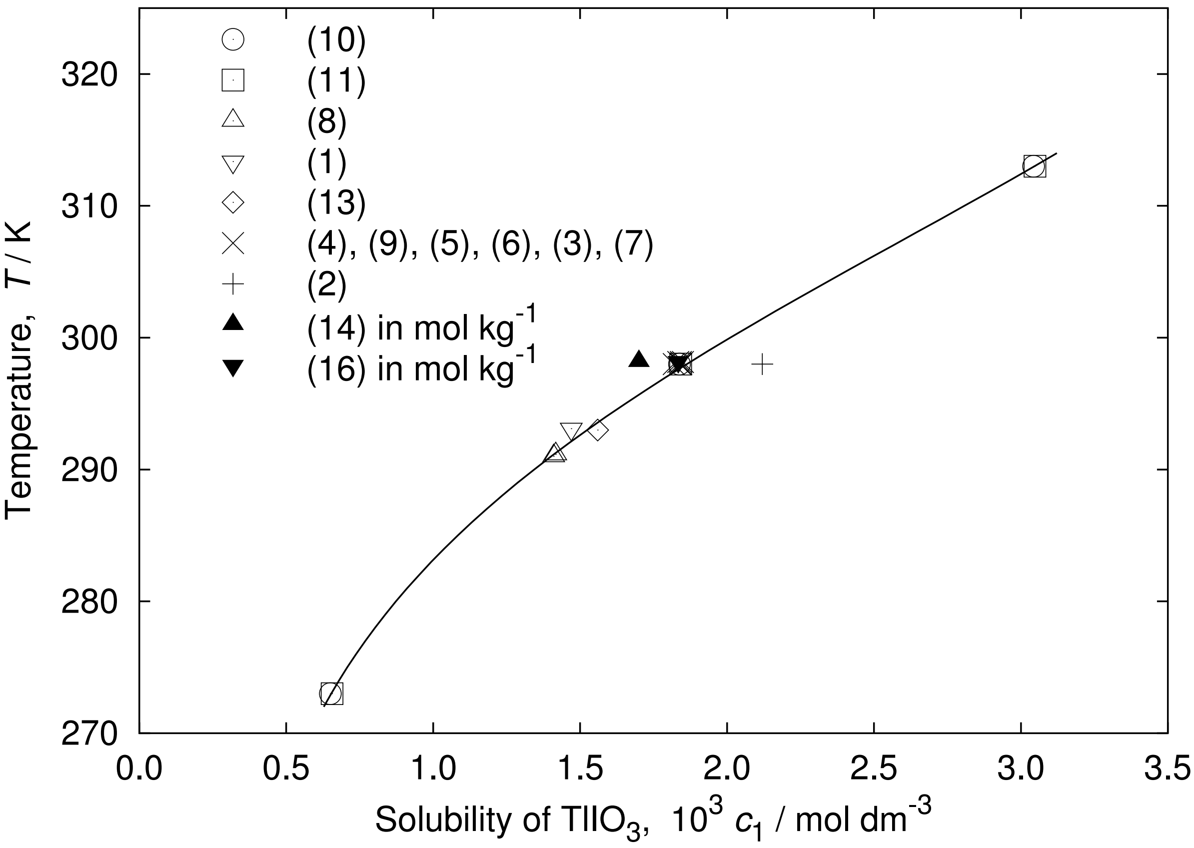

FIG. 11. Temperature dependence of solubility of $TlIO_3$ in water

The Ternary System

The solubilities in aqueous solutions of alkali metal hydroxides and various inorganic compounds have been reported in 6 publications (3, 4, 6, 10, 11, 16). A summary of experimental data for the aqueous ternary systems at 298 K is given in the following table.

TABLE 109. Solubility studies of $TlIO_3$ in water containing alkali metal hydroxides and various inorganic salts at 298 K

Added compound		$TlIO_3$ solubility range	Reference
Formula	c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}	
NaOH	0–0.1	1.843–2.003	(6)
KOH	0–0.08910	1.838–2.608	(10)
NaCl	0–0.10	1.844–2.620	(3)
NaCl	0–0.1	1.843–2.629	(6)
KCl	0–0.10	1.844–2.625	(3)
KCl	0–0.05422	1.838–2.359	(10)
$MgCl_2$	0–0.05	1.844–2.744	(3)
$MgCl_2$	0–0.05	1.819–2.755	(4)
$CdCl_2$	0–0.05	1.819–2.708	(4)
NaF	0–0.04646	1.838–2.185	(10)

$NaNO_3$	0–0.1	1.844–2.486	(3)
KNO_3	0–1.0	1.844–4.821	(3)
KNO_3	0–0.04279	1.843–2.207	(11)
$Mg(NO_3)_2$	0–0.05	1.844–2.621	(3)
Na_2SO_4	0–0.05	1.844–2.740	(3)
K_2SO_4	0–0.5	1.844–5.128	(3)
K_2SO_4	0–0.02861	1.838–2.486	(10)
$MgSO_4$	0–0.05	1.844–2.640	(3)
$MgSO_4$	0–0.05	1.819–2.625	(4)
$ZnSO_4$	0–0.05	1.844–2.789	(3)
$CdSO_4$	0–0.05	1.819–2.798	(4)
KSCN	0–0.05695	1.838–2.420	(10)
$K_4[Fe(CN)_6]$	0–0.006132	1.838–4.326	(10)
NaN_3	0–0.02705	1.843–2.119	(11)
$NaClO_4$	0.1[a]	2.410[a]	(16)
$HClO_4$	0.0001–0.1011[a]	1.834–2.801[a]	(16)

[a] in mol kg^{-1}

Generally the solubility of $TlIO_3$ increases with increasing the concentration of added salt in case of all systems at 298 K. Bell and George (10) and Nair and Nancollas (11) used the solubility data to determine dissociation constants for the ion pairs TlX (X = Cl, OH, SCN, F, NO_3 etc). Bell and George (10) and Nair and Nancollas (11) measured also the solubility in the inorganic salt solutions at 273 and 313 K. The results of La Mer and Goldman (3) were used to calculate the activity coefficients of $TlIO_3$ in aqueous solutions according to Davies equation (15). Korenman et al. (12) studied the solubility in aqueous solutions of nitrates and sulfates at 291–293 K and observed similar dependencies as others at 298 K. Ramette and Palmer (16) measured the solubility of thallium iodate in 0.1 mol kg^{-1} solution of sodium perchlorate and in aqueous perchloric acid solutions over the temperature range 275.4–348.2 K. The presence of acid increased the $TlIO_3$ solubility. Failey has studied the solubility of $TlIO_3$ in solutions of the sodium salts of benzenehexacarboxylic acid (6) and benzenecarboxylic acids (9). An addition of the sodium salts resulted in an increase of the solubility $TlIO_3$. The solubility data were used to test the theory of Debye–Hückel.

The solubility of $TlIO_3$ in aqueous–organic solvent mixtures have been reported in 5 publications (5, 7, 8, 13, 17). La Mer and Goldman (5) and Pedersen (8) have studied the solubilities of $TlIO_3$ in ethanol–water mixtures and 1,4-dioxane–water, respectively. The solubility in the aqueous–organic solvent mixtures decreased with increase of the concentration of the organic solvent component. The solubilities of $TlIO_3$ in aqueous urea solutions were measured by Pedersen at 291.00 K (8), Gyunner and Fedorenko at 293.2 K (13), and Failey at 298.2 K (17). They stated equivocally that the addition of urea to water resulted in an increase in the solubility of $TlIO_3$. Keefer and Reiber (7) and Failey (17) measured the solubilities of $TlIO_3$ in 8 amino acid solutions; their results agreed better than 2 % for alanine and better than 0.5 % for glycine. The solubility increased with increasing the concentration of any amino acid. Keefer and Reiber stated that the solubility phenomena observed may be explained by assuming complex ion formation to occur between thallium ions and amino acid anions, the glycinate and alaninate.

One study for the aqueous ternary $TlIO_3$–$LiIO_3$–H_2O system was reported in (14). The system is of a simple eutonic type and no double salts were found. The reader will find the detailed compilation in (18).

The Multicomponent System

The solubility studies for aqueous quaternary are summarized in Table 110.

TABLE 110. Summary of the $TlIO_3$ solubility studies in aqueous quaternary systems

System	T / K	Reference
$TlIO_3$–sodium mellitate–NaCl–H_2O	298	(6)
$TlIO_3$–sodium mellitate–Na_2SO_4–H_2O	298	(6)
$TlIO_3$–sodium mellitate–$NaNO_3$–H_2O	298	(6)
$TlIO_3$–sodium mellitate–$MgSO_4$–H_2O	298	(6)
$TlIO_3$–sodium mellitate–$MgCl_2$–H_2O	298	(6)
$TlIO_3$–sodium benzoate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium benzoate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–sodium o-phthalate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium o-phthalate–$NaNO_3$–H_2O	298	(9)

$TlIO_3$–sodium m-phthalate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium m-phthalate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–sodium p-phthalate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium p-phthalate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–sodium trimasate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium trimasate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–sodium pyromasate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium pyromasate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–sodium benzen pentacarboxylate–NaCl–H_2O	298	(9)
$TlIO_3$–sodium benzen pentacarboxylate–$MgCl_2$–H_2O	298	(9)
$TlIO_3$–KNO_3–K_2SO_4–H_2O	291–293	(12)
$TlIO_3$–KNO_3–$Al_2(SO_4)_3$–H_2O	291–293	(12)
$TlIO_3$–$NaNO_3$–$NaClO_4$–H_2O	293	(19)
$TlIO_3$–$Na_2S_2O_3$–$NaNO_3$–H_2O	293	(20)
$TlIO_3$–$Na_2S_2O_3$–$NaClO_4$–H_2O	293	(20)

Failey (6, 9) studied the solubility of $TlIO_3$ in solutions of sodium carboxylates both with and without the addition of a third salt. He reported that in the solutions of sodium benzenehexacarboxylate an anomalous solvent effect was observed (6). The unexpectedly high solubility of $TlIO_3$ in solutions of sodium benzenehexacarboxylate suggested that addition of the third salts, by modifying the electrical field around the benzenehexacarboxylate hexavalent ions, might cause a diminution in solubility instead of its increase to be expected as a result of the increased ionic strength. Fedorenko and Gyunner (19) measured the solubility of $TlIO_3$ at ionic strength $I = 3.0$. In this study, sodium nitrate and perchlorate were change to adjust the constant ionic strength. The result of the solubility study was used to calculate the mean cumulative stability constants of thallium nitrate–complexes. Solubility studies reported by Gyunner and Fedorenko (20) have shown that the mixed complex ions $[\mathrm{Tl(S_2O_3)NO_3}]^{2-}$ and $[\mathrm{Tl(S_2O_3)_2}]^{3-}$ were formed in the solutions.

Chan and Liu (21) studied the solubilities of $TlIO_3$ in solutions over wide pH range of 7.50–12.70. The species of various complexes formed in aqueous solutions were established. The solubility increased slightly with the pH increase. Fedorenko (22) measured the solubilities for the $TlIO_3$–$Na_5P_3O_{10}$–Na_2SO_4–NaOH–H_2O system. Sodium sulfate and hydroxide were used to adjust a constant ionic strength at 2.4 and at pH of 13.0 ± 0.1. Gyunner and Fedorenko (20) studied the solubilities for the $TlIO_3$–$Na_2S_2O_3$–$NaNO_3$–$NaClO_4$–H_2O system at 293 K and ionic strength of 2.7. The formation of the mixed complex ions $[\mathrm{Tl(S_2O_3)(NO_3)}]^{2-}$ and $[\mathrm{Tl(S_2O_3)(NO_3)_2}]^{3-}$ was found.

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22. A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1543 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 841 (1974).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

W. Böttger, Z. Phys. Chem. **46**, 521–619 (1903).

Variables:

One temperature: 293.10 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 111. Solubility study of $TlIO_3$ in water at 293.10 K by conductivity method

t / °C	Equilibration time / h	$10^6\kappa_{\text{(satd. soln.)}}$ / S cm^{-1}	$10^6\kappa_{\text{(water)}}$ / S cm^{-1}	10^3c_1 / mol dm^{-3}
20.10	250	156.8[a]	0.93[a]	
	172	156.9[a]	0.93[a]	
	156	156.1[a]	0.93[a]	
20.10	339	156.1[a]	0.93[a]	
	268	156.3[a]	0.93[a]	
	82	156.1[a]	0.93[a]	
19.95	67	154.0	1.06	1.47[b]
	—	154.1	1.06	1.47[b]
	—	154.5	1.06	1.47[b]
		(av.) 154.2	1.06	1.47

[a] Both $\kappa_{\text{(satd. soln.)}}$ and $\kappa_{\text{(water)}}$ by the author were not included in the average values.

[b] Calculated by the compilers.

Using the average values of the conductivities of the saturated solutions and water, and molar conductances of 68.8 S cm^2 mol^{-1} for Tl^+ and 35.5 S cm^2 mol^{-1} for IO_3^- (these values are contemporary acceptable), the author computed a solubility from eqs. (54) and (55).

$$\kappa = \kappa_{\text{(satd. soln.)}} - \kappa_{\text{(water)}} \tag{54}$$

$$c_1 = \frac{1000\,\kappa}{\lambda^{\infty}_{\text{Tl+}} + \lambda^{\infty}_{\text{IO}_3^-}} \tag{55}$$

$K_{\text{s}} = c_1^2 = 2.16 \times 10^{-6}$ mol^2 dm^{-6} at 19.95 °C. Dissociation degree of the salt was determined at 97.1 %.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of $TlIO_3$ in water was determined from measurements of electrolytic conductivities of the saturated solutions and of pure water. The conductivity cells, which employed Pt electrodes, were constructed to prevent entrapment of air bubbles. After sealing, the entire cell was immersed in a water bath and rotated in the dark for 7–17 min. Equilibrium was reached when the conductivity remained constant within an experimental error (± 0.1 %). The conductance bridge and the thermometer were calibrated. The cell constant was determined with the use of 0.01 mol dm^{-3} KCl solution.

Source and Purity of Materials:

Salts (from Merck) were recrystallized several times. Water was twice distilled. Thallium iodate was prepared from potassium iodate and thallium nitrate.

Estimated Error:

Solubility: precision ± 0.2 %.
Temperature: stability ± 0.1 K; accuracy ± 0.02 K.

Components:
(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
J. F. Spencer, Z. Phys. Chem. **80**, 701–8 (1912).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 112. Solubility of $TlIO_3$ in water at 25 °C by emf method

E / V	$c(\mathrm{Tl}^+)$ / mol dm^{-3}	$10^3c(\mathrm{IO}_3^-)$ / mol dm^{-3}	10^6K_s / mol^2 dm^{-6}	10^3c_1 / mol dm^{-3}
0.5612	0.00208	2.16	4.49	2.11
0.6186	0.0193	2.36	4.55	2.13
0.6335	0.0343	0.132	4.52	2.12
0.6458	0.0553	0.0823	4.55	2.13
0.6639	0.1108	0.0409	4.53	2.13
			(av.) 4.53	(av.) 2.12

The solubility product was calculated from $K_{\mathrm{s}} = [\mathrm{Tl}^+][\mathrm{IO}_3^-]$ and the solubility of TlIO_3 was given by $c_1 = \sqrt{K_{\mathrm{s}}}$. Since the author measured activities of Tl^+ and IO_3^- in fact, the value K_{s} has a thermodynamic character. However, initial TlIO_3 concentrations were not specified and no activity coefficients could be calculated to obtain the solubility product expressed with concentrations.

Auxiliary Information

Method/Apparatus/Procedure:

An electrode of the third order was prepared in a closed bottle by shaking a mixture of Hg, $\mathrm{Hg_2(IO_3)_2}$, and TlIO_3 with solutions of TlNO_3 of various concentrations. After the saturation the single potential was measured against 0.1 mol dm^{-3} calomel electrode at the selected temperature. The thallium concentration was given by the expression:

$$E = 0.7207 + 0.0595 \log[\mathrm{Tl}^+] \text{ at 25 °C.}$$

Similar potentiometric measurements were performed with an electrode of the second order prepared by shaking Hg and $\mathrm{Hg_2(IO_3)_2}$ with solutions of KIO_3. The iodate concentration was calculated from the expression:

$$E = 0.4027 - 0.0595 \log[\mathrm{IO}_3^-].$$

Equilibrium concentrations of thallium and iodate ions in TlIO_3 saturated solution were used to calculate the solubility product and the solubility of TlIO_3.

Source and Purity of Materials:

Nothing specified.

Estimated Error:

Solubility: precision ± 0.5 % (compilers).

Temperature: nothing specified.

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium chloride; NaCl; [7647-14-5]
or (2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Sodium sulfate; Na_2SO_4; [7757-82-6]
or (2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Magnesium nitrate; $Mg(NO_3)_2$; [10377-60-3]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
or (2) Potassium chloride; KCl; [7447-40-7]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
or (2) Zinc sulfate; $ZnSO_4$; [7733-02-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632–45 (1929).

Variables:

Added salts and their concentrations
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 113. Solubility of $TlIO_3$ in aqueous solutions of various inorganic salts at 25 °C

Added salt	c_2 / mol dm^{-3}	Number of experiments	$10^3 c_1$ / mol dm^{-3}
None	—	15	1.844
NaCl	0.10	4	2.620
$NaNO_3$	0.1	6	2.486
Na_2SO_4	0.03333	4	2.543
	0.05	4	2.740
$MgCl_2$	0.03333	4	2.544
	0.05	4	2.744
$Mg(NO_3)_2$	0.03333	4	2.459
	0.05	4	2.621
$MgSO_4$	0.005	4	2.068
	0.01	5	2.172
	0.025	5	2.387
	0.05	3	2.640
KCl	0.01	4	2.005
	0.02	4	2.107
	0.05	4	2.335
	0.10	4	2.625
KNO_3	0.01	4	1.987
	0.02	3	2.079

	0.05	6	2.270
	0.1	4	2.492
	0.3	3	3.126
	0.5	2	3.673
	1.0	2	4.821
K_2SO_4	0.001	4	1.912
	0.002	4	1.958
	0.005	4	2.075
	0.01	4	2.193
	0.013	4	2.252
	0.03	4	2.513
	0.03333	4	2.544
	0.05	6	2.747
	0.064	4	2.877
	0.15	2	3.520
	0.25	4	4.028
	0.5	4	5.128
$ZnSO_4$	0.05	5	2.789

Auxiliary Information

Method/Apparatus/Procedure:

Details of the saturating column used were described in (1). The iodate content in the saturated solution was determined iodometrically. 1 cm^3 of 9 mol dm^{-3} sulfuric acid was added to 25 cm^3 of the sample solution. The solution was then placed in the dark for 5 min, 50 cm^3 of solution containing 3 g of sodium hydrogencarbonate was added and the content of the flask titrated with standard sodium arsenite using starch as an indicator. The end-point was obtained by back titrating with standard 0.0075 mol dm^{-3} iodine solution.

Source and Purity of Materials:

Eighty g of potassium iodate in solution and 100 g of thallium nitrate in solution were added dropwise and simultaneously to 6 dm^3 of water at 60 °C with constant stirring. The precipitate was allowed to digest and the stirring continued. The salt was washed by decantation 6 times with water, placed in a beaker containing 500 cm^3 of water, allowed to digest on a hot-plate at 80 °C, and then sedimented to obtain crystals of uniform size.

The best chemically pure grade sodium chloride, nitrate and sulfate and potassium chloride, nitrate and sulfate were twice recrystallized from water, dried in an oven at 120 °C, ground in an agate mortar and dried again at 120 °C for 24 h. Kahlbaum's "for analysis" zinc sulfate was dried to constant weight in an air-bath and kept in a desiccator over sulfuric acid. Kahlbaum's "for analysis" grade magnesium chloride was found to have an small amount of ammonia. The chloride content was determined by titrating against silver nitrate solution before use. No information on magnesium nitrate was reported. Chemically pure grade magnesium sulfate was twice recrystallized, dried in an oven at 50 °C for 3 d, and then kept in a desiccator over phosphorous pentaoxide for a week.

Estimated Error:

Solubility: precision ± 0.4 %.

Temperature: stability ± 0.005 K (authors (1)).

References:

[1] V. K. La Mer, C. V. King, and C. F. Mason, J. Am. Chem. Soc. **49**, 363 (1927); J. N. Brönsted, and V. K. La Mer, J. Am. Chem. Soc. **46**, 555 (1924).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
or (2) Cadmium chloride; $CdCl_2$; [10108-64-2]
or (2) Cadmium sulfate; $CdSO_4$; [10124-36-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. B. Friedman and V. K. La Mer, J. Am. Chem. Soc. **53**, 103–5 (1931).

Variables:

Concentration of added salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 114. Solubility of $TlIO_3$ in aqueous solutions of magnesium and cadmium salts at 25 °C

Added salt	c_2 / mol dm^{-3}	Number of detns.	10^3c_1 / mol dm^{-3}
None	—	5	1.819 ± 0.006
$MgSO_4$	0.05	8	2.625 ± 0.007
$MgCl_2$	0.05	5	2.755 ± 0.009
$CdSO_4$	0.05	5	2.798 ± 0.003
$CdCl_2$	0.05	4	2.708 ± 0.002

Auxiliary Information

Method/Apparatus/Procedure:

The saturating column method was used as described in (1). The iodate content in the saturated solution was determined by iodometric titration using sodium arsenite (2).

Source and Purity of Materials:

$TlIO_3$ was prepared by adding the solutions of thallium nitrate and potassium iodate to a large amount of water at 60 °C with constant stirring. The precipitate was allowed to digest and the stirring continued. The salt was washed by decantation 6 times with water, after which it was placed in a beaker containing 500 cm^3 of water, allowed to digest on a hot-plate at 80 °C, and then sedimented to obtain crystals of uniform size (2). Magnesium sulfate was recrystallized twice and dried to the anhydrous state. Chemically pure grade cadmium sulfate (from J. T. Baker) was recrystallized and monohydrate was obtained. The source of magnesium and cadmium chlorides was not reported.

Estimated Error:

Solubility: deviations from average between ± 0.1 % and ± 0.3 %.
Temperature: stability ± 0.005 K (authors (1)).

References:

[1] J. N. Brönsted and V. K. La Mer, J Am. Chem. Soc. **46**, 555 (1924).
[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]

(2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **53**, 473–6 (1931).

Variables:

Concentration of ethanol: 0–94.7 %

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 115. Solubility of $TlIO_3$ in ethanol–water mixtures at 25 °C

$100w_2$	$100x_2$[a]	10^3c_1 / mol dm^{-3}	ρ / g cm^{-3}
0	0	1.841	0.99708
4.25	1.71	1.416	0.9894
11.56	4.86	0.9147	0.9780
21.37	9.61	0.5038	0.9644
31.61	15.31	0.2971	0.9478
40.51	21.03	0.1345	0.9304
46.91	25.68	0.0832	0.9167
94.71	87.50	—[b]	

[a] Calculated by the compiler using the 1999 IUPAC recommended atomic weights.

[b] Solubility too small to be detected.

Auxiliary Information

Method/Apparatus/Procedure:

The saturating column method was used as described in (1). The iodate content in the saturated solution was determined iodometrically. Sulfuric acid was added to the sample solution. After the flask containing the solution was placed in the dark for 5 min, sodium hydrogencarbonate solution was added and the content of the flask was titrated with standard sodium arsenite solution using starch as an indicator. The end-point was obtained by back titrating with standard iodine solution.

Source and Purity of Materials:

Eighty g of potassium iodate in solution and 100 g of thallium(I) nitrate in solution were added dropwise and simultaneously to 6 dm^3 of water at 60 °C with constant stirring. The precipitate was allowed to digest and the stirring continued. The salt was washed by decantation 6 times with water, after which it was placed in a beaker containing 500 cm^3 of water, allowed to digest on a hot-plate at 80 °C, and then sedimented to obtain crystals of uniform size. Ethanol was distilled from potassium hydroxide and silver nitrate.

Estimated Error:

Solubility: nothing specified.

Temperature: stability ± 0.005 K (authors (1)).

References:

[1] J. N. Brönsted, and V. K. La Mer, J. Am. Chem. Soc. **46**, 555 (1924); V. K. La Mer, C. V. King, and C. F. Mason, J. Am. Chem. Soc. **49**, 363 (1927).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Benzenehexacarboxylic acid, hexasodium salt (sodium mellitate); $C_{12}O_{12}Na_6$; [145-44-8]
(3) Sodium hydroxide; NaOH; [1310-73-2]
or (3) Sodium chloride; NaCl; [7647-14-5]
or (3) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (3) Sodium sulfate; Na_2SO_4; [7757-82-6]
or (3) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (3) Magnesium sulfate; $MgSO_4$; [7487-88-9]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

C. F. Failey, J. Am. Chem. Soc. **55**, 3112–6 (1933).

Variables:

Concentration of inorganic compounds and sodium mellitate
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 116. Solubility of $TlIO_3$ in aqueous solutions of sodium mellitate containing various inorganic compounds at 25.00 °C

Added inorganic compound	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}
None	—	0	1.843
	—	0.0001	1.967
	—	0.0002	2.092
	—	0.0005	2.426
	—	0.001	2.901
	—	0.002	3.636
	—	0.005	4.980
	—	0.1	6.184
NaOH	0.1	0	2.003
NaCl	0.02	0	2.118
	0.04	0	2.277
	0.1	0	2.629
	0.004	0.001	2.850
	0.020	0.001	2.781
	0.028	0.001	2.781
	0.040	0.001	2.797
	0.060	0.001	2.843
	0.100	0.001	2.967
$NaNO_3$	0.05	0.005	4.210
	0.10	0.005	4.004
	0.20	0.005	3.936

	0.50	0.005	4.352
	0.001	0.001	2.901
	0.004	0.001	2.844
	0.012	0.001	2.786
	0.020	0.001	2.762
	0.028	0.001	2.750
	0.040	0.001	2.746
	0.060	0.001	2.768
	0.080	0.001	2.802
$\mathrm{Na_2SO_4}$	0.004248	0.001	2.858
	0.01062	0.001	2.872
	0.02124	0.001	2.894
$\mathrm{MgCl_2}$	0.000408	0.001	2.732
	0.00102	0.001	2.520
	0.00204	0.001	2.319
	0.00612	0.001	2.243
	0.01020	0.001	2.295
	0.02040	0.001	2.460
	0.04080	0.001	2.731
$\mathrm{MgSO_4}$	0.000420	0.001	2.738
	0.001050	0.001	2.538
	0.00210	0.001	2.373
	0.00420	0.001	2.306
	0.0105	0.001	2.360
	0.0210	0.001	2.505
	0.0420	0.001	2.696

Auxiliary Information

Method/Apparatus/Procedure:

A stock solution of sodium mellitate was prepared by adding an equivalent amount of sodium hydroxide to a solution of mellitic acid. Solvents were rotated with the solid phases of the salts added in a water-bath at constant temperature. The analytical method, described in (1), was used to determine the solubilities of the iodate in these various solutions.

Source and Purity of Materials:

The preparation method of thallium iodate was similar to that of La Mer and Goldman (1). Mellitic acid was prepared by oxidizing hexamethylbenzene with potassium permanganate in cold. The lead salt was precipitated, decomposed with hydrogen sulfide and crude acid was twice recrystallized from diluted nitric acid. Calculated for $C_{12}H_6O_{12}$: C–42.10; H–1.77 mass %. Found in analysis: C–42.28; H–2.02 mass %. The neutralization of 0.1056 g of acid required 18.42 cm^3 of 0.0999 mol dm^{-3} sodium hydroxide; thus the acid purity was 99.4 %. Source of inorganic salts was not reported.

Estimated Error:

Solubility: nothing specified; precision probably no worser than in (1), it is ± 0.5 %.
Temperature: precision ± 0.01 K.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Glycine (aminoacetic acid); $C_2H_5NO_2$; [56-40-6]
or (2) Glycine, *N*-methyl- (sarcosine); $C_3H_7NO_2$; [107-97-1]
or (2) Alanine (α-alanine, 2-aminopropionic acid); $C_3H_7NO_2$; [6898-94-8]
or (2) β-Alanine (3-aminopropanoic acid); $C_3H_7NO_2$; [107-95-9]
or (2) Butanoic acid, 2-amino- (α-amino-n-butyric acid); $C_4H_9NO_2$; [80-60-4]
or (2) Alanine, 2-methyl- (α-aminoisobutyric acid); $C_4H_9NO_2$; [62-57-7]
or (2) Pentanoic acid, 2-amino- (α-amino-n-valeric acid, L-norvaline); $C_5H_{11}NO_2$; [6600-40-4]

or (2) Glycine, *N*-glycyl- (*N*-glycylglycine); $C_4H_8N_2O_3$; [556-50-3]
or (2) Glycine, *N*,*N*-dimethyl- (*N*,*N*-dimethylglycine); $C_4H_9NO_2$; [1118-68-9]
or (2) Urea; CH_4N_2O; [57-13-6]
or (2) 2,5-Piperazinedione (diketopiperazine); $C_4H_6N_2O_2$; [106-57-0]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

C. F. Failey, J. Am. Chem. Soc. **55**, 4374–8 (1933).

Variables:

Concentration of amino compounds
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 117. Solubility of $TlIO_3$ in aqueous solutions of amino compounds at 25.00 °C

Added amino compound	c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
None	—	1.843
Glycine	0.025	1.875
	0.05	1.907
	0.10	1.971
	0.15	2.033
	0.20	2.098
N-Methylglycine	0.05	1.880
	0.10	1.917
	0.15	1.954
	0.20	1.993
α-Alanine[a]	0.025	1.872
	0.05	1.907
	0.10	1.963
	0.20	2.084
β-Alanine	0.025	1.863
	0.05	1.884
	0.10	1.919

	0.15	1.964
	0.20	2.001
2-Aminobutanoic acid	0.10	1.906
	0.20	1.971
2-Methylalanine	0.05	1.874
	0.10	1.901
	0.15	1.934
	0.20	1.956
2-Aminopentanoic acid	0.10	1.895
	0.20	1.939
N-Glycylglycine	0.20	2.282
N,*N*-Dimethylglycine	0.025	1.859
	0.05	1.873
	0.10	1.901
	0.20	1.957
Urea	0.025	1.845
	0.05	1.851
	0.10	1.861
	0.15	1.876
	0.20	1.891
2,5-Piperazinedione	0.10	1.844

[a] No information about optical activity was described by the author.

Auxiliary Information

Method/Apparatus/Procedure:

Samples were rotated overnight in a water thermostat at the selected temperature. The method of analysis of thallium iodate was used as described in (1). The iodate content in the saturated solution was determined iodometrically. Dilute sulfuric acid was added to the sample solution. The solution was then placed in the dark for 5 min, sodium hydrogencarbonate was added and the solution was titrated with standard sodium arsenite using starch as an indicator. The end-point was obtained by back-titrating with standard iodine solution. Thallium iodate solubilities are the mean of duplicate analyses.

Source and Purity of Materials:

Thallium iodate was prepared by dropping solutions of analytical reagent potassium iodate and twice recrystallized thallium nitrate into a large volume of water at 60 °C. The precipitate was allowed to digest and the stirring continued. The salt was washed by decantation 6 times with water after which it was placed in a beaker containing 500 cm^3 of water, allowed to digest on a hot plate at 80 °C, and then smaller particles removed by sedimentation (1).

N,*N*-Dimethylglycine was prepared by hydrolyzing its ethyl ester with sulfuric acid and removing the acid with barium hydroxide. β-Alanine was prepared from succinimide (2). The remaining amino acids recrystallized from commercial materials were all salts free and the nitrogen contents of the amino acids are given as follows:

Name	Formula	Nitrogen (mass %) found by analysis	Calcd. by the compilers (mass %)
Glycine	$C_2H_5NO_2$	18.70	18.66
N-Methylglycine	$C_3H_7NO_2$	15.57	15.72
α-Alanine	$C_3H_7NO_2$	15.59	15.72
2-Aminobutanoic acid	$C_4H_9NO_2$	13.72	13.58
2-Methylalanine	$C_4H_9NO_2$	13.56	13.58
2-Aminopentanoic acid	$C_5H_{11}NO_2$	11.84	11.96
N-Glycylglycine	$C_4H_8N_2O_3$	20.90	21.20
Urea	CH_4N_2O	46.64	46.65
2,5-Piperazinedione	$C_4H_6N_2O_2$	24.57	24.55

Estimated Error:

Solubility: the average difference between duplicate analyses was 0.16 %.

Temperature: precision ± 0.01 K.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

[2] F. H. Holm, Arch. Pharm. **242**, 590 (1904).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Glycine (aminoacetic acid); $C_2H_5NO_2$; [56-40-6]
or (2) Alanine; $C_3H_7NO_2$; [6898-94-8]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Keefer and H. G. Reiber, J. Am. Chem. Soc. **63**, 3504–7 (1941).

Variables:

Amino acids and their concentration
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto and Ryo Miyamoto

Experimental Data

TABLE 118. Solubility of $TlIO_3$ in aqueous solutions of amino acids at 25.00 °C

Amino acid	c_2 / mol kg^{-1}	$10^3 c_1$ / mol kg^{-1}
None	—	1.850
Glycine	0.0498	1.909
	0.1000	1.976
	0.1503	2.037
	0.2010	2.103
Alanine[a]	0.0498	1.896
	0.1000	1.947
	0.1503	1.999
	0.2010	2.043

[a] Information about both optical activity and structural isomer was not described by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility determinations were performed by the method of Keefer et al. (1). Amino acid solutions were prepared from boiled water using a calibrated volumetric equipment. An excess of thallium iodate was placed in a glass-stoppered Pyrex flask and amino acid solution was added. The flasks were rotated in a thermostat at the selected temperature for at least 12 h. It was found that the equilibrium was obtained in 4–5 h. The solubilities were approached from both sides of equilibrium. The determination of iodate ion, in the presence of thallium ion, using sodium thiosulfate was carried out. The iodometric titrations performed on aliquots of a potassium iodate solution in the presence and absence of thallium ion were the same to 0.1 % in a preliminary experiments.

Source and Purity of Materials:

Thallium iodate was prepared by the method of La Mer and Goldman (2). Eighty g of potassium iodate in solution and 100 g of thallium nitrate were added dropwise and simultaneously to 6 dm^3 of water at 60 °C with constant stirring. The precipitate was allowed to digest and the stirring continued. The salt was washed by decantation 6 times with water, after which it was placed in a beaker containing 500 cm^3 of water, allowed to digest on a hot-plate at 80 °C, and then sedimented to obtain crystals of uniform size. Chemically pure glycine and alanine were recrystallized twice from water by the addition of ethanol. The product was dried in a vacuum oven at about 35 °C. Distilled water was used.

Estimated Error:

Solubility: accuracy ± 0.1 %.

Temperature: precision ± 0.02 K (authors (1)).

References:

[1] R. M. Keefer and H. G. Reiber, and C. S. Bisson, J. Am. Chem. Soc. **62**, 2951 (1940).

[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]
or (2) Urea; CH_4N_2O; [57-13-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

K. J. Pedersen, K. Dan. Vidensk. Selsk. Mat.-Fys. Medd. **18** (12), 1–26 (1941).

Variables:

Concentration of dioxane (0–0.75 mol dm^{-3}) or of urea (0–1.00 mol dm^{-3})
Temperature: 291.00 K (1,4-dioxane) and 291.15 K (urea)

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 119. Solubility of $TlIO_3$ in aqueous solutions of dioxane and urea at 17.85 and 18.00 °C

t / °C	Organic solvent	c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
18.00	1,4-Dioxane	0	1.417
		0.250	1.315
		0.500	1.213
		0.750	1.117
17.85	Urea	0	1.410
		0.250	1.467
		0.500	1.526
		0.750	1.586
		1.000	1.644

Auxiliary Information

Method/Apparatus/Procedure:

Excess of thallium iodate and dioxane–water or urea–water mixtures were placed into glass-stoppered bottles. The bottles were rotated in an electrically regulated water thermostat. Samples of the solution were analyzed after different times of rotation in order to make sure that saturation was attained. Aliquots of the saturated solutions were sucked from the bottles through a porous glass filter into a pipet. The iodate content was determined iodometrically at least 2 times.

Source and Purity of Materials:

Thallium iodate was prepared from thallium sulfate and potassium iodate. 1,4-Dioxane (Haardt & Co.) was left for 2 d over solid sodium hydroxide, refluxed with sodium for several hours, and then distilled in an all-glass apparatus. The main fraction had a freezing point of 11.65 °C, compared with 11.80 °C for pure dioxane (1). Urea (Kahlbaum, "for scientific purpose") was used without further purification. It contained traces of calcium which were not removed by recrystallization from alcohol. 1–3 mg of ash and $(1\text{–}2) \times 10^{-5}$ moles of calcium were found per mole of urea. The impurity is without any importance for the measurements.

Estimated Error:

Solubility: within the limit of accuracy of the analytical method (author).

Temperature: nothing specified.

References:

[1] K. Hess and H. Frahm, Ber. Dtsch. Chem. Ges. **71B**, 2627 (1938).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-9]
(2) Sodium chloride; NaCl; [7647-14-5]
or (2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Sodium nitrate; $NaNO_3$; [7631-99-4]
(3) Benzoic acid, sodium salt (sodium benzoate); $C_7H_5O_2Na$; [532-32-1]
or (3) 1,2-Benzenedicarboxylic acid, disodium salt (sodium *o*-phthalate); $C_8H_4O_4Na_2$; [15968-01-1]
or (3) 1,3-Benzenedicarboxylic acid, disodium salt (sodium *m*-phthalate); $C_8H_4O_4Na_2$; [10027-33-5]
or (3) 1,4-Benzenedicarboxylic acid, disodium salt (sodium *p*-phthalate); $C_8H_4O_4Na_2$; [10028-70-3]
or (3) 1,3,5-Benzenetricarboxylic acid, trisodium salt (sodium trimesate); $C_9H_3O_6Na_3$; [17274-08-7]
or (3) 1,2,4,5-Benzenetetracarboxylic acid, tetrasodium salt (sodium pyromellitate); $C_{10}H_2O_8Na_4$; [148-04-9]
or (3) Benzenepentacarboxylic acid, pentasodium salt (sodium benzenepentacarboxylate); $C_{11}HO_{10}Na_5$; [145-45-9]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

C. F. Failey, J. Am. Chem. Soc. **68**, 2619–24 (1946).

Variables:

Concentrations of sodium benzenecarboxylates and inorganic salts
Temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 120. Solubility of $TlIO_3$ in aqueous solutions of sodium benzenecarboxylates containing NaCl, $NaNO_3$ or $MgCl_2$ at 25.00 °C

Added sodium carboxylate	c_3 / mol dm^{-3}	Added inorganic salt	c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
Sodium benzoate	0	None	—	1.834
	0.02		—	2.057
	0.05		—	2.228
	0.10		—	2.429
	0.20		—	2.723
	0.02	NaCl	0.01	2.141
	0.02		0.03	2.285
	0.02		0.10	2.667
	0.02	$MgCl_2$	0.00398	2.145
	0.02		0.00994	2.261
	0.02		0.01988	2.422
	0.02		0.0398	2.681

Sodium *o*-phthalate	0.001333	None	—	1.930
	0.002667		—	2.005
	0.00667		—	2.172
	0.01333		—	2.377
	0.03333		—	2.798
	0.0667		—	3.287
	0.00667	NaCl	0.002	2.182
	0.00667		0.004	2.196
	0.00667		0.008	2.224
	0.00667		0.01	2.251
	0.00667		0.03	2.373
	0.00667		0.06	2.542
	0.00667		0.10	2.735
	0.00667	$NaNO_3$	0.002	2.179
	0.00667		0.00398	2.218
	0.00667		0.004	2.193
	0.00667		0.008	2.217
	0.00667		0.00994	2.306
	0.00667		0.01	2.229
	0.00667		0.01988	2.446
	0.00667		0.020	2.280
	0.00667		0.0398	2.697
	0.00667		0.050	2.416
	0.00667		0.10	2.607
Sodium *m*-phthalate	0.00667	None	—	2.053
	0.01333		—	2.171
	0.03333		—	2.401
	0.0667		—	2.655
	0.00667	NaCl	0.01	2.136
	0.00667		0.03	2.276
	0.00667		0.06	2.458
	0.00667		0.10	2.664
	0.00667	$MgCl_2$	0.00398	2.134
	0.00667		0.00994	2.242
	0.00667		0.01988	2.398
	0.00667		0.0398	2.660
Sodium *p*-phthalate	0.00667	None	—	2.046
	0.01333		—	2.156
	0.03333		—	2.373
	0.0667		—	2.613
	0.00667	NaCl	0.01	2.129
	0.00667		0.03	2.270
	0.00667		0.06	2.452
	0.00667		0.10	2.658
	0.00667	$MgCl_2$	0.00398	2.131

	0.00667		0.00994	2.241
	0.00667		0.01988	2.402
	0.00667		0.0398	2.663
Sodium trimesate	0.003333	None	—	2.069
	0.00667		—	2.189
	0.01667		—	2.416
	0.03333		—	2.661
	0.003333	NaCl	0.01	2.142
	0.003333		0.03	2.279
	0.003333		0.06	2.455
	0.003333		0.10	2.661
	0.003333	$MgCl_2$	0.00398	2.128
	0.003333		0.00994	2.233
	0.003333		0.01988	2.393
	0.003333		0.0398	2.653
Sodium pyromellitate	0.0004	None	—	1.944
	0.0008		—	2.029
	0.002		—	2.214
	0.004		—	2.458
	0.010		—	2.818
	0.0152		—	3.053
	0.02		—	3.224
	0.002	NaCl	0.01	2.252
	0.002		0.03	2.359
	0.002		0.06	2.517
	0.002		0.10	2.709
	0.002	$MgCl_2$	0.000796	2.183
	0.002		0.001988	2.160
	0.002		0.00398	2.162
	0.002		0.00994	2.245
	0.002		0.01988	2.401
	0.002		0.0398	2.665
Sodium benzenepentacarboxylate	0.0001067	None	—	1.908
	0.0002666		—	2.029
	0.000533		—	2.212
	0.001334		—	2.636
	0.002666		—	3.123
	0.001334	NaCl	0.01	2.587
	0.001334		0.03	2.607
	0.001334		0.06	2.701
	0.001334		0.10	2.842
	0.001334	$MgCl_2$	0.000796	2.424
	0.001334		0.001988	2.253
	0.001334		0.00398	2.195
	0.001334		0.00994	2.258

0.001334	0.01998	2.408
0.001334	0.0398	2.671

Auxiliary Information

Method/Apparatus/Procedure:

Solvents were rotated with the solid phase for 24 h at the selected temperature. Analytical methods used were those of La Mer and Goldman (1). The iodate content in the saturated solution was determined iodometrically. 1 cm^3 of 9 mol dm^{-3} sulfuric acid was added to 25 cm^3 of the sample solution. The solution was then placed in dark for 5 min, 50 cm^3 of solution containing 3 g of $NaHCO_3$ was added and the content of the flask titrated with standard sodium arsenite using starch as an indicator. The end-point was obtained by back-titrating with standard iodine solution.

Source and Purity of Materials:

Benzoic acid, *o*-, *m*-, and *p*-phthalic acids, and trimesic acid were obtained from the Eastman Kodak Co. The other acids except *p*-phthalic acid were repeatedly recrystallized from water. After *p*-phthalic acid was freed from *p*-toluene, the acid was repeatedly recrystallized from water. Pyromellic and benzenpentacarboxylic acids were prepared by oxidation of the corresponding tetra- or pentamethyl substituted benzene with potassium permanganate in the cold. The two compounds were prepared and purified in a laboratory of the Department of Chemistry, College of Physicians and Surgeons, Columbia University. The sodium salts were prepared in solutions by adding the equivalent amounts of sodium hydroxide and the corresponding acids.

The analyses of the acids prepared gave the following results.

Acid		Cald. (mass %)			Found (mass %)			m.p.
Name	Formula	C	H	Mol. wt.[a]	C	H	Mol. wt.[a]	t / °C
Benzoic acid	$C_7H_6O_2$	68.84	4.95	122.1	68.83	4.96	122.0	122
o-Phtalic acid	$C_8H_6O_4$	57.83	3.64	166.1	57.83	3.64	166.2	196
m-Phthalic acid	$C_8H_6O_4$	57.83	3.64	166.1	57.88	3.53	166.3	343
p-Phthalic acid	$C_8H_6O_4$	57.83	3.64	166.1	57.78	3.65	166.8	> 300 sublime
Trimesic acid	$C_9H_6O_6$	51.44	2.88	210.1	51.49	2.80	211.3	> 362
Pyromellitic acid	$C_{10}H_6O_8$	47.25	2.38	254.2	47.18	2.32	254.0	271
Benzenepenta-carboxylic acid	$C_{11}H_6O_{10}$	44.31	2.03	298.2	44.50	1.91	not given	239

[a] Calculated by the compilers from the equivalent weight reported by the author.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.02 K.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium fluoride; NaF; [7681-49-4]
or (2) Potassium chloride; KCl; [7447-40-7]
or (2) Potassium hydroxide; KOH; [1310-58-3]
or (2) Potassium thiocyanate; KSCN; [333-20-0]
or (2) Ferrate($4-$), hexakis(cyano-*C*)-, tetrapotassium (potassium hexacyanoferrate(II)); $K_4[Fe(CN)_6]$; [13943-58-3]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. P. Bell and J. H. B. George, Trans. Faraday Soc. **49**, 619–27 (1953).

Variables:

Concentration of inorganic salts
Temperature: 273, 298 and 313 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 121. Solubility of $TlIO_3$ in aqueous solutions of inorganic salts at various temperatures

Added inorganic salt	t / °C	$10^3 c_2$ / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}
None	0	—	0.650
	25	—	1.838
	40	—	3.043
NaF	25	4.83	1.919
		10.34	1.972
		22.57	2.070
		33.83	2.125
		46.46	2.185
KCl	0	5.71	0.697
		14.12	0.738
		24.82	0.778
		40.59	0.831
		51.74	0.853
	25	4.90	1.930
		12.57	20.25
		25.65	2.158
		40.81	2.266
		54.22	2.359
	40	5.81	3.196
		15.39	3.380
		30.44	3.595
		48.92	3.805

		57.38	3.881
KOH	0	4.91	0.690
		10.38	0.721
		20.38	0.761
		35.70	0.809
		49.83	0.844
		71.16	0.896
		93.53	0.943
	25	3.00	1.915
		5.36	1.951
		8.10	1.989
		15.40	2.070
		19.76	2.121
		33.04	2.233
		47.79	2.353
		65.93	2.455
		89.10	2.608
	40	5.60	3.212
		10.66	3.333
		21.43	3.514
		36.18	3.745
		50.07	3.905
		69.72	4.123
		88.46	4.313
KSCN	0	4.63	0.693
		5.04	0.699
		10.03	0.730
		16.95	0.758
		23.64	0.787
		24.80	0.794
	25	4.09	1.927
		18.31	2.100
		31.62	2.232
		45.00	2.346
		56.95	2.420
	40	5.54	3.188
		15.63	3.383
		32.29	3.620
		45.05	3.772
		55.90	3.885
$K_4[Fe(CN)_6]$	0	0.519	0.763
		1.094	0.840
		1.721	0.901
		2.291	0.955
		4.011	1.051

	25	0.504	2.053
		1.386	2.307
		2.343	2.502
		3.767	2.740
		6.132	3.036
	40	0.486	3.304
		0.942	3.505
		1.488	3.694
		2.328	3.970
		3.859	4.326
K_2SO_4	0	4.48	0.732
		9.87	0.782
		15.84	0.827
		21.52	0.861
		32.48	0.921
	25	1.98	1.948
		4.53	2.044
		9.52	2.177
		18.44	2.349
		28.61	2.486
	40	2.46	3.244
		6.75	3.465
		13.76	3.709
		20.70	3.895
		30.59	4.108

Method/Apparatus/Procedure:

The solubility measurements were made by passing about 50 cm^3 of salt solutions slowly through a column of solid thallium iodate (8 cm deep and 0.5 cm^2 cross-section) in a saturator resembling that described in (1). Solutions were brought to thermostat temperature before passing through the saturator, and repeated tests showed that complete saturation was reached after one passage. KI (0.2 g) was added to a weighed 10 cm^3 sample of solution and after allowing the precipitate of thallium iodide to settle the bulk of solution was filtered. 1 cm^3 1 mol dm^{-3} sulfuric acid was added to a weighed sample of this solution and the iodine liberated titrated with 0.005 mol dm^{-3} thiosulfate solution almost to the end-point. The titration was carried out by weight except that the final addition of thiosulfate (about 0.3 cm^3) was made by volume from a microburet after the addition of starch as an indicator. The solubilities were converted to molarity by using the densities of the salt solutions.

Source and Purity of Materials:

Thallium iodate was prepared by dropping solutions of analytical reagent potassium iodate and twice recrystallized thallium nitrate into a large volume of water at 65 °C. After stirring for 30 min the precipitate was allowed to settle and was washed 6 times with lukewarm water by decantation. It was digested with 3 times its weight of water for 4 h at 80 °C and most of the smaller particles removed by sedimentation. Sodium fluoride and potassium chloride, hydroxide, thiocyanate, sulfate and hexacyanoferrate(II) were of analytical reagent quality.

Estimated Error:

Solubility: standard deviation 0.3 % at 0 °C and 0.1 % at 25 and 40 °C.
Temperature: nothing specified.

References:

[1] C. W. Davies, J. Chem. Soc. **1938**, 277.

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Sodium azide; NaN_3; [26628-22-8]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. S. K. Nair and G. H. Nancollas, J. Chem. Soc. **1957**, 318–23.

Variables:

Concentration of potassium nitrate or sodium azide
Temperature: 273–313 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 122. Solubility of $TlIO_3$ in aqueous solutions of potassium nitrate or sodium azide at various temperatures

Added salt	t / °C	10^3c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
None	0	—	0.656
	10	—	1.016
	25	—	1.842
	40	—	3.048
KNO_3	0	32.76	0.778
		47.84	0.808
		50.92	0.813
		57.24	0.826
		59.80	0.830
	25	20.68	2.073
		28.64	2.127
		38.46	2.178
		39.58	2.195
		42.79	2.207
	40	20.56	3.408
		24.46	3.454
		30.44	3.517
		38.24	3.590
		39.39	3.600
		42.54	3.631
NaN_3	10	17.93	1.144
		20.24	1.155
		27.24	1.182
		30.35	1.193
		36.40	1.214
	25	6.49	1.935

9.12	1.973
12.11	2.004
14.81	2.029
20.23	2.072
23.71	2.097
27.05	2.119

Auxiliary Information

Method/Apparatus/Procedure:

Saturators of the type described by Davies (1) were used for solubility measurements. The column of the crystalline thallium iodate was washed 4 times before each determination with the solution being used. Solutions were brought to the thermostat temperature. The repeated tests showed complete saturation after passing twice through the column. Solutions were analyzed volumetrically by treatment with excess of potassium iodide and removal of the precipitated thallium iodide by centrifugation, the precipitate being washed thoroughly. The iodine liberated, after addition of 1 mol dm^{-3} sulfuric acid, was titrated against 0.005 mol dm^{-3} thiosulfate, the final addition being made with a microburette.

Source and Purity of Materials:

Thallium iodate was prepared by Bell and George's method (2). The iodate was prepared by dropping solutions of potassium iodate and (twice recrystallized) thallium nitrate into a large volume of water at 65 °C. After stirring for 0.5 h, the precipitate was allowed to settle and was washed 6 times with water by decantation. It was digested 3 times from with water for 4 h at 80 °C and most of the smaller particles were removed by sedimentation. Sodium azide was recrystallized 3 times from water and dried at 110 °C. "Analytical reagent" materials were employed. Conductivity water, prepared by mixed-bed deionization, was used throughout.

Estimated Error:

Solubility: precision ± 0.1 %.
Temperature: nothing specified.

References:

[1] C. W. Davies, J. Chem. Soc. **1938**, 277.
[2] R. P. Bell and J. H. B. George, Trans. Faraday Soc. **49**, 619 (1953).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(3) Sodium hydroxide; NaOH; [1310-73-2]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

C.-P. Chan and L.-S. Liu, K'o Hsueh T'ung Pao **1964**, No 5, 435–8.

Variables:

pH: 7.50–12.70
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 123. Solubility of $TlIO_3$ at various pH at ionic strength of 2.5 obtained by addition of $NaClO_4$ and NaOH at 25 °C

pH	10^3c_1 / mol dm^{-3}
12.70	1.964
12.60	1.950
12.48	1.935
12.30	1.920
12.00	1.905
7.50–10.50	1.890

The authors reported solubilities of $TlIO_3$ upon an addition of KSCN. However, the results are presented in a very complex form which was not clear to the compilers.

Auxiliary Information

Method/Apparatus/Procedure:

A given volume of sodium perchlorate solutions were placed into brown conical flasks. Sodium hydroxide was used to adjust proper pH. The suitable amounts of $TlIO_3$ were added to the solutions. The flasks were sealed and rotated in a big temperature-controlled-bath at the selected temperature for 10 h. After equilibration, aliquots of the sample solutions were withdrawn with a pipet equipped with cotton wool. The iodate concentration was determined iodometrically. KI and sulfuric acid solutions were added to the sample solutions. The liberated iodine ion concentration was determined with the thiosulfate solution standardized by using potassium chromate. A pH meter was made in China.

Source and Purity of Materials:

All reagents were of “analytically pure”. Thallium iodate was prepared from thallium nitrate and potassium iodate. Redistilled water was used.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.05 K.

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium triphosphate; $Na_5P_3O_{10}$; [7758-29-4]
(3) Sodium sulfate; Na_2SO_4; [7757-82-6]
(4) Sodium hydroxide; NaOH; [1310-73-2]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1543–6 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 841–3 (1974).

Variables:

Composition of the solution: $Na_5P_3O_{10}$ (0–0.14 mol dm^{-3}) and Na_2SO_4 (0–0.70 mol dm^{-3})
One temperature: 293 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 124. Solubility of $TlIO_3$ in aqueous solutions containing $Na_5P_3O_{10}$, Na_2SO_4 and NaOH at ionic strength of 2.4 at 20 °C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	c_4 / mol dm^{-3}	pH (initial solution)	10^3c_1 /mol dm^{-3}
0	0.70	0.3	12.90	5.80
0.02	0.60	0.3	12.96	6.60
0.04	0.50	0.3	12.95	7.50
0.06	0.40	0.3	13.00	8.33
0.07	0.35	0.3	12.98	8.80
0.08	0.30	0.3	12.98	9.26
0.09	0.25	0.3	12.95	9.82
0.10	0.20	0.3	13.02	10.38
0.11	0.15	0.3	12.94	10.76
0.12	0.10	0.3	12.98	11.38
0.13	0.05	0.3	12.97	12.30
0.14	0	0.3	13.05	12.90

The equilibrium solid phase was found to be $TlIO_3$. K_s(concentration solubility product) was calculated at 2.21×10^{-5} mol^2 dm^{-6} but conditions were not specified.

Auxiliary Information

Method/Apparatus/Procedure:

The solutions were saturated with thallium iodate at constant temperature. The Na_2SO_4 and NaOH were used to adjust a constant ionic strength and pH. The establishment of equilibrium was inferred from the stable densities of the liquid phases (typically 6 d). After equilibration, the precipitate was separated by filtration on a glass sinter. The solubility of $TlIO_3$ was determined by a pycnometric method, which essentially consisted in the determination of the change in the density (Δd) of the initial solution after its saturation with $TlIO_3$. The Δd was converted into solubility (s) by the following procedure: 3 rigorously defined volumes of each initial solution were treated with different samples of $TlIO_3$ so that even the largest dissolved completely; the density of each mixture (d') was then determined. The differences $\Delta d = d' - d_{\text{init}}$ and the $TlIO_3$ concentrations (c') were calculated. After extrapolating the c' values, the value corresponding to a deviation equal to Δd, *i. e.* to the solubility of $TlIO_3$ in the given solution, was found. The densities of the solutions were measured pycnometerically. A narrow-necked 25 cm^3 quartz pycnometer was used; for each mixture the weight of the solution in the pycnometer was measured not less than 3 times.

Source and Purity of Materials:

Sodium triphosphate was prepared by Quimby's method (1). An aqueous solution of containing 12–15 % of commercial $Na_5P_3O_{10}$ (98 mass % pure) was filtered to remove any insoluble matter, and precipitated most of the triphosphate hexahydrate by adding ethanol with stirring until the ratio of ethanol to water was about 1/4 by volume. After 30 min of stirring the hexahydrate crystals were filtered off and washed twice with 1:1 mixtures of ethanol and water, using an aspirator to remove most of the adhering liquid. Usually 4–5 recrystallizations were performed. The crystals of hexahydrate were air-dried at room temperature and finally vacuum dried. The source and purity of thallium iodate, sodium sulfate and sodium hydroxide were not reported.

Estimated Error:

Density: precision $\pm 3 \times 10^{-5}$ g cm^{-3}.
Solubility: precision $\pm 8 \times 10^{-5}$ mol dm^{-3}.
Temperature: precision ± 0.05 K.

References:

[1] O. T. Quimby, J. Phys. Chem. **58**, 603 (1954).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Urea; CH_4N_2O; [57-13-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1797–9 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 979–80 (1974).

Variables:

Concentration of urea: 0–3.0 mol dm^{-3}
One temperature: 293 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 125. Solubility of $TlIO_3$ in aqueous urea solutions at 20 °C

c_2 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
0	1.56
0.600	1.76
0.900	1.86
1.200	1.93
1.500	2.02
1.800	2.08
2.100	2.17
2.400	2.27
2.700	2.37
3.000	2.43

The solubility product of $TlIO_3$ in water was 2.44×10^{-6} mol^2 dm^{-6}.

Auxiliary Information

Method/Apparatus/Procedure:

The initial solutions were saturated with thallium iodate at the selected temperature. The solubility of $TlIO_3$ was determined iodometrically.

Source and Purity of Materials:

No information on source of the chemicals was reported.

Estimated Error:

Solubility: precision $\pm 4 \times 10^{-5}$ mol dm^{-3} (compilers).
Temperature: precision ± 0.05 K (authors).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium nitrate; $NaNO_3$; [7631-99-4]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

A. M. Fedorenko and E. A. Gyunner, Zh. Neorg. Khim. **19**, 2560–3 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 1397–9 (1974).

Variables:

Concentration of $NaNO_3$: 0–3.0 mol dm^{-3}
One temperature: 293 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 126. Solubility of $TlIO_3$ in aqueous solutions containing $NaNO_3$ and $NaClO_4$ at ionic strength of 3 at 20 °C

c_2 / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}
0	7.00
0.300	7.26
0.600	7.51
0.900	7.77
1.200	8.10
1.500	8.39
1.800	8.67
2.100	8.95
2.400	9.23
2.700	9.49
3.000	9.83

The solubility product of $TlIO_3$ was calculated at ionic strength of 3 to be 4.90×10^{-5} mol^2 dm^{-6}.

The formation constants of the ionic pairs $TlNO_3$ and $Tl(NO_3)_2^-$ were calculated from the solubility data.

$$\mathrm{TlIO_3 + NO_3^- \rightleftharpoons TlNO_3 + IO_3^-};\ K_1 = \frac{[\mathrm{TlNO_3}]}{[\mathrm{Tl^+}][\mathrm{NO_3^-}]} = 0.243 \pm 0.004\ \mathrm{mol^{-1}\ dm^3}$$

$$\mathrm{TlIO_3 + 2NO_3^- \rightleftharpoons Tl(NO_3)_2^- + IO_3^-};\ K_2 = \frac{[\mathrm{Tl(NO_3)_2^-}]}{[\mathrm{Tl^+}][\mathrm{NO_3^-}]^2} = 0.0278 \pm 0.0023\ \mathrm{mol^{-2}\ dm^6}$$

Auxiliary Information

Method/Apparatus/Procedure:

The solubilities of thallium iodate in aqueous solution containing sodium nitrate and perchlorate were measured at constant ionic strength. The constancy of ionic strength was obtained by addition of the calculated amounts of $NaClO_4$. The aqueous solutions were shaken with an excess of thallium iodate in a thermostat at the selected temperature for 5 d, after which the liquid phase was separated from the precipitate by filtration and further used for the solubility determinations. The solubility of thallium iodate was determined iodometrically. Every determination was repeated 4 times.

Source and Purity of Materials:

No information was reported.

Estimated Error:

Solubility: precision $\pm 3 \times 10^{-5}$ mol dm^{-3} (authors).

Temperature: precision ± 0.05 K (authors).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Sodium thiosulfate; $Na_2S_2O_3$; [7772-98-7]
(3) Sodium nitrate; $NaNO_3$; [7631-99-4]
(4) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **20**, 1502–6 (1975); Russ. J. Inorg. Chem. (Engl. Transl.) **20**, 841–3 (1975).

Variables:

Concentration of $Na_2S_2O_3$, $NaNO_3$ and $NaClO_4$
One temperature: 293 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 127. Solubility of $TlIO_3$ in aqueous solutions containing $Na_2S_2O_3$, $NaNO_3$ and $NaClO_4$ at ionic strength of 2.7 at 20 °C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	c_4 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
0	2.7	—	8.81
0.1	2.4	—	10.99
0.2	2.1	—	12.92
0.3	1.8	—	14.85
0.4	1.5	—	16.59
0.5	1.2	—	18.50
0.6	0.9	—	20.30
0.7	0.6	—	22.21
0.8	0.3	—	24.13
0.9	0	—	26.17
0	—	2.7	6.49
0.1	—	2.4	8.41
0.2	—	2.1	10.63
0.3	—	1.8	12.76
0.4	—	1.5	15.31
0.5	—	1.2	17.26
0.6	—	0.9	19.20
0.7	—	0.6	21.50
0.8	—	0.3	23.65
0.9	—	0	26.17
0.200	1.700	0.400	12.46
0.200	0.800	1.300	11.31
0.300	1.700	0.100	14.73
0.300	1.200	0.600	14.06

0.370	1.106	0.484	15.46
0.370	0.800	0.790	15.01

The solubility product of $TlIO_3$ in 2.7 mol dm^{-3} $NaClO_4$ was calculated to be 4.21×10^{-5} mol^2 dm^{-6}.

Auxiliary Information

Method/Apparatus/Procedure:

The solubilities were found from the increases in the density of liquid phase of the mixtures when saturation with thallium iodate occurred (1, 2). Every determination was repeated 4 times.

Source and Purity of Materials:

No information was reported.

Estimated Error:

Solubility: precision $\pm 5 \times 10^{-5}$ mol dm^{-3}.

Temperature: precision ± 0.02 K.

References:

[1] E. A. Gyunner and A. M. Fedorenko, Zh. Neorg. Khim. **15**, 1430 (1970); Russ. J. Inorg. Chem. (Engl. Transl.) **15**, 734 (1970).

[2] A. M. Fedorenko, Zh. Neorg. Khim. **19**, 1543 (1974); Russ. J. Inorg. Chem. (Engl. Transl.) **19**, 841 (1974).

Components:

(1) Thallium iodate; $TlIO_3$; [14767-09-0]
(2) Perchloric acid; $HClO_4$; [7601-90-3]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

R. W. Ramette and D. A. Palmer, J. Solution Chem. **13**, 637–46 (1984).

Variables:

Concentration of $HClO_4$ (0–0.1011 mol dm^{-3}) and $NaClO_4$ (0–0.1 mol dm^{-3})
Temperature: 275.4–348.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 128. Solubility of $TlIO_3$ in aqueous solutions of $HClO_4$ and $NaClO_4$ at various temperatures

t / °C	m_2 / mol kg^{-1}	m_3 / mol kg^{-1}	$10^3 m_1$ / mol kg^{-1}
2.2	0	0.1	1.009
3.3	0	0.1	1.040
15.2	0	0.1	1.714
25.0	0	0.1	2.410
30.0	0	0.1	2.876
35.4	0	0.1	3.420
50.0	0	0.1	5.320
60.0	0	0.1	6.996
75.0	0	0.1	10.10
2.2	0.1011	0	1.141
3.3	0.1011	0	1.182
15.2	0.1011	0	1.960
25.0	0.1011	0	2.801
30.0	0.1011	0	3.374
35.4	0.1011	0	4.026
50.0	0.1011	0	6.361
60.0	0.1011	0	8.518
75.0	0.1011	0	12.53
25.0	0.0001	0	1.834[a]

[a] The value was calculated from the concentration of thallium and iodate ions by the compilers: $[Tl^+] = 1.828 \times 10^{-3}$, $[IO_3^-] = 1.839 \times 10^{-3}$ mol kg^{-1}.

TABLE 129. Solubility product of $TlIO_3$ in the electrolyte solutions at various temperatures

t / °C	$10^7 K_s$ / mol^2 kg^{-2}
2.2	5.52
3.3	5.85
15.2	15.70
25.0	30.68
30.0	43.40
35.4	60.90
50.0	143.9
60.0	244.1
75.0	492.4

A least squares fit yields the equation (the authors)

$$\log K_s = \frac{-4550.5}{T} + 46.164 - 14.715 \log T \tag{56}$$

From this the authors calculated enthalpy, entropy an heat capacity for dissociation of solid thallium iodate at 25.0 °C equal 12.10 ± 0.06 kcal mol^{-1}, 15.4 ± 0.2 cal K^{-1} mol^{-1} and -29 ± 4 cal K^{-1} mol^{-1}, respectively.

Auxiliary Information

Method/Apparatus/Procedure:

Saturated solutions of thallium iodate were prepared in a two-compartment vessel. About 3 g of the salt was placed in each compartment, along with 300 cm^3 of solution. The entire vessel was immersed in a constant temperature bath of water or, at higher temperatures, ethylene glycol. A positive argon pressure inside the vessel was used to force samples of either solution through glass wool plugs into preweighed weight pipettes. Thallium iodate was equilibrated with 10^{-4} mol dm^{-3} $HClO_4$ in a rotated centrifuge tube for the determinations of K_s at 25 °C.

Analyses of the solutions were carried out by potentiostatic coulometry with 100 % current efficiency. For the iodate determination, a cell electrolyte was 1 mol dm^{-3} NaI, and 0.03 mol dm^{-3} H_2SO_4, and deoxygenated with argon. For thallium determinations the cell electrolyte was 2 mol dm^{-3} NaCl, 0.1 mol dm^{-3} CH_3COONa and 0.01 mol dm^{-3} CH_3COOH.

Source and Purity of Materials:

Thallium iodate was prepared by the slow and simultaneous addition of 0.05 mol dm^{-3} Tl_2CO_3 to an equivalent amount of 0.1 mol dm^{-3} HIO_3 in 2 dm^3 of rapidly stirred water at 60 °C. The precipitate was washed several times with water, collected on a glass frit filter, and air-dried at room temperature.

Regent grade sodium perchlorate monohydrate (from G. F. Smith) was used to prepare the 0.1 mol kg^{-1} solution. A 0.1011 mol kg^{-1} $HClO_4$ solution was standardized by titration with NaOH. Double distilled water was used.

Estimated Error:

Solubility: precision better than ± 0.1 % (estimated by the compilers from the background charge).

Temperature: nothing specified; precision better than ± 0.1 K (estimated by the compilers).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. December 2001.

Critical Evaluation:

The Binary System

Data for the solubility of lead iodate in water have been reported in 12 publications. (1–10, 13, 15). Polessitskii (6) measured the solubilities over the wide temperature range 273–373 K and Kohlrausch made it between 282 and 299 K (2). Other investigators (1, 3, 5, 7–10, 13, 15), measured the solubility at one temperature. The solid phase equilibrated with the saturated solution is the anhydrous salt, $\mathrm{Pb(IO_3)_2}$ and other equilibrium solid phases of varying degree of hydration were not reported.

A summary of the experimental solubility data for the binary system is given in Table 130. Geilmann and Höltje (4) have reported the solubility of $\mathrm{Pb(IO_3)_2}$ in water at exactly unspecified temperature.

TABLE 130. Summary for solubility studies of $\mathrm{Pb(IO_3)_2}$ in water at various temperatures

T / K	$10^5 c_1$ / mol dm^{-3}	Analytical method	Reference
273	2.70	Iodometry	(6)
282.32	2.40	Conductometry	(2)
290.3	3.11	Conductometry	(2)
290-293	5.2	Iodometry	(4)
291	3.20	Conductometry	(2)
293.10	3.25 (2.79)^a	Conductometry	(1)
298	3.58	Iodometry	(13)
298	3.608	Iodometry	(5)
298.15	3.61	Iodometry	(7)
298	3.61	Potentiometry	(9)
298	5.255	Gravimetry	(6)
298.15	5.34	Iodometry	(7)
298	5.4	Iodometry (IO_3^-), Gravimetry (Pb^{2+})	(15)
298	5.51	Iodometry	(3)
298.92	4.13	Conductometry	(2)
303	5.0	Spectrophotometry	(10)
308	5.085	No information	(8)

313	7.95	Iodometry	(6)
323	9.34	Iodometry	(6)
333	10.9	Iodometry	(6)
343	14.3	Iodometry	(6)
351	20.5	Iodometry	(6)
373	49.5	Iodometry	(6)

[a] Corrected value, calculated by the compilers, is given in the parenthesis.

Solubility at 298 K The large difference in the solubility values is observed in Table 130. Keefer and Reiber (7) prepared the sample of lead iodate by two different methods. When much larger amount of water was used during the salt preparation the larger crystals were obtained and the solubility determined was lower for 30 %. Earlier, a similar observation was reported by La Mer and Goldman (5).

The solubility value of the iodate prepared in concentrated solution was 5.34×10^{-5} mol dm^{-3} (7), being in agreement with 5.51×10^{-5} of Harkins and Winninghoff (3), 5.4×10^{-5} of Cuny (15), and 5.26×10^{-5} mol dm^{-3} of Polessitskii (6). The solubility of the salt sample obtained in diluted solutions was 3.61×10^{-5} mol dm^{-3} by Keefer and Reiber (7). This value is identical with the result of La Mer and Goldman (5) and of Basinski and Warnke (9). It is slightly different from Edomonds and Birnbaum (13) value of 3.58×10^{-5} mol dm^{-3}. The iodate sample used by Basinski and Warnke (9) was prepared by same method as used La Mer and Goldman (5). Keefer and Reiber (7) stated that the large difference between these two groups of solubility values may be explained by the difference in lead iodate particle size due to its preparation method.

Since greater care was given in (5, 7, 9, 13) to performance of the solubility determinations, the evaluators believe that these data better reflect true solubility and the value of 3.6×10^{-5} mol dm^{-3} may be designated as a tentative one at 298 K.

Solubility at 308 K Misra and Pani (8) have reported the solubility of the lead iodate prepared in the diluted solution. They stated that a hydrolysis in the saturated aqueous solution of lead iodate was found to be appreciable and the solubility product of lead iodate should be corrected. Their value of the solubility determined was 5.08×10^{-5} mol dm^{-3} and may be designated as a tentative one at 308 K.

Solubility over 273–373 K Only one publication is available for the solubilities of $\mathrm{Pb(IO_3)_2}$ over the temperature range 273–373 K by Polessitskii (6). From the result obtained at 298 K, the evaluators assumed that he used a sample of the iodate prepared in concentrated solution and all his results at other temperature are certainly overestimated. No smoothing equation taking into account either the results from (6) or from all papers seems to be rational in this situation. The extrapolated results of Kohlrausch (2) obtained between 282 and 299 K are about 20 % lower than those of Polessitskii (6) but they are still larger for 10 % from the tentative values at 298 K and 308 K.

The detailed information of the calculation procedure for the smoothing equation has been described in the Preface in this volume. All experimental solubilities given in Table 130 were used to calculate the smoothing equation, where the value of 2.79×10^{-5} mol dm^{-3} was used from reference (1). The obtained smoothing equation is

$$\ln(c_1/\text{mol dm}^{-3}) = \frac{1138.93468}{(T/\text{K})} - 8.57269711\ \ln(T/\text{K}) + 14.639485 + 0.0681063856(T/\text{K}), \tag{57}$$

where $\sigma_c = 1.2 \times 10^{-5}$.

The calculated smoothing curve is plotted in the Fig. 12. The solubilities calculated from the smoothing equation at various temperatures are given in Table 131. Their solubilities are designated as doubtful values, because the standard error of the coefficients in above equation is relatively large and is not in agreement with the tentative values at 298 and 308 K selected above.

TABLE 131. Doubtful solubility of $\mathrm{Pb(IO_3)_2}$ in water

T / K	$10^5 c_1$ / mol dm^{-3}
273	2.3
283	2.9
293	3.7
298	4.2
303	4.8
313	6.3
323	8.6
333	11.7
343	16.3
353	22.9
363	32.5
373	46.8

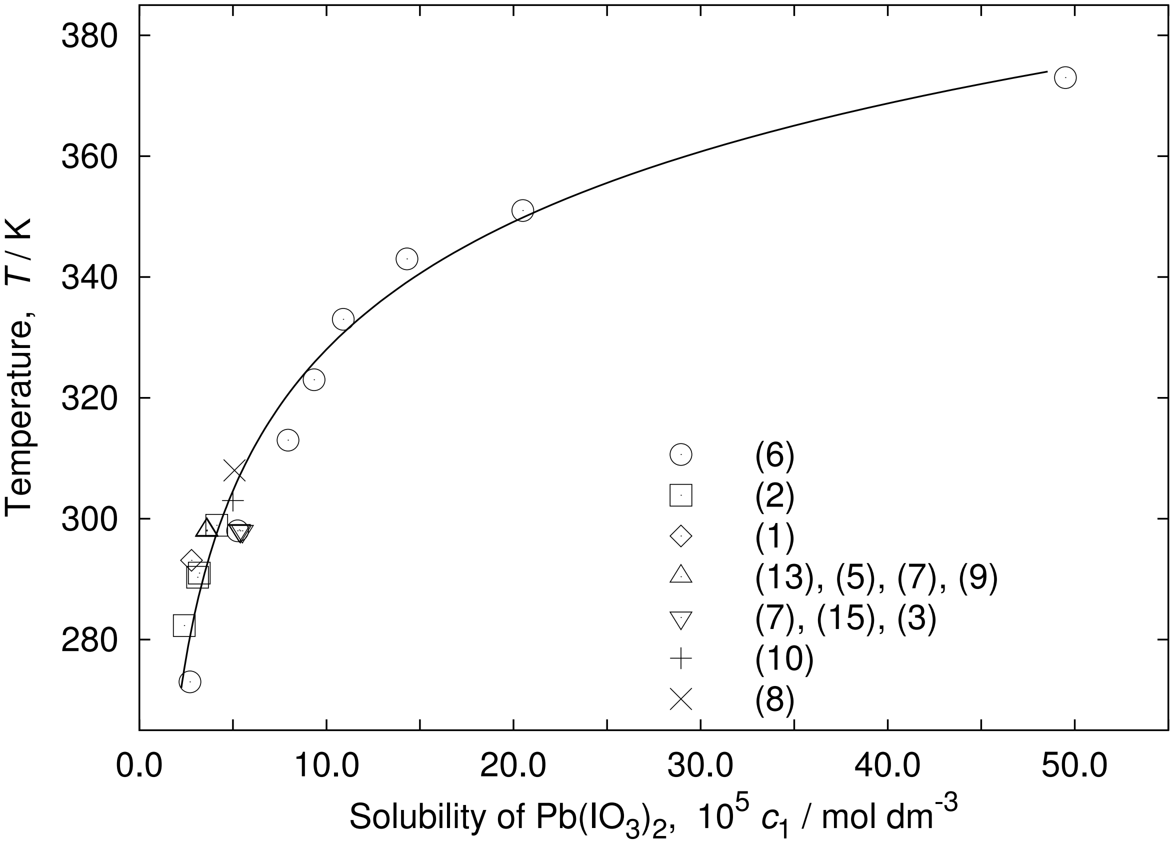

FIG. 12. Temperature dependence of solubility of $\mathrm{Pb(IO_3)_2}$ in water

The Ternary System

Data for the solubility of lead iodate in aqueous solutions containing alkali metal salts as the unsaturating components have been reported in 6 publications (3, 5, 8, 11, 12, 14). The solubility in nitric acid (14), perchloric acid (15) and the aqueous solutions of amino acids (7) have been also reported. A summary of the solubility studied for the aqueous ternary systems containing $\mathrm{Pb(IO_3)_2}$ as only one saturating component is given in Table 132. In the study of Geilmann and Höltje (4), the temperature was not precisely specified.

The solubility of lead iodate increases with increasing the concentration of the added salts as well as mineral acids. Typically, in the aqueous solution of potassium iodate the solubility of $\mathrm{Pb(IO_3)_2}$ decreased with increased concentration of potassium iodate. In both amino acid (glycine and alanine) solutions, plots of the logarithm of the solubility against the amino acid concentration showed abnormal slopes. Keefer and Reiber (7) explained that the abnormal slops may be caused by complex formation between lead and glycine or alanine ions. In the similar way a presence of sodium monochloroacetate influenced the solubility of lead iodate (12).

TABLE 132. Summary for solubility studies of $\mathrm{Pb(IO_3)_2}$ in the aqueous ternary systems

Ternary system	T / K	Added compound c_2 / mol dm^{-3}	$10^4 c_1$ / mol dm^{-3}	Reference
$\mathrm{Pb(IO_3)_2–Pb(NO_3)_2–H_2O}$	298	0–1.5	0.551–0.75	(3)

$Pb(IO_3)_2$–KNO_3–H_2O	298	0–0.200	0.551–1.272	(3)
$Pb(IO_3)_2$–KNO_3–H_2O	298	0–0.1	0.3608–0.694	(5)
$Pb(IO_3)_2$–KNO_3–H_2O	308	0.01–0.20	0.657–1.212	(11)
$Pb(IO_3)_2$–$NaNO_3$–H_2O	298	0–0.1	0.3608–0.699	(5)
$Pb(IO_3)_2$–$NaNO_3$–H_2O	308	0.02–0.30	0.6840–1.335	(14)
$Pb(IO_3)_2$–HNO_3–H_2O	308	0.02–0.30	0.6845–2.021	(14)
$Pb(IO_3)_2$–KCl–H_2O	298	0–0.1	0.3608–0.829	(5)
$Pb(IO_3)_2$–NaCl–H_2O	298	0–0.1	0.3608–0.830	(5)
$Pb(IO_3)_2$–$NaClO_4$–H_2O	308	0–0.30	0.5085–0.9860	(8)
$Pb(IO_3)_2$–$HClO_4$–H_2O	308	0–0.30	0.5085–1.517	(8)
$Pb(IO_3)_2$–KIO_3–H_2O	290–293	$0–6.54 \times 10^{-4}$	0.52–0.007	(4)
$Pb(IO_3)_2$–KIO_3–H_2O	298	$0–1.06 \times 10^{-3}$	0.551–0.219	(3)
$Pb(IO_3)_2$–glycine–H_2O	298	0–0.2023	0.534–0.941	(7)
$Pb(IO_3)_2$–glycine–H_2O	298	0–0.100	0.361–0.509	(7)
$Pb(IO_3)_2$–alanine–H_2O	298	0–0.08142	0.534–0.717	(7)
$Pb(IO_3)_2$–alanine–H_2O	298	0–0.100	0.361–0.577	(7)
$Pb(IO_3)_2$–sodium monochloroacetate–H_2O	298	0.01–0.20	0.715–2.105	(12)

Misra and Pani studied the solubilities for quaternary $Pb(IO_3)_2$–$HClO_4$–$NaClO_4$–H_2O (8), $Pb(IO_3)_2$–$HClO_4$–KIO_3–H_2O (8) and $Pb(IO_3)_2$–HNO_3–$NaNO_3$–H_2O (14) systems. In these studies the hydrolysis of divalent lead ion in the saturated solution of lead iodate was taken into account and discussed. Mohanty and Aditya studied the solubilities of $Pb(IO_3)_3$ in aqueous solutions containing propionic acid and sodium propionate (11), and lactic acid and sodium lactate (12). They have reported the formation of the lead complexes in these conditions. Edomonds and Birnbaum (13) measured the solubilities for lead iodate in solutions containing ammonium acetate and perchlorate at ionic strength of 1. They stated that the interaction involved the formation of $PbC_2H_3O_2^+$. Basinski and Warnke (9) measured the solubilities of lead iodate in aqueous sodium glycolate at constant ionic strength of 3 and at pH = 5. From these solubility data, the complex formation of lead(II) with glycolic acid in the aqueous solution has been deduced.

References:

1. W. Böttger, Z. Phys. Chem. **46**, 521 (1903).
2. F. Kohlrausch, Z. Phys. Chem. **64**, 129 (1908).
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14. R. N. Misra and S. Pani, J. Indian Chem. Soc. **34**, 393 (1957).
15. L. Cuny, J. Pharm. Chim. **28**, 154 (1923).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

W. Böttger, Z. Phys. Chem. **46**, 521–619 (1903).

Variables:

One temperature: 293.10 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 133. Solubility of $\mathrm{Pb(IO_3)_2}$ in water at 293.10 K by conductivity method

t / °C	Equilibration time / h	$\kappa_{\text{(satd. soln.)}}$/ 10^6 S cm^{-1}	$\kappa_{\text{(water)}}$/ 10^6 S cm^{-1}	κ/ 10^6 S cm^{-1}	$10^5 c_1$ / mol dm^{-3} [a]	$10^2 \gamma_1$ / g dm^{-3}
19.95	18	6.42	0.97	5.45	2.73	1.52
19.95	25	6.43	0.97	5.46	2.73	1.52
19.95	27	6.43	0.97	5.46	2.73	1.52
19.94	18	6.82	0.94	5.88	2.94	1.64
19.94	21	6.66	0.94	5.72	2.86	1.59
19.94	19	6.53	0.94	5.59	2.80	1.56
19.94	27	6.46	0.94	5.52	2.76.	1.53
		(av.) 6.53	(av.) 0.95	(av.) 5.58	(av.) 2.79	(av.) 1.55

[a] Calculated by the compilers.

The author reported the solubility of $\mathrm{Pb(IO_3)_2}$ of 6.5×10^{-5} g equiv. liter^{-1} units. The average conductivity of κ and the molar conductances (λ^{∞}) of 64.5 and 35.5 S cm^2 mol^{-1} for $\frac{1}{2}$Pb^{2+} and IO$_3^-$, respectively, were used to calculate the solubility from eqs. (58) and (59). The compilers reculculated the solubility of 2.79 mol dm^{-3} from the data reported by the original author.

$$\kappa = \kappa_{\text{(satd. soln.)}} - \kappa_{\text{(water)}} \tag{58}$$

$$c_1 = \frac{1000\,\kappa}{2\left\{\lambda^{\infty}_{\frac{1}{2}\mathrm{Pb}^{2+}} + \lambda^{\infty}_{\mathrm{IO}_3^-}\right\}} \tag{59}$$

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of $\mathrm{Pb(IO_3)_2}$ in water was determined from measurements of electrolytic conductivities of the saturated solution and of pure water. The conductivity cell, which employed Pt electrodes, was designed to prevent entrapment of air bubbles. After sealing, the entire cell was immersed in a water bath at the selected temperature and rotated in the dark for 18–27 h. Equilibrium was reached when the conductivity remained constant within experimental error. The conductance bridge and the thermometer were calibrated. The cell constant was determined with 0.010 mol dm^{-3} KCl solution. The conductometric results of the solubility were confirmed by iodometric titration of the saturated solution with sodium thiosulfate after addition of potassium iodide.

Source and Purity of Materials:

Lead iodate was prepared from lead nitrate and potassium iodate. All salts (from Merck) were recrystallized several times. Water was twice distilled.

Estimated Error:

Solubility: precision ± 1 %.
Temperature: stability ± 0.1 K.

Components:
(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. Kohlrausch, Z. Phys. Chem. **64**, 129–69 (1908).

Variables:
Temperature: 282.32–298.92 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 134. Solubility of $\mathrm{Pb(IO_3)_2}$ in water at various temperatures

t / °C	$10^6\kappa$ / S cm^{-1}	$10^5 c_1$ / mol dm^{-3} [a]	$10^2\gamma_1$ / g dm^{-3} [b]	$10^6\kappa^*$ / S cm^{-1}
9.17	4.49	2.40	1.34	3.58
17.1	5.79	3.11	1.73	5.67
18	5.96	3.20	1.78	5.96
25.77	7.68	4.13	2.30	9.14

[a] Calculated by the compilers; the equivalent values reported by the author were given in "g equiv. liter^{-1}" units.

[b] Calculated by the author.

For pure water the author found $\kappa_{(\text{water})} = (1.3\text{–}1.4) \times 10^{-6}$ S cm^{-1}, but individual values for each temperature were not given. There is some confusion as to the significance of κ^* values. The solubilities were calculated from the corrected κ values (eqs. (60) and (61) below), and the κ^* values are probably the actually measured values being subject to correction for temperature change of the molar conductivities. The effect of temperature on the electrolytic conductivities is given in eq. (62).

At 18 °C, the molar conductivities (infinite dilution) of $\frac{1}{2}$Pb^{2+} and IO$_3^-$ are 61 and 33.9 S cm^2 mol^{-1}, respectively, and individual values corrected for infinite concentrations at each temperature are given below. The solubilities were calculated by using eqs. (60) and (61).

$$\kappa = \kappa_{(\text{satd. soln.})} - \kappa_{(\text{water})} \tag{60}$$

$$c_1 = \frac{1000\,\kappa}{\Lambda} \tag{61}$$

Temperature dependencies of conductivity were calculated from

$$\kappa_t = \kappa_{18}[1 + 0.0238(t - 18) + 0.0001(t - 18)^2]. \tag{62}$$

The molar conductances corrected for infinity diluted concentration are given below:

t / °C	Λ / S cm^2 mol^{-1}
9.17	93.5
17.1	93.3
18	93.3
25.77	93.1

Auxiliary Information

Method/Apparatus/Procedure:

The solubility determination was performed by conductivity method (1). Conductivities of pure water and the saturated solutions of lead iodate were measured in a glass cell with platinum electrodes and a thermometer. The cell was dipped in a water bath. The solubility was calculated from the conductivities of the saturated solution, pure water and molar conductivities of both ions.

Source and Purity of Materials:

Lead iodate was prepared from lead nitrate and sodium iodate. It was hot extracted and pulverized after drying.

Estimated Error:

Solubility: better than ± 1 % (1).

Temperature: nothing specified; precision probably better than ± 0.1 K (compilers).

References:

[1] F. Kohlrausch, Z. Phys. Chem. **44**, 197 (1903).

Components:

(1) Lead iodate; $Pb(IO_3)_2$; [25659-31-8]
(2) Lead nitrate; $Pb(NO_3)_2$; [10099-74-8]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Potassium iodate; KIO_3; [7758-05-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

W. D. Harkins and W. S. Winninghoff, J. Am. Chem. Soc. **33**, 1827–35 (1911).

Variables:

Concentration of added salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 135. Solubility of $Pb(IO_3)_2$ in water and aqueous solutions of inorganic salts at 25 °C

Added salt	Concn. added salt[a]	c_2 / mol dm^{-3} [b]	Soly. $Pb(IO_3)_2$ [c]	$10^4 c_1$ / mol dm^{-3} [b]
None	0	0	1.105	
	0	0	1.102	
	0	0	1.107	
	0	0	1.101 (s)	
	0	0	1.097 (s)	
	0	0	1.194 (s) [d]	
	0	0	1.097 (s)	
			(av.) 1.102	0.551
$Pb(NO_3)_2$	0.0001	0.00005	0.870	
	0.0001	0.00005	0.870 (s)	
			(av.) 0.870	0.435
	0.001	0.0005	0.408	
	0.001	0.0005	0.414 (s)	
			(av.) 0.411	0.206
	0.010	0.0050	0.185	
	0.010	0.0050	0.186 (s)	
			(av.) 0.186	0.093
	0.100	0.050	0.16	
	0.100	0.050	0.16 (s)	
			(av.) 0.16	0.08
	0.500	0.25	0.30	
	0.500	0.25	0.27 (s)	
			(av.) 0.29	0.15
	3.0	1.5	1.5	
	3.0	1.5	1.5 (s)	
			(av.) 1.5	0.75

KNO_3	0.002	0.002	1.145	
	0.002	0.002	1.138 (s)	
			(av.) 1.141	0.571
	0.010	0.010	1.334	
	0.010	0.010	1.334 (s)	
			(av.) 1.334	0.667
	0.050	0.050	2.037	
	0.050	0.050	2.037 (s)	
			(av.) 2.037	1.019
	0.200	0.200	2.548	
	0.200	0.200	2.540 (s)	
			(av.) 2.544	1.272
KIO_3	0.00005304	0.00005304	0.717	
	0.00005304	0.00005304	0.697	
	0.00005304	0.00005304	0.714 (s)	
	0.00005304	0.00005304	0.734 (s)	
			(av.) 0.715	0.358
	0.00010601	0.00010601	0.432	
	0.00010601	0.00010601	0.442 (s)	
			(av.) 0.437	0.219

[a] Concentration in equivalents per liter.

[b] Calculated by the compilers.

[c] Solubility in 10^{-4} equivalents per liter.

[d] The value was rejected owing to exceed 2σ.

(s) The value was obtained by approaching equilibrium from the side of supersaturation.

Auxiliary Information

Method/Apparatus/Procedure:

The saturated solutions were obtained by rotating the corresponding salts and water in glass bottles for 24 h in a thermostat (1). The solutions were filtered through an asbestos filter. Some of the data were obtained by approaching equilibrium from the side of supersaturation reached at 40 °C. The concentration of the saturated solution was determined by adding KI to the solution of the iodate, and liberated iodine was determined by titration with sodium thiosulfate solution standardized against pure iodine and against a solution of pure copper sulfate.

Source and Purity of Materials:

Lead iodate was made by mixing lead nitrate with potassium iodate as very diluted solutions; the nitrate was kept constantly in excess. The product was washed with water. Potassium iodate and lead nitrate (of Kahlbaum) were recrystallized twice from water. Conductivity water was used.

Estimated Error:

Solubility: precision between ± 0.05 % and ± 0.3 % (compilers).
Temperature: precision ± 0.01 K (authors (1)).

References:

[1] W. D. Harkins, J. Am. Chem. Soc. **33**, 1807 (1911).

Components:
(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
L. Cuny, J. Pharm. Chim. **28**, 154–8 (1923).

Variables:
One temperature: 298 K

Prepared By:
Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Solubility of $\mathrm{Pb(IO_3)_2}$ in water at 25 $^\circ\mathrm{C}$ is 30 mg dm^{-3} or 5.4×10^{-5} mol dm^{-3} (as calculated by the compilers).

Auxiliary Information

Method/Apparatus/Procedure:
A saturated solution of $\mathrm{Pb(IO_3)_2}$ was prepared by an unspecified procedure. 10 cm^3 of 25 % $\mathrm{H_2SO_4}$ and 20 cm^3 of 10 % KI were added to 50 cm^3 aliquot of the saturated solution filtrate. After 15 min, the solution was titrated with standard 0.05 mol dm^{-3} $\mathrm{Na_2S_2O_3}$. Results of 11 determinations with $\mathrm{Pb(IO_3)_2}$ precipitated from $\mathrm{Pb(CH_3COO)_2}$ and KIO_3 determined by iodometry by similar procedure agreed with the results of gravimetric determination by means of $PbCrO_4$ precipitation.

Source and Purity of Materials:
$\mathrm{Pb(IO_3)_2}$ was prepared from $\mathrm{Pb(CH_3COO)_2}$ and KIO_3 of unspecified purities.

Estimated Error:
Solubility: differences between the iodometric and gravimetric determinations were maximally 1 % but typically 0.5 %.
Temperature: nothing specified.

Components:
(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Potassium iodate; KIO_3; [7758-05-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
W. Geilmann and R. Höltje, Z. Anorg. Allg. Chem. **152**, 59–72 (1926).

Variables:
Concentration of KIO_3: 0–0.00065 mol dm^{-3}
Unstable temperature: 290–293 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 136. Solubility of $\mathrm{Pb(IO_3)_2}$ in water at 17–20 °C

Standing time / h	γ_1 / mg dm^{-3}	$10^5 c_1$ / mol dm$^{-3\mathrm{a}}$
0.3	47	
0.3	45	
	(av.) 46	8.4
1.0	45	
1.0	37	
	(av.) 41	7.4
1.5	31	
1.5	31	
	(av.) 31	5.6
2.0	29	
2.0	29	
	(av.) 29	5.2
7.0	30	
7.0	31	
	(av.) 31	5.6
14	28	
14	27	
	(av.) 28	5.0
78	27	
78	29	
	(av.) 28	5.0
	(av.) 29 ± 2 [b]	5.2 ± 0.4

[a] Calculated by the compilers.

[b] The average value was calculated from the last 5 average values by the authors.

TABLE 137. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of $\mathrm{KIO_3}$ at 17–20 °C

γ_2 / mg dm^{-3}	$10^4 c_2$ / mol dm^{-3}	γ_1 / mg dm^{-3}	$10^4 c_1$ / mol dm^{-3} [a]	$10^{13} K_s$ / mol^3 dm^{-9}
4.3	0.20	20.5		3.3 (3.2)[b]
4.3	0.20	20.5		3.3 (3.2)[b]
		(av.) 20.5	0.368	(av.) 3.3 (3.2)[b]
10.7	0.500	14.8		2.8
10.7	0.500	15.5		3.1
		(av.) 15.2	0.273	(av.) 3.0
19.3	0.902	9.9		2.8
19.3	0.902	10.3		3.0
		(av.) 10.1	0.181	(av.) 2.9
49.0	2.29	3.9		4.2 (4.1)[b]
49.0	2.29	2.5		2.6 (2.5)[b]
		(av.) 3.2	0.057	(av.) 3.4 (3.3)[b]
96.0	4.49	1.1	0.020	4.0
96.0	4.49	(2.5)	(0.045)	—
140	6.54	0.4	0.007	3.0

[a] Calculated by the compilers.

[b] Recalculated by the compilers using the 2003 IUPAC recommended atomic weighs.

Auxiliary Information

Method/Apparatus/Procedure:

The lead iodate was shaken with water or an aqueous solution of potassium iodate. After standing for various times and centrifuging, an aliquot of the saturated solution was analyzed for the iodate ion by iodometric titration. Potassium iodide and hydrochloric acid were added before titration with thiosulfate solution. The solubility of lead iodate in presence of potassium iodate was calculated from the total iodate concentration determined and the potassium iodate concentration added.

Source and Purity of Materials:

Lead iodate was prepared by mixing lead chloride solution with an equivalent amount of potassium iodate solution. The solution was centrifuged immediately, and the precipitate was washed with water 4 times giving pure $\mathrm{Pb(IO_3)_2}$.

Estimated Error:

Solubility: precision ± 7 %.

Temperature: nothing specified on its stability.

Components:

(1) Lead iodate; $Pb(IO_3)_2$; [25659-31-8]
(2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Sodium chloride; NaCl; [7647-14-5]
or (2) Potassium chloride; KCl; [7447-40-7]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791–3 (1930).

Variables:

Concentration of alkali metal salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 138. Solubility of $Pb(IO_3)_2$ in aqueous solutions of various salts at 25 °C

Added salt	c_2 / mol dm^{-3}	Number of determination	$10^5 c_1$ / mol dm^{-3}
None	—	5	3.608
$NaNO_3$	0.1	3	6.99
KNO_3	0.1	3	6.94
NaCl	0.1	4	8.30
KCl	0.1	4	8.29

The authors observed that small crystals (precipitated from more concentrated solutions) produced higher solubility results.

Auxiliary Information

Method/Apparatus/Procedure:

The saturating column method was used as described in details in (1). Lead iodate was placed in solubility tubes, washed with water or the salt solutions circulated continuously for several months. Equilibrium was approached from supersaturation as well as from undersaturation. The analysis of the iodate content was carried out by adding 3 g of KI to a 200 cm^3 aliquot of the saturated solution and 2 cm^3 50% H_2SO_4 was then added. It was titrated with dilute thiosulfate solution from a weight buret after 10 min. Starch was used as an indicator.

Source and Purity of Materials:

Lead iodate was precipitated by simultaneous dropwise addition of equivalent solutions of chemically pure lead nitrate and potassium iodate in a large amount of water under constant stirring at about 60 °C. The supernatant liquid was replaced with fresh water 8 times during the precipitation process. No information on source of alkali metal salts was given.

Estimated Error:

Solubility: precision ± 0.2 %. (authors).

Temperature: precision ± 0.005 K (authors (1)).

References:

[1] V. K. La Mer, C. V. King, and C. F. Mason, J. Am. Chem. Soc. **49**, 363 (1927); J. N. Brönsted and V. K. La Mer, J. Am. Chem. Soc. **46**, 555 (1924).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Potassium iodate; KIO_3; [7758-05-6]
or (2) Calcium nitrate; $\mathrm{Ca(NO_3)_2}$; [10124-37-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

A. Polessitskii, C. R. Dokl. Acad. Sci. URSS **4**, 197–200 (1935).

Variables:

Concentration of inorganic salts
Temperature: 273–373 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 139. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of inorganic salts at 0–100 °C

Added salt	t / °C	c_2 / mol dm^{-3}	γ_1 / g dm^{-3}	$10^4 c_1$ / mol dm^{-3}
None	0	—	0.01504	0.270
	25	—	0.02925	0.5255
	40	—	0.0443	0.795
	50	—	0.0520	0.934
	60	—	0.0610	1.09
	70	—	0.0795	1.43
	78	—	0.114	2.05
	100	—	0.275	4.95
KNO_3	25	0.2	0.0585	1.051
		1.4	0.186	3.33
		2.25	0.307	5.51
KIO_3	25	0.0001	0.0076	0.136
		0.001	0.0003	0.0053
$\mathrm{Ca(NO_3)_2}$	25	1.30	0.362	6.51
		2.27	0.585	10.50
		3.07	0.803	14.44
		3.6	0.929	16.7
		4.22	1.030	18.5
		5.18	1.100	19.7

Auxiliary Information

Method/Apparatus/Procedure:

Lead iodate crystals were stirred with water in a thermostat for 24 h. After settling the solutions for 1 h, their samples were withdrawn with a pipet equipped with a cotton-wool filter. The solubilities at high temperatures were determined in a special apparatus with facilities to keep the saturated solutions at constant temperature by the vapor of boiling liquids: water and ethanol. The solubility in water at 25 $^\circ\mathrm{C}$ was determined by the evaporation method and those at the other temperatures were determined iodometrically. The solubility of lead iodate in concentrated potassium iodate solutions was determined by the method of radioactive indicator.

Source and Purity of Materials:

Lead iodate was prepared by adding solutions containing equivalent quantities of recrystallized lead nitrate and potassium iodate. The precipitate was filtered off and washed with hot water, or washed by decantation with large amount of cold water during strong stirring. Source and purity of KNO_3, $\mathrm{Ca(NO_3)_2}$ and KIO_3 was not given.

Estimated Error:

Nothing specified.

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]

(2) Ammonium perchlorate; $\mathrm{NH_4ClO_4}$; [7790-98-9]

(3) Ammonium acetate; $\mathrm{NH_4C_2H_3O_2}$; [631-61-8]

(4) Water; H_2O; [7732-18-5]

Original Measurements:

S. M. Edomonds and N. Birnbaum, J. Am. Chem. Soc. **62**, 2367–9 (1940).

Variables:

Concentration of ammonium perchlorate and acetate

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 140. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of ammonium perchlorate and acetate at 25 °C

c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$10^4 c_1$ / mol dm^{-3}
0	0	0.358
0	1.00	1.950
0.01	0.99	2.842
0.02	0.98	3.036
0.05	0.95	3.557
0.10	0.90	4.370
0.20	0.80	5.584
0.50	0.50	7.265
1.00	0	9.11

Stability constant of $\mathrm{PbC_2H_3O_2^+}$ ion was found to be 100 mol^{-1} dm^3.

Auxiliary Information

Method/Apparatus/Procedure:

Solutions of unit ionic strength were prepared by dilution of ammonium acetate solution with ammonium perchlorate solution. The mixtures, to which a few grams of lead iodate had been added, were contained in glass-stoppered 125 cm^3 Pyrex flasks which were shaken at the selected temperature for 3–7 d. Dissolved iodate was determined upon filtered aliquots iodometrically. KI and HCl were added and the liberated I_2 titrated with $\mathrm{Na_2S_2O_3}$ solution.

Source and Purity of Materials:

Lead iodate was prepared by simultaneous dropwise addition of equivalent solutions of lead nitrate and potassium iodate in a large volume of water under constant stirring at about 60 °C. The supernatant liquid was replaced with fresh water 8 times during precipitation process (1). The precipitate dried at 110 °C was analyzed for iodate by iodometric titration. The purity of the prepared lead iodate was 99.91 %. Reagent grade ammonium perchlorate and acetate were used.

Estimated Error:

Solubility: precision ± 0.3 %.

Temperature: precision ± 0.01 K.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791 (1930).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Alanine; $\mathrm{C_3H_7NO_2}$; [6898-94-8]
or (2) Glycine (aminoacetic acid); $\mathrm{C_2H_5NO_2}$; [56-40-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Keefer and H. G. Reiber, J. Am. Chem. Soc. **63**, 689–92 (1941).

Variables:

Concentration of alanine (0–0.1 mol $\mathrm{dm^{-3}}$) and concentration of glycine (0–0.2 mol $\mathrm{dm^{-3}}$)
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto and Cezary Guminski

Experimental Data

TABLE 141. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of alanine and glycine at 25.00 °C

Amino acid	Sample number of $\mathrm{Pb(IO_3)_2}$	c_2 / mol kg^{-1}	$10^5 c_1$ / mol kg^{-1}
Alanine[a]	I	0	5.34 ± 0.07
		0.01081	5.56
		0.01521	5.65
		0.02163	5.69
		0.02704	6.08
		0.05419	6.30
		0.08142	7.17
	II	0	3.61 ± 0.04
		0.0248	4.05
		0.0495	4.66
		0.075	5.06
		0.100	5.57
Glycine	I	0.00502	5.47
		0.01003	5.63
		0.01505	5.65
		0.02510	6.15
		0.05026	6.40
		0.08052	6.75
		0.10074	7.51
		0.1210	7.70
		0.2023	9.41
	II	0.0249	4.05
		0.0499	4.45
		0.0749	4.81
		0.100	5.09

[a] Information about both optical activity and structural isomer was not described by the authors.

The authors stated that the large differences in the results from the two sources of $\mathrm{Pb(IO_3)_2}$ may be explained by the difference in particle size due to the method of preparation and solubility determinations of lead iodate.

Dissociation constants of lead glycinate and lead alaninate ions were determined to be 6.7×10^{-6} and 3.0×10^{-6} mol dm^{-3}, respectively.

Auxiliary Information

Method/Apparatus/Procedure:

The method of solubility determination was described in (1). Alanine and glycine solutions were prepared from boiled water using calibrated volumetric equipment. An excess of lead iodate was placed in a glass-stoppered Pyrex flask and alanine or glycine solution added. The flasks were rotated in a thermostat for several days. The iodate content in the saturated solution was determined iodometrically.

Source and Purity of Materials:

A first sample of lead iodate was prepared by a dropwise addition of solutions of 0.05 mol dm^{-3} lead nitrate and 0.1 mol dm^{-3} potassium iodate to 3 dm^3 of water with constant stirring. The precipitate was filtered, washed thoroughly, and air dried at room temperature. A second sample of lead iodate was prepared by the method of La Mer and Goldman (2). Lead iodate was precipitated by simultaneous dropwise addition of equivalent solutions of lead nitrate and potassium iodate in 23 dm^3 jar filled with water under constant stirring at about 60 °C. The supernatant liquid was replaced with fresh water 8 times during the precipitation process, which occupied several days. The potassium nitrate resulting from metathesis did not exceed 0.02 mol dm^{-3} at any time. The air dried lead iodate, when analyzed iodometrically, was 99.9 % pure. The source and purity of alanine and glycine were not reported, but probably chemically pure substances were recrystallized twice from water by addition of ethanol. Distilled water was used.

Estimated Error:

Solubility: precision about ± 1 %.

Temperature: precision ± 0.02 K (authors (1)).

References:

[1] R. M. Keefer, H. G. Reiber, and C. S. Bisson, J. Am. Chem. Soc. **62**, 2951 (1940).

[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791 (1930).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Potassium nitrate; KNO_3; [7757-79-1]
(3) Propanoic acid (propionic acid); $\mathrm{C_3H_6O_2}$; [79-09-4]
(4) Propanoic acid, sodium salt (sodium propionate); $\mathrm{NaC_3H_5O_2}$; [137-40-6]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

R. C. Mohanty and S. Aditya, J. Indian Chem. Soc. **32**, 234–40 (1955).

Variables:

Concentrations of KNO_3, propionic acid and sodium propionate
One temperature: 308 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 142. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of KNO_3, propionic acid and sodium propionate at 35 °C

pH	c_4 / mol dm^{-3}	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$-\log c_1$	$10^4 c_1$ / mol dm^{-3} $^{\rm a}$
4.7	0.01	0.001	0	3.9983	1.004
	0.02	0.002	0	3.8817	1.313
	0.04	0.004	0	3.7486	1.784
	0.06	0.006	0	3.6588	2.194
	0.08	0.008	0	3.5926	2.555
	0.10	0.010	0	3.5377	2.899
	0.12	0.012	0	3.4955	3.195
	0.15	0.015	0	3.4533	3.521
	0.20	0.020	0	3.3573	4.392
5.7	0.01	0.01	0	4.0070	0.984
	0.02	0.02	0	3.8761	1.330
	0.04	0.04	0	3.7573	1.746
	0.06	0.06	0	3.6317	2.335
	0.08	0.08	0	3.6066	2.474
	0.12	0.12	0	3.5105	3.087
	0.15	0.15	0	3.4554	3.504
	0.20	0.20	0	3.3967	4.011
4.7	0	0	0.01	4.1825	0.657
	0	0	0.02	4.1500	0.708
	0	0	0.04	4.1100	0.776
	0	0	0.06	4.0800	0.832
	0	0	0.08	4.0500	0.791
	0	0	0.10	4.0283	0.937
	0	0	0.12	3.9983	1.004
	0	0	0.15	3.9583	1.101

	0	0	0.20	3.9165	1.212
5.7	0	0	0.20	3.9985	1.003[b]

[a] Calculated by the compilers.

[b] Probably erroneous value.

The dissociation constant of $\mathrm{PbC_3H_5O_2^+}$ and $\mathrm{Pb(C_3H_5O_2)_2}$ were determined from the solubility data.

$$\mathrm{Pb(C_3H_5O_2)_2} \rightleftharpoons \mathrm{(PbC_3H_5O_2)^+} + \mathrm{(C_3H_5O_2)^-} \tag{63}$$

$$K_1 = \frac{[\mathrm{(PbC_3H_5O_2)^+}][\mathrm{C_3H_5O_2^-}]}{[\mathrm{Pb(C_3H_5O_2)_2}]} = 3.1 \times 10^{-2} \text{mol dm}^{-3} \tag{64}$$

$$\mathrm{(PbC_3H_5O_2)^+} \rightleftharpoons \mathrm{Pb^{2+}} + \mathrm{C_3H_5O_2^-} \tag{65}$$

$$K_2 = \frac{[\mathrm{Pb^{2+}}][\mathrm{C_3H_5O_2^-}]}{[\mathrm{PbC_3H_5O_2^+}]} = 2.2 \times 10^{-3} \text{mol dm}^{-3} \tag{66}$$

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of lead iodate in aqueous solutions containing sodium propionate and propionic acid was determined at the selected temperature and pH of 4.7 or 5.7 by taking lead iodate in sodium propionate solutions, shaking and equilibrating. The solubility of lead iodate in potassium nitrate solution was determined. The filtered solution was analyzed for the iodate content by iodometric titration with thiosulfate standarized against standard iodate.

Source and Purity of Materials:

Lead iodate was prepared in the way described in (1). Sodium propionate solution was prepared by mixing extra pure propionic acid (Merck) and sodium hydroxide solutions of known concentrations. Source of potassium nitrate was not reported.

Estimated Error:

Nothing specified.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791 (1930).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Propanoic acid, 2-hydroxy- (lactic acid); $\mathrm{C_3H_6O_3}$; [50-21-5]
(3) Propanoic acid, 2-hydroxy-, monosodim salt (sodium lactate); $\mathrm{NaC_3H_5O_3}$; [72-17-3]
(4) Acetic acid, chloro-, sodium salt (sodium monochloroacetate); $\mathrm{NaC_2H_2ClO_2}$; [3926-63-3]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

R. C. Mohanty and S. Aditya, J. Indian Chem. Soc, **32**, 249–51 (1955).

Variables:

Concentration of acid and salts
One temperature: 308 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 143. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of sodium lactate containing lactic acid and of sodium monochloroacetate at 35 °C

c_4 / mol dm^{-3}	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$-\log c_1$	$10^4 c_1$ / mol dm^{-3} [a]
0	0.01	0.0001	4.0267	0.940
0	0.02	0.0002	3.9942	1.013
0	0.04	0.0004	3.7899	1.622
0	0.06[b]	0.0006	3.6896	2.044
0	0.08	0.0008	3.6502	2.238
0	0.10	0.0010	3.5965	2.532
0	0.12	0.0012	3.5831	2.612
0.01	0	0	4.1457	0.715
0.02	0	0	4.0982	0.798
0.04	0	0	4.0266	0.941
0.06	0	0	3.9893	1.025
0.08	0	0	3.8835	1.308
0.10	0	0	3.8145	1.533
0.12	0	0	3.7938	1.608
0.20	0	0	3.6768	2.105

[a] Calculated by the compilers.

[b] The value of 0.03 in the original paper should be read 0.06.

From the solubility data the dissociation constants of $Pb(Lac)_2$ and $PbLac^+$ (Lac = lactate) were reported as follows:

$$\mathrm{Pb(Lac)_2} \rightleftharpoons \mathrm{(PbLac)^+} + \mathrm{(Lac)^-} \tag{67}$$

$$K_1 = \frac{[\mathrm{(PbLac)^+}][\mathrm{(Lac)^-}]}{[\mathrm{Pb(Lac)_2}]} = 4 \times 10^{-2} \text{mol dm}^{-3} \tag{68}$$

$$\mathrm{(PbLac)^+} \rightleftharpoons \mathrm{Pb^{2+}} + \mathrm{(Lac)^-} \tag{69}$$

$$K_2 = \frac{[\mathrm{Pb^{2+}}][\mathrm{(Lac)^-}]}{[\mathrm{(PbLac)^+}]} = 4 \times 10^{-3} \text{mol dm}^{-3} \tag{70}$$

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of lead iodate in solutions containing sodium lactate and lactic acid as well as sodium monochloroacetate was determined at the selected temperature by taking lead iodate in sodium lactate and lactic acid solutions, shaking and equilibrating. The filtered solution was analyzed for the iodate content by iodometric titration with thiosulfate.

Source and Purity of Materials:

Lead iodate was prepared by the method reported in (1). The stock solution of sodium lactate was prepared by mixing equivalent amounts of lactic acid and sodium hydroxide. The stock solution of sodium monochloroacetate was prepared by mixing equivalent amounts of monochloroacetic acid and sodium hydroxide.

Estimated Error:

Nothing specified.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791 (1930).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Perchloric acid; $HClO_4$; [7601-90-3]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Potassium iodate; KIO_3; [7758-05-6]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

R. N. Misra and S. Pani, J. Indian Chem. Soc. **34**, 387–92 (1957).

Variables:

Concentrations of $HClO_4$ (0–0.3 mol dm^{-3}), $NaClO_4$ (0–0.28 mol dm^{-3}) and KIO_3 (7×10^{-3}–1.6×10^{-4} mol dm^{-3})
One temperature: 308 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 144. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of various inorganic compounds at 35 °C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	$10^5 c_4$ / mol dm^{-3}	$10^5 c_1$ / mol dm^{-3}	$10^{12} K_s$ / mol^3 dm^{-9}
0	0	0	5.085	—
0.02	0	0	6.530	—
0.04	0	0	7.495	—
0.06	0	0	8.865	—
0.08	0	0	8.980	—
0.10	0	0	9.590	—
0.12	0	0	10.37	—
0.14	0	0	10.98	—
0.16	0	0	11.50	—
0.18	0	0	12.02	—
0.20	0	0	12.73	—
0.25	0	0	13.95	—
0.30	0	0	15.17	—
0.25	0.05	0	14.23	—
0.20	0.10	0	13.43	—
0.18	0.12	0	12.90	—
0.16	0.14	0	12.64	—
0.14	0.16	0	12.21	—
0.12	0.18	0	11.86	—
0.10	0.20	0	11.51	—
0.08	0.22	0	11.16	—
0.06	0.24	0	10.72	—
0.04	0.26	0	10.37	—
0.02	0.28	0	9.86	—
0	0.30	0	9.860	—

0	0.25	0	9.520	—
0	0.20	0	9.175	—
0	0.18	0	9.005	—
0	0.16	0	8.660	—
0	0.14	0	8.485	—
0	0.12	0	8.230	—
0	0.10	0	8.060	—
0	0.08	0	7.720	—
0	0.06	0	7.205	—
0	0.04	0	6.860	—
0	0.02	0	6.515	—
0.20	0	0	12.73	8.250
0.20	0	6.667	10.87	8.835
0.20	0	8.000	10.47	8.974
0.20	0	9.333	10.14	8.888
0.20	0	10.67	9.555	8.476
0.20	0	12.00	9.315	8.736
0.20	0	13.33	8.910	8.648
0.20	0	16.63	8.115	8.764

In the saturated solution of $\mathrm{Pb(IO_3)_2}$, hydrolysis of Pb^{2+} is appreciable. The hydrolysis was discussed from point of view of the solubility data.

Auxiliary Information

Method/Apparatus/Procedure:

The solubilities of lead iodate from the same sample in various solutions were determined at the selected temperature using probably the method of the predecessors from the same laboratory (1).

Source and Purity of Materials:

As in (2), lead iodate was precipitated by simultaneous dropwise addition of equivalent solutions of chemically pure lead nitrate and potassium iodate in a large amount of water under constant stirring at about 60 °C. The supernatant liquid was replaced with fresh water 8 times during the precipitation process. Other chemicals were of reagent grade quality.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.2 K.

References:

[1] R. C. Mohanty and S. Aditya, J. Indian Chem. Soc, **32**, 234 (1955).

[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **52**, 2791 (1930).

Components:

(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Sodium nitrate; $NaNO_3$; [7631-99-4]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

R. N. Misra and S. Pani, J. Indian Chem. Soc. **34**, 393–8 (1957).

Variables:

Concentration of HNO_3 (0–0.3 mol dm^{-3}) and $NaNO_3$ (0–0.3 mol dm^{-3})
One temperature: 308 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 145. Solubility of $\mathrm{Pb(IO_3)_2}$ in aqueous solutions of HNO_3 and $NaNO_3$ at 35 °C

c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	$10^5 c_1$ / mol dm^{-3}
0.02	0	6.840
0.04	0	7.530
0.06	0	8.210
0.08	0	8.810
0.10	0	9.810
0.12	0	9.840
0.14	0	10.27
0.16	0	10.69
0.18	0	11.21
0.20	0	11.55
0.25	0	12.08
0.30	0	13.35
0.28	0.02	13.35
0.26	0.04	13.44
0.24	0.06	13.69
0.22	0.08	14.38
0.20	0.10	14.80
0.18	0.12	15.48
0.16	0.14	15.92
0.14	0.16	16.60
0.12	0.18	17.11
0.10	0.20	17.79
0.05	0.25	18.99
0	0.30	20.21
0	0.25	17.88
0	0.20	15.79
0	0.18	14.72

0	0.16	13.86
0	0.14	13.00
0	0.12	11.98
0	0.10	10.96
0	0.08	10.180
0	0.06	9.565
0	0.04	8.040
0	0.02	6.845

The formation of nitrato–lead complex and hydrolysis of the lead salt were discussed in the original paper.

Auxiliary Information

Method/Apparatus/Procedure:

No information was given, but the method seems to be the same as previously (1).

Source and Purity of Materials:

All details may be found in the previous paper (1).

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.2 K (authors (1)).

References:

[1] R. N. Misra and S. Pani, J. Indian Chem. Soc. **34**, 387 (1957).

Components:

(1) Lead iodate; $Pb(IO_3)_2$; [25659-31-8]
(2) Acetic acid, hydroxy-, monosodium salt (sodium glycolate); $C_2H_3O_3Na$; [2836-32-0]
(3) Sodium perchlorate; $NaClO_4$; [7601-89-0]
(4) Perchloric acid; $HClO_4$; [7601-90-3]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

A. Basinski and Z. Warnke, Omagiu Raluca Ripan **1966**, 137–42.

Variables:

Concentration of glycolate: 0–1.20 mol dm^{-3}
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 146. Solubility of $Pb(IO_3)_2$ in aqueous solution of sodium glycolate at ionic strength of 3 at 25 °C

c_2 / mol dm^{-3}	$10^4 c_1$ / mol dm^{-3}
0	0.361
0.01	1.78
0.03	2.52
0.05	3.06
0.07	3.33
0.10	4.22
0.20	6.17
0.40	9.55
0.60	12.81
0.70	14.56
0.80	15.97
0.90	17.35
1.00	18.79
1.10	20.38
1.20	21.53

The equilibrium solid phase was found to be in agreement with its theoretical composition within the limits of experimental error.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility measurements were carried out at constant pH of 5.0 ± 0.1 maintained by means of perchloric acid addition. The constant ionic strength was maintained by the addition of a suitable amount of $NaClO_4$. The mixtures containing solid $Pb(IO_3)_2$ in excess were prepared in volumetric flasks. The flasks were placed into a water thermostat equipped with electromagnetic stirrers at the selected temperature. The lead content in the sample solution was determined by the potentiometric titration with thioacetamide (1) in the presence of a silver–sulfide electrode. A pH meter was used for the titration. The first sample was withdrawn after 3 d of steady mixing, and then successive determinations were carried out every 24 h until 3 consistent results were obtained.

Source and Purity of Materials:

Lead iodate was prepared from lead nitrate and potassium iodate. Both reagents (from Polskie Odczynniki Chemiczne) were recrystallized from water. Sodium glycolate was obtained by mixing together the equivalent quantities of glycolic acid and sodium hydroxide. Sodium perchlorate (from Fluka, Switzerland) was recrystallized from water. Perchloric acid (from Xenon, Poland) was diluted and thereafter potentiometrically titrated with a standard NaOH solution. Redistilled water was used.

Estimated Error:

Solubility: precision ± 0.3 %.

Temperature: precision ± 0.1 K.

References:

[1] L. M. Andreasov, E. I. Vail, V. A. Kremer, and V. A. Shelikhovskii, Zh. Anal. Khim. **13**, 657 (1958); J. Anal. Chem. USSR (Engl. Transl.), **13**, 739 (1958).

Components:
(1) Lead iodate; $\mathrm{Pb(IO_3)_2}$; [25659-31-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. A. Piperaki and T. P. Hadjiioannou, Chim. Chron, New Ser. **6**, 375–84 (1977).

Variables:
One temperature: 303 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The compilers assume that the determination was performed at the same temperature as in (1). The solubility of $\mathrm{Pb(IO_3)_2}$ at 30 °C is then:

$$5.0 \times 10^{-5} \text{ mol dm}^{-3}.$$

The authors quoted the solubility value of 4.3×10^{-5} mol dm^{-3} but no reference and temperature was reported.

Auxiliary Information

Method/Apparatus/Procedure:
No information on equilibration of $\mathrm{Pb(IO_3)_2}$ with water and sampling of the saturated solution was reported. A semiautomatic spectrophotometric method was used to analyze the concentration of lead(II) in the sample by the indirect catalytic (periodate-diethylaniline reaction catalyzed by manganese(II)) titrimetric determination. An excess of EDTA standard solution was added to the aliquot of the sample and back titrated with manganese(II) standard solution.

Source and Purity of Materials:
All reagents were of reagent grade purity. Deionized, double distilled water was used.

Estimated Error:
Solubility: accuracy and precision ± (1–2) %.
Temperature: nothing specified.

References:

[1] T. P. Hadjiioannou and T. A. Kephalas, Mikrochim. Acta **6**, 1215 (1969).

Components:

(1) Ammonium iodate; NH_4IO_3; [13446-09-8]
(2) Ammonia; NH_3; [7664-41-7]
(3) Ammonium perchlorate; NH_4ClO_4; [7790-98-9]
or (3) Ammonium chloride; NH_4Cl; [12125-02-9]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

T. V. Stupko, I. D. Isaev, G. L. Pashkov, and V. E. Mironov, Zh. Neorg. Khim. **35**, 3086–9 (1990); Russ. J. Inorg. Chem. (Engl. Transl.) **35**, 1752–4 (1990).

Variables:

Concentration of ammonia: 0–10 mol dm^{-3}
One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

Miyamoto and Salomon (1) have already compiled and critically evaluated six studies of the NH_4IO_3 solubility in the second volume of this series. For completeness, the newly published solubility data are given in Table 147.

The solubility of NH_4IO_3 decreases by adding ammonia to aqueous ammonium perchlorate or chloride solutions at 298.2 K. The data shown in Table 147 have tentative character.

TABLE 147. Solubility of NH_4IO_3 in solutions of ammonium perchlorate or chloride in presence of ammonia at 298.2 K

Added salt	c_3 / mol dm^{-3}	c_2 / mol dm^{-3}	Solubility ratio[a]	10^2c_1 / mol dm^{-3}
NH_4ClO_4	1	0	1	6.37
	1	2.0	0.636	4.05[b]
	1	4.0	0.443	2.82[b]
	1	6.0	0.298	1.90[b]
	1	8.0	0.188	1.20[b]
	1	10.0	0.126	0.80[b]
NH_4Cl	1	0	1	7.5
	1	2.0	0.656	4.9[b]
	1	4.0	0.431	3.2[b]
	1	6.0	0.268	2.0[b]
	1	8.0	0.161	1.4[b]
	1	10.0	0.120	0.9[b]

[a] Solubility ratio is defined as s/s_0; s_0 and s are the solubilities of NH_4IO_3 in 1.0 mol dm^{-3} aqueous solutions of added salt at various ammonia concentrations.

[b] Calculated by the compilers from ratio of the saturated concentrations in presence and absence of ammonia.

The equilibrium solid phases were not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of ammonium iodate in aqueous solutions of both ammonium perchlorate and ammonium chloride varying the ammonia concentration in the solution were determined by mixing an excess of the solid salt with the solution under isothermal conditions until equilibrium was established. The specimens were removed using a pipette with a compact cotton-wool filter. Concentration of iodate ions was determined iodometrically in the solutions saturated with the salt.

Source and Purity of Materials:

No information is given.

Estimated Error:

Solubility: precision ± 0.5–1.0 %.

Temperature: precision ± 0.1 K.

References:

[1] H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 461.

PART 3. COMPILATION AND EVALUATION OF SOLUBILITY DATA OF SCANDIUM, YTTRIUM, LANTHANUM AND LANTHANIDE HALATES, AND ACTINIDE IODATES

VIII. YTTRIUM, LANTHANUM AND LANTHANIDE BROMATES

A. Yttrium Bromate

Components:

(1) Yttrium bromate; $\mathrm{Y(BrO_3)_3}$; [15162-95-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2003.

Critical Evaluation:

The Binary System

The solubility study of $\mathrm{Y(BrO_3)_3}$ in water at 298 K has been appeared in 4 publications (1, 2, 4, 5). The $\mathrm{Y(BrO_3)_3}$ solubility value of James and Langelier (1) was calculated to be 1.85 mol $\mathrm{kg^{-1}}$ in water at 298 K by the evaluators. The compilation of the solubility study by Serebrennikov et al. (2) was published in preceding volume (3) and that of Batyreva et al. (4) is inserted in the $\mathrm{La(BrO_3)_3}$–H_2O system in this volume. Serebrennikov et al. (2), Batyreva et al. (4) and Batyreva et al. (5) reported the solubility of 1.77, 1.80 and 1.92 mol $\mathrm{kg^{-1}}$, respectively. The mean value of the 4 reported values (1, 2, 4, 5) is 1.84 mol $\mathrm{kg^{-1}}$ with the standard deviation of 0.03 mol $\mathrm{kg^{-1}}$ is designated as a tentative solubility at 298 K. The solid phase equilibrated with the saturated solution is $\mathrm{Y(BrO_3)_3 \cdot 9H_2O}$ (2, 4).

The Ternary System

Both Serebrennikov et al. (2) and Batyreva et al. (4) reported a phase diagram of the ternary $\mathrm{Y(BrO_3)_3}$–$\mathrm{KBrO_3}$–H_2O system at 298 K. The detailed information of the solubility study by Serebrennikov et al. (2) can be found in preceding volume (3). Batyreva et al. (5) studied the ternary $\mathrm{La(BrO_3)_3}$–$\mathrm{Y(BrO_3)_3}$–H_2O system at 298K. Two series of solid solutions were found to exist in this system. The smaller branch corresponds to the formation in bottom phase of a solid solution in lanthanum bromate nonahydrate. The larger one corresponds to the formation of a solid solution in yttrium bromate hydrate. This system is of the eutonic type. The composition of the eutonic point is the following: $\mathrm{La(BrO_3)_3}$–37.98 mass %; $\mathrm{Y(BrO_3)_3}$–21.83 mass %. The corresponding compilation can be found in the $\mathrm{La(BrO_3)_3}$–H_2O system section in this volume.

References:

1. C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913 (1909).

2. V. V. Serebrennikov, V. A. Batyreva, and I. S. Larionova, Zh. Neorg. Khim. **27**, 2959 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 1677 (1982).
3. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 240.
4. V. A. Batyreva, I. S. Popova, and G. I. Putii, *Sintez i Reaktsionnaya Sposobnost Veshchestv*, (Tomsk, 1984), 71.
5. V. A. Batyreva, O. V. Nikitina, and O. A. Loskutova, Zh. Neorg. Khim. **45**, 1740 (2000); Russ. J. Inorg. Chem. (Engl. Transl.). **45**, 1600 (2000).

(Erratum: In Solubility Data Series Vol. 30 p. 240, "soly of $\mathrm{Y(BrO_3)_3}$ = 0.50 mol kg^{-1} and soly of $\mathrm{KBrO_3}$ = 1.77 mol kg^{-1}" should be read "soly of $\mathrm{Y(BrO_3)_3}$ = 1.77 mol kg^{-1} and soly of $\mathrm{KBrO_3}$ = 0.50 mol kg^{-1}").

Components:

(1) Yttrium bromate; $Y(BrO_3)_3$; [15162-95-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913–17 (1909).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported that 100 parts of water dissolve 168 parts of $Y(BrO_3)_3 \cdot 9H_2O$. The solubility of 46.7 mass %, 3.23 mol % or 1.85 mol kg^{-1} is calculated by the compiler. Yttrium bromate nonahydrate melts at 74 °C. When the nonahydrate was heated at 100 °C for a long time, it was converted into the trihydrate. The anhydrous salt was obtained by heating to 150 °C.

Auxiliary Information

Method/Apparatus/Procedure:

It seems that the authors used the nonahydrated salt of yttrium bromate in their studies. The equilibration method and a method of the saturated solution analysis were not specified.

Source and Purity of Materials:

The material used in the preparation of yttrium bromate nonahydrate was purified by a modification of the Muthmann and Böhm chromate process. The detailed information of the chromate process was not specified. Yttrium bromate nonahydrate was prepared by the double decomposition of yttrium sulfate with barium bromate. The product was filtered, the filtrate boiled down and allowed to stand, when any remaining barium bromate separated. The refiltered liquid was further concentrated and the yttrium bromate crystallized. The crystals were then drained upon a Hirsch funnel at a filter pump and dried in the air upon filter paper. The analytical method for Br_2O_5 and Y_2O_3 determination were not specified. The analytical results of the product were as follows: found: Br_2O_5–56.74; Y_2O_3–17.85; H_2O (difference)–25.41 mass %; calculated for $Y(BrO_3)_3 \cdot 9H_2O$ (original reported data): Br_2O_5–56.74; Y_2O_3–17.80; H_2O (difference)–25.54 mass %. Because these values calculated by the authors were contained erroneous data these values recalculated by the compiler using 2003 IUPAC recommended atomic weights were as follows: Br_2O_5–56.67; Y_2O_3–17.79; H_2O (difference)–25.54 mass %.

Estimated Error:

Solubility: nothing specified.
Temperature: nothing specified.

Components:

(1) Yttrium bromate; $Y(BrO_3)_3$; [15162-95-5]

(2) Potassium bromate; $KBrO_3$; [7758-01-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Batyreva, I. S. Popova, and G. I. Putii, *Sintez i Reaktsionnaya Sposobnost Veshchestv*, (Tomsk, 1984), 71–3.

Variables:

Composition of the salts

One temperature: 298 K

Prepared By:

Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Compositions of the saturated solutions in $Y(BrO_3)_3$–$K(BrO_3)_3$–H_2O system at 25 °C were presented in a form of hardly readable diagram. The results were read out from the figure and recalculated to mol % by the compilers.

TABLE 148. Composition of saturated solutions in the ternary $Y(BrO_3)_3$–$K(BrO_3)_3$–H_2O system at 25 °C

$Y(BrO_3)_3$		$KBrO_3$		
$100w_1$	$100x_1$	$100w_2$	$100x_2$	Solid phase[a]
46.0[b]	3.1	0	0	A
44.0	3.0	1.9	0.4	A+B
41.5	2.8	3.2	0.6	B
37.5	2.4	4.0	0.7	B
23.5	1.2	6.2	0.9	B
3.0	0.1	7.4	0.9	B
0	0	7.1	0.8	B

[a] The solid phases are: A = $Y(BrO_3)_3$, B = $KBrO_3$.

[b] For the binary system the compilers compute the solubility of $Y(BrO_3)_3$ = 1.80 mol kg^{-1}.

The eutonic point (reported numerically) was observed at 48.4 mass % $\mathrm{Y(BrO_3)_3}$, 0.9 mass % $KBrO_3$ and 50.6 mass % H_2O (3.5, 0.2 and 96.3 mol %, respectively, as recalculated by the compilers). Refraction index and specific gravity of the saturated solutions (both presented in a figure) confirmed the shape of the liquidus line and the existence of the eutonic point. The equilibrium solid phase crystallizing in the vicinity of the eutonic point is a mixture of $\mathrm{Y(BrO_3)_3}$ hydrate and $KBrO_3$ crystals.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal solubility method was used. Equilibrium in the system was reached after 7 d of continuous stirring with a glass bullet. Potassium content was determined by flame photometry and yttrium content was found from complexometric titration with EDTA. The refraction index and specific gravity of the saturated solutions were determined by a refractometer and pycnometer, respectively. Compositions of the solid phases were determined by the Schreinemakers' method and confirmed by X-ray analysis.

Source and Purity of Materials:

Nothing specified.

Estimated Error:

Solubility: nothing specified; reading-out procedure ± 0.3 mass %.
Temperature: nothing specified.

Components:
(1) Lanthanum bromate; $\mathrm{La(BrO_3)_3}$; [28958-23-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. August 2003.

Critical Evaluation:

The Binary System

Data for the solubility in the binary $\mathrm{La(BrO_3)_3}$–H_2O system have been reported in 4 publications (1–4). James and Langelier (1) reported the solubility value of 5.22 mol % or 3.06 mol kg^{-1} at 298 K. In this report the authors quoted an earlier obtained value of 4.78 mol % (2.79 mol kg^{-1}, calculated by the evaluators) by Marignac (5) at 288 K. The latter information seems to be a private communication and no experimental details are available. The value of Marignac (5) is the lowest one in a comparison with the values obtained by the following investigators. James et al. (2) measured the solubility of lanthanum bromate in water over the temperature range of 273–308 K. Serebrennikov et al. (3) as well as Batyreva et al. (4) studied the solubility at 298 K. Experimental data of lanthanum bromate in water at 298 K is summarized in Table 149.

The equilibrium solid phase is $\mathrm{La(BrO_3)_3 \cdot 9H_2O}$ (1–4).

TABLE 149. Experimental solubility data of $\mathrm{La(BrO_3)_3}$ in water at 298 K

$100w_1$	$100x_1$	m_1 / mol kg^{-1}	Analytical method	Reference
61.5	5.22	3.06	No information	(1)
62.74	5.486	3.22	Gravimetry (La^{3+})	(2)
59.49	4.811	2.81	Complexometric titration (La^{3+})	(3)
59.50	4.820	2.81	Complexometric titration (La^{3+})	(4)

The reported solubilities are spread, but the reason of the differences is not clear. The evaluators assumed that the solubility values at 298 K in (3) and (4) were measured by same investigator's group. The agreement between the results by James et al. (2) and those in (3) and (4) fails. Remeasurement of the solubility of lanthanum bromate in water at this temperature is needed to decide on a recommended solubility. Reluctantly, the arithmetic mean of the 4 reported values was calculated, and the value of 2.98 mol kg^{-1} is obtained. The standard deviation is ± 0.10 mol kg^{-1}. The calculated value is designated as a doubtful solubility.

The temperature dependence of the solubility reported by James et al. (2) with James and Langelier (1), Serebrennikov et al. (3) and Batyreva et al. (4) is shown in the Fig. 13. The $\mathrm{La(BrO_3)_3}$ solubilities reported by James et al. (2) were used to obtain the smoothing equation. The detailed information of the calculation procedure for the smoothing equation was described in the Preface in this volume. The obtained equation is

$$Y_x = \frac{-182828.431}{(T/\mathrm{K})} - 1308.14213\ln(T/\mathrm{K}) + 7351.16403 + 2.40181438(T/\mathrm{K}), \tag{71}$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \tag{72}$$

$\sigma_Y = 0.028$ and $\sigma_x = 0.00066$.

The solubilities calculated from the fitting equation are given in Table 150. These values, calculated from the smoothing equation, are designated as doubtful solubilities.

TABLE 150. Doubtful solubility of $\mathrm{La(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	3.27	1.88
278	3.60	2.07
283	3.97	2.30
288	4.40	2.55
293	4.91	2.86
298	5.53	3.25
303	6.33	3.75
308	7.41	4.45

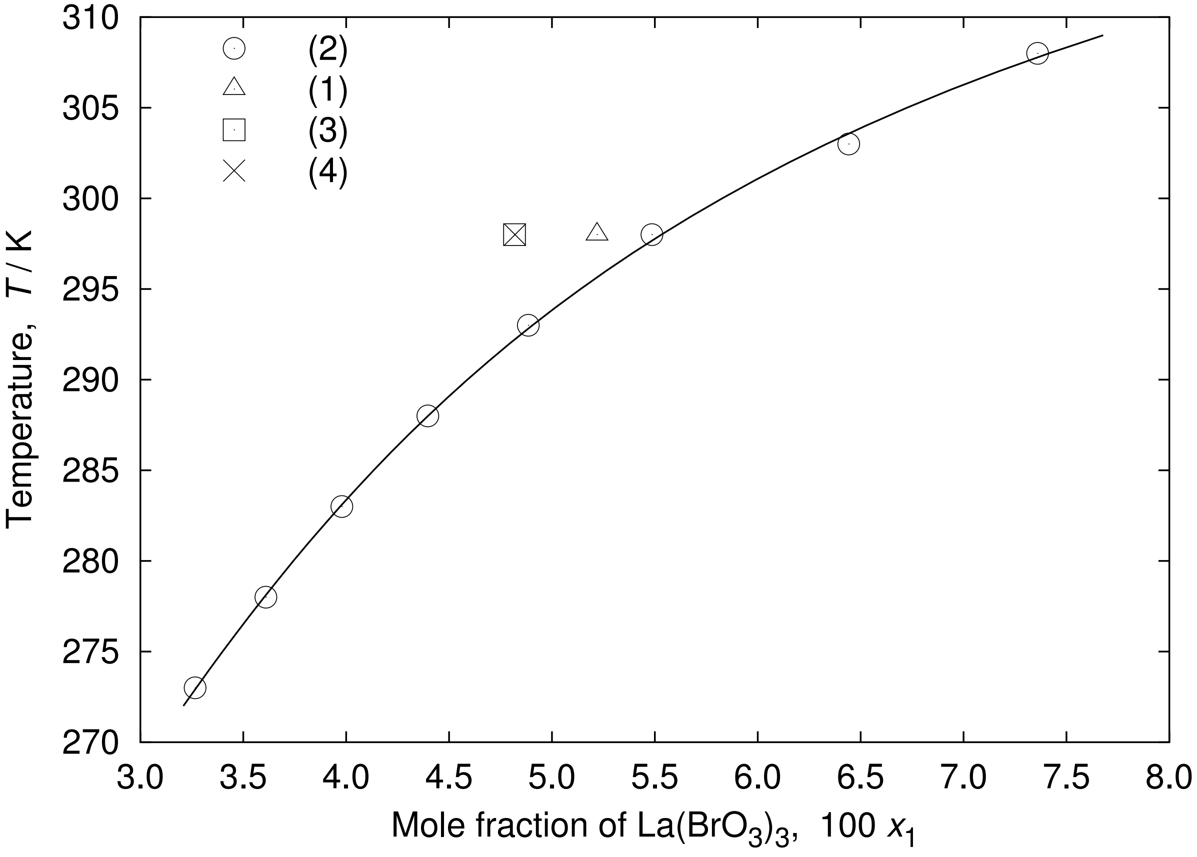

FIG. 13. Temperature dependence of solubility of $La(BrO_3)_3$ in water

The Ternary System

Serebrennikov et al. (3) reported the solubilities in the ternary $La(BrO_3)_3$–$Pr(BrO_3)_3$–H_2O system. In this system, a continuous series of solid solutions is formed. Batyreva et al. (4) studied two ternary systems, $La(BrO_3)_3$–$Y(BrO_3)_3$–H_2O and $La(BrO_3)_3$–$Yb(BrO_3)_3$–H_2O, by the isothermal method. Two series of solid solutions were found to exist in these systems. The systems belong to Type IV in the Roozeboom's classification.

References:

1. C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913 (1909).

2. C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132 (1927).

3. V. V. Serebrennikov, I. L. But, and V. A. Batyreva, Zh. Neorg. Khim. **29**, 2701 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 1548 (1984).

4. V. A. Batyreva, O. V. Nikitina, and O. A. Loskutova, Zh. Neorg. Khim. **45**, 1740 (2000); Russ. J. Inorg. Chem. (Engl. Transl.) **45**, 1600 (2000).

5. Marignac, as quoted in (1).

Components:

(1) Lanthanum bromate; $La(BrO_3)_3$; [28958-23-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James and W. F. Langelier, J. Am. Chem Soc. **31**, 913–17 (1909).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported that 100 parts of water dissolve 416 parts of $La(BrO_3)_3 \cdot 9H_2O$ at 25 °C. The solubility of 61.5 mass %, 5.22 mol % or 3.06 mol kg^{-1} is calculated by the compiler. When dried at 100 °C, this salt transforms into $La(BrO_3)_3 \cdot 2H_2O$; the anhydrous salt is formed at 150 °C. It decomposes on further heating to a higher temperature.

Auxiliary Information

Method/Apparatus/Procedure:

It seems that the authors used the nonahydrate salt of lanthanum bromate for the solubility determination. A method for the solubility determination was not specified.

Source and Purity of Materials:

Lanthanum bromate nonahydrate was prepared by the double decomposition of lanthanum sulfate with barium bromate. The product was filtered, the filtrate boiled down and allowed to stand, when any remaining barium bromate was separated. The refiltered liquid was further concentrated and the lanthanum bromate was crystallized. The crystals were then drained upon a Hirsch funnel at a filter pump and dried in the air upon filter paper. The analytical results of the product were as follows: found: Br_2O_5–52.83; La_2O_3–23.95; H_2O (difference)–23.22 mass %; calculated for $La(BrO_3)_3 \cdot 9H_2O$ formula: Br_2O_5–52.53; La_2O_3–23.80; H_2O (difference)–23.67 mass %. The analytical methods of both Br_2O_5 and La_2O_3 determinations were not specified.

Estimated Error:

Solubility: nothing specified.
Temperature: nothing specified.

Components:

(1) Lanthanum bromate; $La(BrO_3)_3$; [28958-23-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132–5 (1927).

Variables:

Temperature: 273–308 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 151. Solubility of $La(BrO_3)_3$ in water at various temperatures

	$La(BrO_3)_3 \cdot 9H_2O$		$La(BrO_3)_3$	
t / °C	$100w_B$ [a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	64.83	184.6	49.48	3.266
5	68.22	214.7	52.06	3.610
10	71.53	251.3	54.59	3.979
15	74.89	298.4	57.02	4.397
20	78.40	363.0	59.83	4.885
25	82.21	462.1	62.74	5.486
30	87.31	688.6	66.63	6.442
35	91.38	1061.5	69.74	7.360

[a] B (elementary entities) in w_B is $La(BrO_3)_3 \cdot 9H_2O$.

[b] Mass ratio is reported as the value of parts of $La(BrO_3)_3 \cdot 9H_2O$ per 100 parts of H_2O in the original paper.

[c] Cited in the Linke's book (1) and confirmed by the compilers.

[d] Calculated by the compilers from the mass % solubility of $La(BrO_3)_3$.

Equilibrium solid was the lanthanum bromate nonahydrate.

Auxiliary Information

Method/Apparatus/Procedure:

Lanthanum bromate nonahydrate was used. Equilibrium was approached from both supersaturation and undersaturation. It seems that the authors used a method for La determination described in their preceding paper (2). Bottles with solutions were rotated in a thermostat for more than 9 h. Samples of saturated solution were withdrawn, weighed, diluted and precipitated with oxalic acid solution. The mixture was filtered after 12 h, the precipitate was washed, ignited to oxide and weighed. All results were mean values of duplicate analyses.

Source and Purity of Materials:

Lanthanum bromate was prepared by stirring the pure lanthanum sulfate with an excess of pure barium bromate suspended in water.

Estimated Error:

Solubility: precision within 1 %, but at higher temperatures within 1.5 %.
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 343.
[2] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Lanthanum bromate; $La(BrO_3)_3$; [28958-23-8]
(2) Praseodymium bromate; $Pr(BrO_3)_3$; [15162-93-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. V. Serebrennikov, I. L. But, and V. A. Batyreva, Zh. Neorg. Khim. **29**, 2701–2 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 1548–9 (1984).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 152. Composition of saturated solutions in the ternary $La(BrO_3)_3$–$Pr(BrO_3)_3$–H_2O system at 25 °C

$La(BrO_3)_3$		$Pr(BrO_3)_3$		
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	Solid phase[b]
59.49[c]	4.811	0	0	A
51.99	4.007	5.26	0.404	C
45.54	3.329	9.19	0.669	C
43.86	3.335	12.75	0.966	C
40.49	2.957	14.19	1.032	C
38.48	2.806	16.12	1.171	C
36.20	3.695	19.42	1.441	C
26.80	1.922	26.98	1.927	C
25.04	1.741	27.17	1.882	C
19.29	1.309	31.67	2.141	C
14.16	0.949	36.13	2.412	C
13.65	0.926	37.28	2.519	C
7.86	0.516	41.32	2.802	C
0	0	47.02[c]	2.958	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $La(BrO_3)_3 \cdot 9H_2O$; B = $Pr(BrO_3)_3 \cdot 9H_2O$; C = solid solution of both salts with molar ratio similar to that in the solution.

[c] For binary systems the compilers compute the following values:

$$\text{Solubility of } La(BrO_3)_3 = 2.81 \text{ mol kg}^{-1},$$
$$\text{Solubility of } Pr(BrO_3)_3 = 1.69 \text{ mol kg}^{-1}.$$

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal method was used. Equilibrium was reached after 7 d of continuous mixing. The total concentration of lanthanum and praseodymium was determined by complexometric titration with Trilon. The praseodymium content was then determined by spectrophotometric method. The composition of the solid phases was found by the Schreinemakers' method of residues.

Source and Purity of Materials:

No information given.

Estimated Error:

Solubility: precision no better than ± 0.5 % (compilers).

Temperature: nothing specified.

Components:

(1) Lanthanum bromate; $\mathrm{La(BrO_3)_3}$; [28958-23-8]
(2) Yttrium bromate; $\mathrm{Y(BrO_3)_3}$; [15162-95-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Batyreva, O. V. Nikitina, and O. A. Loskutova, Zh. Neorg. Khim. **45**, 1740–3 (2000); Russ. J. Inorg. Chem. (Engl. Transl.) **45**, 1600–3 (2000).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 153. Composition of saturated solutions in the ternary $\mathrm{La(BrO_3)_3}$–$\mathrm{Y(BrO_3)_3}$–H_2O system at 298 K

$\mathrm{La(BrO_3)_3}$		$\mathrm{Y(BrO_3)_3}$		Solid phase[b]
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	
59.50[c]	4.820	0	0	A
56.41	4.551	2.92	0.261	B
50.92	3.966	6.84	0.589	B
44.15	3.365	12.62	1.064	B
33.82	2.512	21.76	1.787	B+C
33.78	2.524	22.10	1.826	B+C
34.12	2.558	21.91	1.816	B+C
34.35	2.573	21.63	1.791	B+C
27.24	1.888	24.92	1.910	C
26.03	1.878	28.16	2.247	C
23.46	1.727	31.72	2.582	C
20.11	1.393	32.04	2.454	C
17.34	1.192	34.44	2.619	C
14.13	0.964	37.24	2.809	C
11.52	0.740	36.64	2.602	C
9.86	0.653	39.92	2.922	C
8.24	0.589	45.52	3.595	C
5.31	0.351	44.35	3.238	C
2.27	0.148	46.57	3.348	C
0	0	47.60[c]	3.346	D

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{La(BrO_3)_3 \cdot 9H_2O}$; B = solid solution $\mathrm{(Y, La)(BrO_3)_3 \cdot 9H_2O}$ based on the La salt; C = solid solution $\mathrm{(La, Y)(BrO_3)_3 \cdot 9H_2O}$ based on the Y salt; D = $\mathrm{Y(BrO_3)_3 \cdot 9H_2O}$.

[c] For the binary systems the compilers compute the following values:

Solubility of $\mathrm{Y(BrO_3)_3}$ = 1.92 mol $\mathrm{kg^{-1}}$,
Solubility of $\mathrm{La(BrO_3)_3}$ = 2.81 mol $\mathrm{kg^{-1}}$.

The composition of the eutonic point is: $\mathrm{La(BrO_3)_3}$, 37.98 mass % and $\mathrm{Y(BrO_3)_3}$, 21.83 mass %.

Auxiliary Information

Method/Apparatus/Procedure:

Equilibrium in the system was attained by continuous stirring the bottom phase and the saturated solution with a glass rod for 7 d. Component contents in equilibrium liquid phase and solid residues were determined by chelatometric titration and emission spectroscopy for lanthanum and yttrium. The compositions of solid phase were determined by the Schreinemakers' method of residues. The solid phases were identified crystal-optical analysis and X-ray powder diffraction.

Source and Purity of Materials:

Lanthanum and yttrium bromate nonahydrates were synthesized by dissolving their oxides in freshly prepared bromic acid.

Estimated Error:

Solubility: not stated, but precision ± 0.3 % (compilers assumed).
Temperature: accuracy ± 0.5 K.

Components:

(1) Lanthanum bromate; $La(BrO_3)_3$; [28958-23-8]

(2) Ytterbium bromate; $Yb(BrO_3)_3$; [28972-23-8]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Batyreva, O. V. Nikitina, and O. A. Loskutova, Zh. Neorg. Khim. **45**, 1740–3 (2000); Russ. J. Inorg. Chem. (Engl. Transl.) **45**, 1600–3 (2000).

Variables:

Composition of the salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 154. Composition of saturated solutions in the $La(BrO_3)_3$–$Yb(BrO_3)_3$–H_2O system at 298 K

$La(BrO_3)_3$		$Yb(BrO_3)_3$		
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	Solid phase[b]
59.50[c]	4.820	0	0	A
53.25	4.210	5.15	0.382	B
48.12	3.661	8.51	0.608	B
37.05	2.651	16.59	1.114	B
35.02	2.618	20.75	1.456	B
32.52	2.345	21.50	1.456	B+C
32.50	2.392	22.51	1.555	B+C
32.50	2.297	20.50	1.360	B+C
28.11	1.991	24.99	1.661	C
25.45	1.797	27.51	1.824	C
20.89	1.452	31.25	2.039	C
14.32	0.944	35.04	2.169	C
10.11	0.671	39.60	2.468	C
1.75	0.113	46.26	2.795	C
0	0	47.50[c]	2.844	D

[a] Calculated by the compilers.

[b] The solid phases are: A = $La(BrO_3)_3 \cdot 9H_2O$; B = solid solution $(Yb, La)(BrO_3)_3 \cdot 9H_2O$; C = solid solution $(La, Yb)(BrO_3)_3 \cdot 9H_2O$; D = $Yb(BrO_3)_3 \cdot 9H_2O$.

[c] For binary systems the compilers compute the following:

$$\text{Solubility of } La(BrO_3)_3 = 2.81 \text{ mol kg}^{-1},$$
$$\text{Solubility of } Yb(BrO_3)_3 = 1.63 \text{ mol kg}^{-1}.$$

The composition of the eutonic point was found to be 32.51 mass % of $\mathrm{La(BrO_3)_3}$ and 21.75 mass % of $\mathrm{Yb(BrO_3)_3}$.

Auxiliary Information

Method/Apparatus/Procedure:

Equilibrium in the system was attained by continuously stirring the bottom phase with the saturated solution with a glass rod for 7 d. Component contents in the equilibrium liquid phases and solid residues were determined by chelatometric titration and differential spectroscopy for ytterbium and emission spectroscopy for lanthanum. The compositions of solid phase were determined by the Schreinemakers' method of residues. The solid phases were identified crystal-optical analysis and X-ray powder diffraction.

Source and Purity of Materials:

The starting reagents were nonahydrates which were synthesized by dissolving the appropriate rare earth element oxides in freshly prepared bromic acid.

Estimated Error:

Solubility: not stated, but precision ± 0.3 % (compilers assumed)

Temperature: accuracy ± 0.5 K.

Components:
(1) Praseodymium bromate; $\mathrm{Pr(BrO_3)_3}$; [15162-93-3]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. August 2003.

Critical Evaluation:

The Binary System

Solubility data for the binary $\mathrm{Pr(BrO_3)_3}$–H_2O system have been reported in 5 publications (1–5). James and Langelier (1) reported the solubility to be 3.32 mol % or 1.91 mol kg^{-1} at 298 K. James et al. (2) measured the solubility of praseodymium bromate in pure water over temperature range of 273–318 K. Staveley (3) and Serebrennikov et al. (4, 5) studied the solubility at 298 K. They have reported that the solid phase equilibrated with the saturated solution is $\mathrm{Pr(BrO_3)_3 \cdot 9H_2O}$.

The compilation of the solubility study by Serebrennikov et al. (5) is inserted in the $\mathrm{La(BrO_3)_3}$–H_2O system in this volume. All experimental solubilities of praseodymium bromate in water at 298 K are given in Table 155.

TABLE 155. Experimental solubility data of $\mathrm{Pr(BrO_3)_3}$ in water at 298 K

$100w_1$	$100x_1$	m_1 / mol kg^{-1}	Analytical method	Reference
50.0	3.33	1.91	No information	(1)
50.59	3.397	1.95	Gravimetry ($\mathrm{Pr^{3+}}$)	(2)
50.0	3.33	1.91	Iodometry ($\mathrm{BrO_3^-}$)	(3)
48.00	3.072	1.76	No information, but probably the same as in (5)	(4)
47.02	2.958	1.69	Spectrophotometry ($\mathrm{Pr^{3+}}$)	(5)

The reported solubilities are spread, but a reason of the differences is not clear. Reluctantly, the arithmetic mean of the 5 reported values was calculated, and the solubility of 1.84 mol kg^{-1} is obtained. The standard deviation is ± 0.05 mol kg^{-1}. The calculated value may be treated as tentative.

The temperature dependence of the solubility reported by James et al. (2) with James and Langelier (1), Staveley (3) and Serebrennikov et al. (4, 5) is shown in the Fig. 14. The $\mathrm{Pr(BrO_3)_3}$ solubilities reported by James et al. (2) were used to obtain the smoothing equation. The detailed information of the calculation procedure for the smoothing equation was described in the Preface in this volume. The obtained equation is

$$Y_x = \frac{-262176.469}{(T/\mathrm{K})} - 1727.83842 \ln{(T/\mathrm{K})} + 9854.3635 + 2.91402161(T/\mathrm{K}), \tag{73}$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \tag{74}$$

$\sigma_Y = 0.0089$ and $\sigma_x = 8.1 \times 10^{-5}$.

The solubilities calculated from the fitting equation are given in Table 156. The values calculated from the smoothing equation are designated as doubtful solubilities.

TABLE 156. Doubtful solubility of $\mathrm{Pr(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	1.89	1.07
278	2.16	1.23
283	2.45	1.39
288	2.75	1.57
293	3.06	1.75
298	3.40	1.95
303	3.78	2.18
308	4.21	2.44
313	4.71	2.75

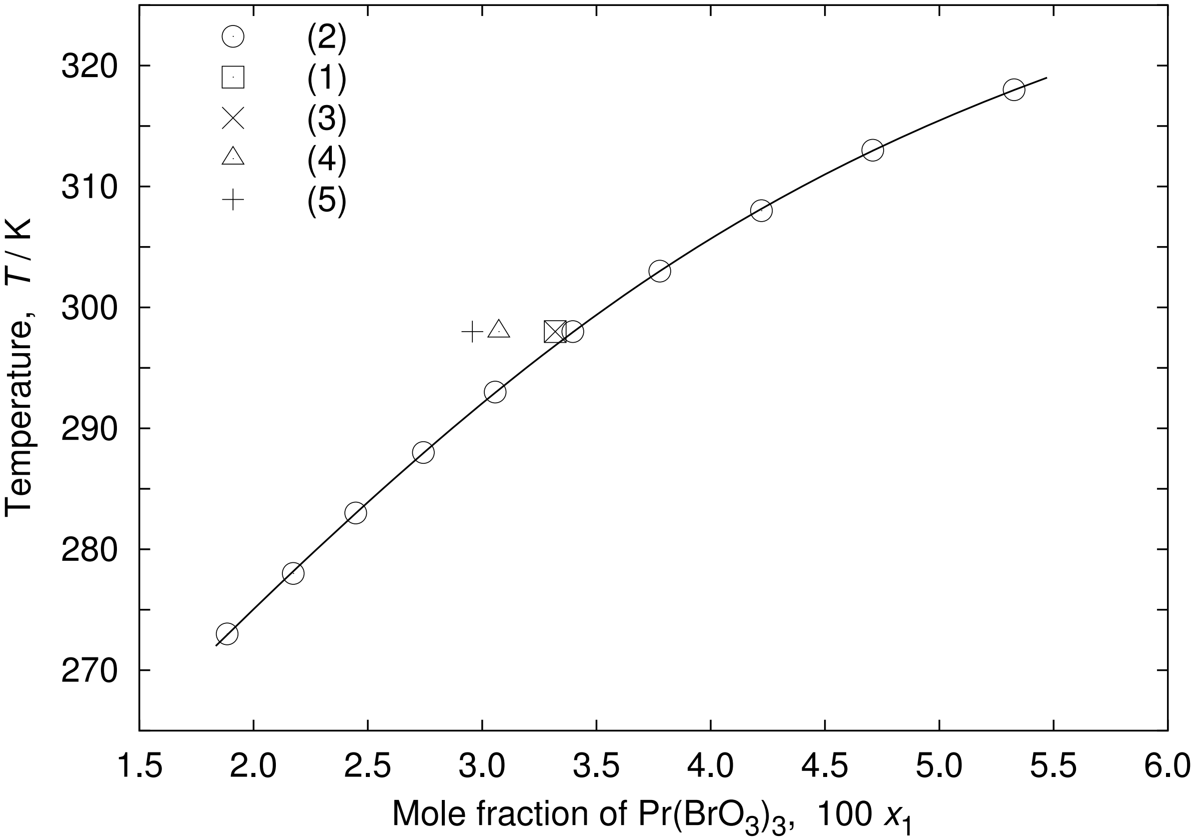

FIG. 14. Temperature dependence of solubility of $\mathrm{Pr(BrO_3)_3}$ in water

The Ternary System

Two ternary systems, $\mathrm{Pr(BrO_3)_3}$–$\mathrm{Nd(BrO_3)_3}$–H_2O (4) and $\mathrm{Pr(BrO_3)_3}$–$\mathrm{La(BrO_3)_3}$–H_2O (5) were studied by groups of Serebrennikov (4, 5). In both systems, a continuous series of solid solutions is formed.

References:

1. C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913 (1909).
2. C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem. Soc. **49**, 132 (1927).
3. L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231 (1968).
4. V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **23**, 1947 (1978); Russ. J. Inorg. Chem. (Engl. Transl.) **23**, 1070 (1978).
5. V. V. Serebrennikov, I. L. But, and V. A. Batyreva, Zh. Neorg. Khim. **29**, 2701 (1984); Russ. J. Inorg. Chem. (Engl. Transl.) **29**, 1548 (1984).

Components:

(1) Praseodymium bromate; $Pr(BrO_3)_3$; [15162-93-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913–17 (1909).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported that 100 parts of water dissolve 190 parts of $Pr(BrO_3)_3 \cdot 9H_2O$ at 25 °C. The solubility of 50.0 mass %, 3.32 mol % or 1.91 mol kg^{-1} is calculated by the compiler. This salt transforms into $Pr(BrO_3)_3 \cdot 2H_2O$ when dried at 100 °C. The anhydrous salt was decomposed with evolution of heat and light by heating to 150 °C, while it can exist at 130 °C.

Auxiliary Information

Method/Apparatus/Procedure:

It seems that the authors used $Pr(BrO_3)_3 \cdot 9H_2O$ for the solubility determination. The equilibration method and a method of the saturated solution analysis were not described.

Source and Purity of Materials:

$Pr(BrO_3)_3 \cdot 9H_2O$ was prepared by the double decomposition of praseodymium sulfate with barium bromate. The product was filtered, the filtrate was boiled down and allowed to stand, when any remaining barium bromate was separated. The refiltered liquid was further concentrated and the praseodymium bromate was crystallized. The crystals were then drained upon a Hirsch funnel at a filter pump and dried in the air upon filter paper. The Pr_2O_3 was estimated by titrating the oxalate with potassium permanganate. The analytical results of the product were as follows: found: Br_2O_5–52.55; Pr_2O_3–24.00; H_2O (difference)–23.45 mass %; calculated for $Pr(BrO_3)_3 \cdot 9H_2O$ (original reported data): Br_2O_5–52.45; Pr_2O_3–23.97; H_2O (difference)–23.61 mass %. The analytical methods of both Br_2O_5 and Pr_2O_3 determination were not specified. Because these values calculated by the authors were contained erroneous data the values recalculated by the compiler using 2003 IUPAC recommended atomic weights were as follows: Br_2O_5–52.38; Pr_2O_3–24.01; H_2O (difference)–23.61 mass %.

Estimated Error:

Solubility: nothing specified.
Temperature: nothing specified.

Components:

(1) Praseodymium bromate; $\mathrm{Pr(BrO_3)_3}$; [15162-93-3]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem. Soc. **49**, 132–5 (1927).

Variables:

Temperature: 273–318 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 157. Solubility of $\mathrm{Pr(BrO_3)_3}$ in water at various temperatures

	$\mathrm{Pr(BrO_3)_3 \cdot 9H_2O}$		$\mathrm{Pr(BrO_3)_3}$	
t / °C	$100w_\mathrm{B}$ [a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	46.96	88.55	35.87	1.885
5	51.42	105.86	39.28	2.173
10	55.25	123.46	42.20	2.447
15	59.03	144.1	45.09	2.743
20	62.66	167.9	47.87	3.057
25	66.23	196.1	50.59	3.397
30	69.83	235.5	53.34	3.777
35	73.58	278.5	56.21	4.222
40	77.23	339.3	59.00	4.709
45	81.29	434.5	62.10	5.327

[a] B (elementary entities) in w_B is $\mathrm{Pr(BrO_3)_3 \cdot 9H_2O}$.

[b] Mass ratio is reported as the value of parts of $\mathrm{Pr(BrO_3)_3 \cdot 9H_2O}$ per 100 parts of H_2O in the original paper.

[c] Cited in the Linke's book (1) and confirmed by the compilers.

[d] Calculated by the compilers from the mass % solubility of $\mathrm{Pr(BrO_3)_3}$.

Auxiliary Information

Method/Apparatus/Procedure:

Praseodymium bromate nonahydrate was used. The equilibrium was approached from both supersaturation and undersaturation. It seems that the authors used the method for Pr determination as it was described in their previous paper (2). Bottles with solutions were rotated in a thermostat for more than 9 h. Samples of the saturated solution were withdrawn, weighed, diluted, precipitated with oxalic acid solution. The mixture was filtered after 12 h and the precipitate was washed, ignited to oxide and weighed. All results are mean of duplicate analyses.

Source and Purity of Materials:

Praseodymium was obtained by the fractionation of praseodymium-neodymium bromate very rich in praseodymium. The method was found to be superior.

Estimated Error:

Solubility: precision within 1 %, but at higher temperatures within 1.5 %.
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1339.
[2] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Praseodymium bromate; $\mathrm{Pr(BrO_3)_3}$; [15162-93-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

L. A. K. Staveley, D. R. Markham and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Pr(BrO_3)_3}$ in water at 25 °C is 1.91 mol kg^{-1}. Composition of equilibrium solid was not specified but may be assumed to be nonahydrate.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $\mathrm{Pr(BrO_3)_3}$ is 6.35 J.

Auxiliary Information

Method/Apparatus/Procedure:

A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, prepared by an unspecified way, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of $\mathrm{Na_2S_2O_3}$.

Source and Purity of Materials:

The bromate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrated bromate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

Components:

(1) Praseodymium bromate; $\mathrm{Pr(BrO_3)_3}$; [15162-93-3]

(2) Neodymium bromate; $\mathrm{Nd(BrO_3)_3}$; [15162-92-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **23**, 1947–51 (1978); Russ. J. Inorg. Chem. (Engl. Transl.) **23**, 1070–2 (1978).

Variables:

Composition of two salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 158. Composition of saturated solutions in the ternary $\mathrm{Pr(BrO_3)_3}$–$\mathrm{Nd(BrO_3)_3}$–H_2O system at 25 °C

$\mathrm{Pr(BrO_3)_3}$		$\mathrm{Nd(BrO_3)_3}$		
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	Solid phase[b]
48.00[c]	3.072	0	0	A
36.50	2.257	9.55	0.587	A+B
32.60	2.153	17.10	1.122	A+B
31.00	2.110	20.31	1.374	A+B
28.80	1.926	21.58	1.434	A+B
25.90	1.646	21.70	1.370	A+B
19.90	1.217	25.50	1.550	A+B
19.70	1.301	30.00	1.969	A+B
12.80	0.690	24.80	1.329	A+B
16.90	1.070	30.50	1.919	A+B
15.00	0.955	32.70	2.069	A+B
10.10	0.586	32.10	1.849	A+B
10.60	0.685	37.90	2.433	A+B
7.50	0.465	38.70	2.385	A+B
7.10	0.444	39.60	2.462	A+B
6.20	0.399	42.10	2.693	A+B
4.97	0.310	41.65	2.585	A+B
0	0	43.28[c]	2.538	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{Pr(BrO_3)_3 \cdot 9H_2O}$; A+B = solid solution of crystalline nonahydrate of $\mathrm{Pr(BrO_3)_3}$ and $\mathrm{Nd(BrO_3)_3}$; B = $\mathrm{Nd(BrO_3)_3 \cdot 9H_2O}$.

[c] For binary systems the compilers compute the following values:

$$\text{Solubility of } \mathrm{Pr(BrO_3)_3} = 1.76 \text{ mol kg}^{-1},$$
$$\text{Solubility of } \mathrm{Nd(BrO_3)_3} = 1.45 \text{ mol kg}^{-1}.$$

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal solubility method was used. Equilibrium in the systems was reached after 7 d of continuous stirring. No information of analytical method for the liquid phase was reported. The compositions of the solid phases were determined by the Schreinemakers' method.

Source and Purity of Materials:

The bromate nonahydrates were made by dissolving "chemically pure" grade oxides in freshly prepared bromic acid; these oxides were prepared from the twice recrystallized hydroxides.

Estimated Error:

Solubility: precision no better than ± 0.5 % (compilers).
Temperature: nothing specified.

Components:
(1) Neodymium bromate; $\mathrm{Nd(BrO_3)_3}$; [15162-92-2]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2003.

Critical Evaluation:

The Binary System

Solubilities of neodymium bromate in pure water have been reported in 5 publications (1–5). James and Langelier (1) reported the solubility of 2.76 mol % or 1.58 mol kg^{-1} at 298 K. James et al. (2) measured the solubilities of neodymium bromate in pure water over temperature range of 273–318 K. The solubility increases monotonically with increasing the temperature. Serebrennikov et al. (3, 4) and Batyreva et al. (5) studied the solubility at 298 K. They have reported that the solid phase equilibrated with the saturated solution is $\mathrm{Nd(BrO_3)_3 \cdot 9H_2O}$.

The compilation of the solubility study by Serebrennikov et al. (3) is inserted in the $\mathrm{Pr(BrO_3)_3}$–H_2O system in this volume. All experimental solubilities of neodymium bromate in water at 298 K are given in Table 159.

TABLE 159. Experimental solubility data of $\mathrm{Nd(BrO_3)_3}$ in water at 298 K

$100w_1$	$100x_1$	m_1 / mol kg^{-1}	Analytical method	Reference
45.4	2.76	1.58	No information	(1)
46.06	2.831	1.62	Gravimetry (Nd^{3+})	(2)
43.28	2.538	1.45	No information	(3)
43.26	2.536	1.44	No information	(4)
43.0	2.5	1.4	Complexometric titration (Nd^{3+})	(5)

The reported solubilities are spread, but the reason of the differences is not clear. Reluctantly, the arithmetic mean of the 5 reported values was calculated, and the solubility of 1.5 mol kg^{-1} is obtained. The standard deviation is 0.04 mol kg^{-1}, and the value may be treated as tentative.

The temperature dependence of the solubility reported by James et al. (2) with James and Langelier (1), Serebrennikov et al. (3, 4), and Batyreva et al. (5) is shown in the Fig. 15. The $\mathrm{Nd(BrO_3)_3}$ solubility reported by James et al. (2) was used to obtain the smoothing equation. The detailed information of the calculation procedure for the smoothing equation has been described in the Preface in this volume. The obtained equation is

$$Y_x = \frac{-247190.853}{(T/\mathrm{K})} - 1579.43209 \ln{(T/\mathrm{K})} + 9054.83791 + 2.58896959(T/\mathrm{K}), \tag{75}$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \tag{76}$$

$\sigma_Y = 0.022$ and $\sigma_x = 0.00024$.

The solubilities calculated from the fitting equation are given in Table 160. These values calculated from the smoothing equation are designated as doubtful solubilities.

TABLE 160. Doubtful solubility of $\mathrm{Nd(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	1.48	0.83
278	1.72	0.97
283	1.98	1.12
288	2.25	1.28
293	2.53	1.44
298	2.82	1.61
303	3.14	1.80
308	3.48	2.00
313	3.85	2.23
318	4.29	2.49

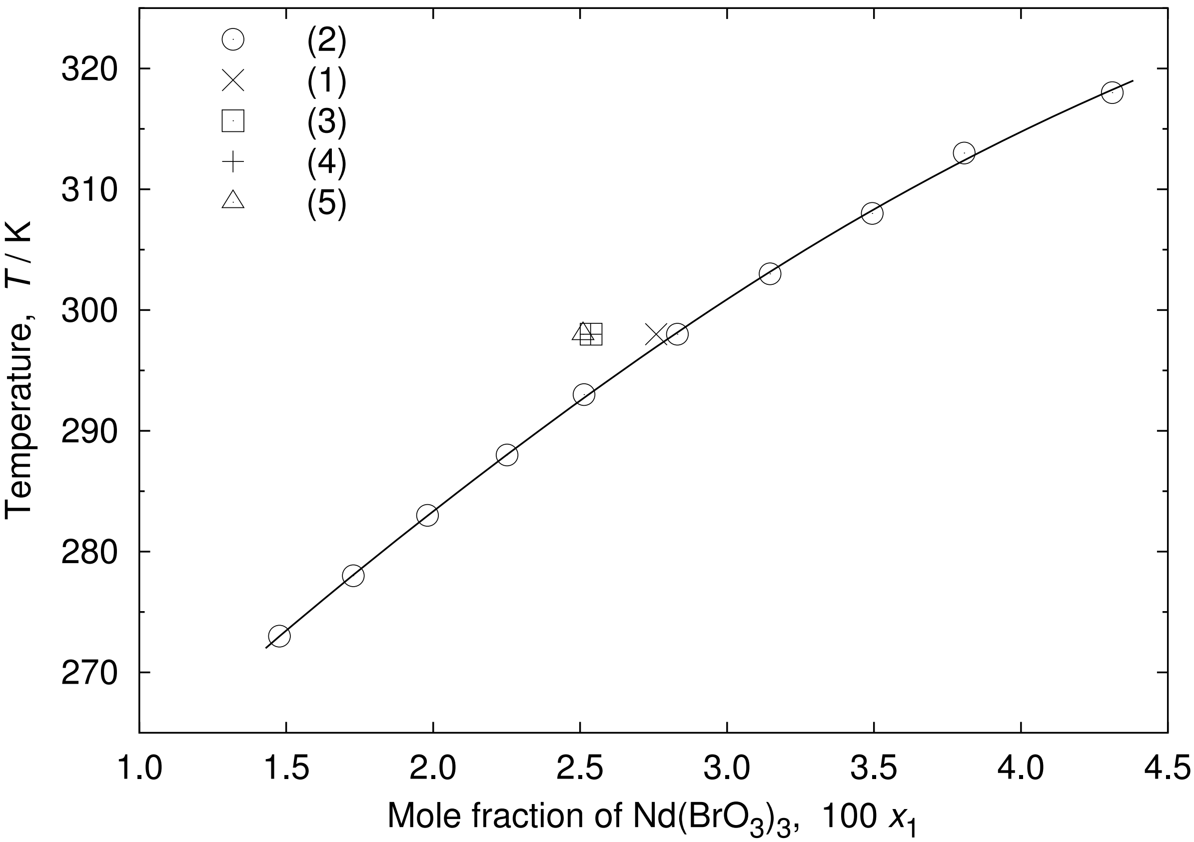

FIG. 15. Temperature dependence of solubility of $\mathrm{Nd(BrO_3)_3}$ in water

The Ternary System

Three ternary systems $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Pr(BrO_3)_3}$–$\mathbf{H_2O}$ (3), $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Yb(BrO_3)_3}$–$\mathbf{H_2O}$ (4) and $\mathrm{Nd(BrO_3)_3}$–$\mathbf{KBrO_3}$–$\mathbf{H_2O}$ (5) were studied by groups of Serebrennikov and Batyreva using the isothermal method. In the $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Pr(BrO_3)_3}$–$\mathbf{H_2O}$ system solid solution of crystalline hydrate of praseodymium and neodymium bromate was formed. In the $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Yb(BrO_3)_3}$–$\mathrm{H_2O}$ system two series of solid solutions were formed, and in the $\mathrm{Nd(BrO_3)_3}$–$\mathbf{KBrO_3}$–$\mathbf{H_2O}$ system solid solution was not reported. The reader finds the compilation of the $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Pr(BrO_3)_3}$–$\mathrm{H_2O}$ system in the $\mathrm{Nd(BrO_3)_3}$–$\mathbf{H_2O}$ section in this volume.

References:

1. C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913 (1909).
2. C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132 (1927).
3. V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **23**, 1947 (1978); Russ. J. Inorg. Chem. (Engl. Transl.) **23**, 1070 (1978).
4. V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **26**, 2837 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 1518 (1981).

5. V. A. Batyreva, I. S. Popova, and G. I. Putii, *Sintez i Reaktsionnaya Sposobnost Veshchestv*, (Tomsk, 1984), p. 71.

Components:

(1) Neodymium bromate; $Nd(BrO_3)_3$; [15162-92-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913–17 (1909).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported that 100 parts of water dissolve 146 parts of $Nd(BrO_3)_3 \cdot 9H_2O$ at 25 °C. The solubility of 45.4 mass %, 2.76 mol % or 1.58 mol kg^{-1} is calculated by the compiler. When heated to 100 °C the nonahydrated bromate loses water, forming $Nd(BrO_3)_3 \cdot 2H_2O$. The anhydrous salt was obtained by heating to 150 °C and it decomposes with evolution of heat at a higher temperature.

Auxiliary Information

Method/Apparatus/Procedure:

It seems that the authors used $Nd(BrO_3)_3 \cdot 9H_2O$ for the solubility determination. The equilibration method and a method of the saturated solution analysis were not described.

Source and Purity of Materials:

The preparation method of neodymium bromate nonahydrate was similar to that of lanthanum bromate salt. Neodymium bromate nonahydrate was prepared by the double decomposition of neodymium sulfate with barium bromate. The product was filtered, the filtrate boiled down and allowed to stand. The refiltered liquid was further concentrated and the neodymium bromate crystallized. The crystals were then drained upon a Hirsch funnel at the filter pump and dried in the air upon filter paper. The analytical methods for Br_2O_5 and Nd_2O_3 determination were not specified. The analytical results of the product were as follows: found: Br_2O_5–52.19; Nd_2O_3–24.34; H_2O (difference)–23.47 mass %; calculated for $Nd(BrO_3)_3 \cdot 9H_2O$: Br_2O_5–52.12; Nd_2O_3–24.39; H_2O (difference)–23.49 mass %.

Estimated Error:

Solubility: nothing specified.
Temperature: nothing specified.

Components:

(1) Neodymium bromate; $Nd(BrO_3)_3$; [15162-92-2]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132–5 (1927).

Variables:

Temperature: 273–318 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 161. Solubility of $Nd(BrO_3)_3$ in water at various temperatures

	$Nd(BrO_3)_3 \cdot 9H_2O$		$Nd(BrO_3)_3$	
t / °C	$100w_B$ [a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	39.89	66.35	30.52	1.447
5	44.46	80.06	34.01	1.728
10	48.61	94.57	37.19	1.980
15	52.68	111.32	40.30	2.252
20	56.26	128.6	43.04	2.514
25	60.21	151.3	46.06	2.831
30	63.75	175.9	48.77	3.146
35	67.29	205.8	51.48	3.494
40	70.19	235.4	53.70	3.807
45	74.37	289.9	56.90	4.311

[a] B (elementary entities) in w_B is $Nd(BrO_3)_3 \cdot 9H_2O$.

[b] Mass ratio is reported as the value of parts of $Nd(BrO_3)_3 \cdot 9H_2O$ per 100 parts of H_2O in the original paper.

[c] Cited in the Linke's book (1) and confirmed by the compilers.

[d] Calculated by the compilers from the mass % solubility of $Nd(BrO_3)_3$.

Auxiliary Information

Method/Apparatus/Procedure:

The bromate nonahydrate was used. The equilibrium was approached from both supersaturation and undersaturation. It seems that the authors used the method for Nd determination described in their previous paper (2). Bottles with oversaturated solutions were placed in a thermostat and rotated for more than 9 h. A sample of the saturated solution was withdrawn, weighed, diluted, precipitated with oxalic acid solution. The mixture was filtered after 12 h and the precipitate washed, ignited to oxide and weighed. All results are mean of duplicated analyses.

Source and Purity of Materials:

Neodymium bromate nonahydrate was prepared by stirring the pure neodymium sulfate with an excess of pure barium bromate suspended in water.

Estimated Error:

Solubility: precision within ± 1 %, but at higher temperatures within ± 1.5 %.
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1183.
[2] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Neodymium bromate; $\mathrm{Nd(BrO_3)_3}$; [15162-92-2]

(2) Ytterbium bromate; $\mathrm{Yb(BrO_3)_3}$; [28972-23-8]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **26**, 2837–40 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 1518–9 (1981).

Variables:

Composition of two salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 162. Composition of saturated solutions in the ternary $\mathrm{Nd(BrO_3)_3}$–$\mathrm{Yb(BrO_3)_3}$–H_2O system at 25 °C

$\mathrm{Nd(BrO_3)_3}$		$\mathrm{Yb(BrO_3)_3}$		
$100w_1$	$100x_1$ [a]	$100w_2$	$100x_2$ [a]	Solid phase[b]
43.26[c]	2.536	0	0	A
38.02	2.312	7.40	0.427	B
32.60	2.208	18.73	1.203	B
18.03	1.127	28.94	1.716	C
14.12	0.969	37.88	2.466	B+D
15.66	1.058	35.52	2.276	B+D
9.50	0.617	39.56	2.436	D
4.31	0.257	40.02	2.267	D
0	0	47.36[c]	2.829	E

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{Nd(BrO_3)_3 \cdot 9H_2O}$; B = solid solution based on $\mathrm{Nd(BrO_3)_3 \cdot 9H_2O}$; C = No information; D = solid solution based on $\mathrm{Yb(BrO_3)_3 \cdot 9H_2O}$; E = $\mathrm{Yb(BrO_3)_3 \cdot 9H_2O}$.

[c] For binary systems, the compilers compute the following values:

Solubility of $\mathrm{Nd(BrO_3)_3}$ = 1.44 mol kg^{-1},
Solubility of $\mathrm{Yb(BrO_3)_3}$ = 1.62 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal solubility method was used. Equilibrium in the systems was reached after 7 d of continuous stirring (1). No detailed information on method for analysis of the composition of the liquid phase was reported. The composition of the solid phases were determined by the Schreinemakers' method.

Source and Purity of Materials:

The bromate nonahydrate was made by dissolving the chemically pure grade oxides in pure, freshly prepared bromic acid; these oxides were prepared from the twice recrystallized hydroxides (1).

Estimated Error:

Solubility: precision no better than ± 0.5 % (compilers).

Temperature: nothing specified.

References:

[1] V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **23**, 1947 (1978); Russ. J. Inorg. Chem. (Engl. Transl.) **23**, 1070 (1978).

Components:

(1) Neodymium bromate; $\mathrm{Nd(BrO_3)_3}$; [15162-92-2]
(2) Potassium bromate; $KBrO_3$; [7758-01-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. A. Batyreva, I. S. Popova, and G. I. Putii, *Sintez i Reaktsionnaya Sposobnost Veshchestv*, (Tomsk, 1984), p. 71–3.

Variables:

Composition of two salts
One temperature: 298 K

Prepared By:

Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Compositions of the saturated solutions in $\mathrm{Nd(BrO_3)_3}$–$KBrO_3$–H_2O system at 25 °C were presented in a form of hardly readable diagram. The results were read out from the figure and recalculated to mol % by the compilers.

TABLE 163. Composition of saturated solutions in the ternary $\mathrm{Nd(BrO_3)_3}$–$KBrO_3$–H_2O system at 25 °C

$\mathrm{Nd(BrO_3)_3}$		$KBrO_3$		
$100w_1$	$100x_1$	$100w_2$	$100x_2$	Solid phase[a]
43.0	2.5	0	0	A
40.5	2.3	1.3	0.2	B
38.0	2.1	1.5	0.3	B
30.5	1.5	1.7	0.3	B
22.5	1.0	2.0	0.3	B
18.5	0.7	2.3	0.3	B
1.5	0.05	5.4	0.6	B
0	0	5.7	0.7	B

[a] The solid phases are: A = $\mathrm{Nd(BrO_3)_3 \cdot 9H_2O}$, B = $KBrO_3$

For the binary system the compilers compute the solubility of $\mathrm{Nd(BrO_3)_3}$ = 1.43 mol $\mathrm{kg^{-1}}$.

The eutonic point (reported numerically) was observed at 49.2 mass % $\mathrm{Nd(BrO_3)_3}$, 1.4 mass % $KBrO_3$ and 49.4 mass % H_2O (3.3, 0.3 and 96.4 mol %, respectively, as recalculated by the compilers). Refraction index and specific gravity of the saturated solutions (both presented in a figure) confirmed the shape of the liquidus line and the existence of the eutonic point. The equilibrium solid phase crystallizing in the vicinity of the eutonic point is a mixture of $\mathrm{Nd(BrO_3)_3}$ and $KBrO_3$ crystals.

Auxiliary Information

Method/Apparatus/Procedure:

The isothermal solubility method was used. Equilibrium in the system was reached after 7 d of continuous stirring with a glass bullet. Potassium content was determined by flame photometry and neodymium content was found from complexometric titration with EDTA. The refraction index and specific gravity of the saturated solutions were determined by a refractometer and pycnometer, respectively. Compositions of the solid phases were determined by the Schreinemakers' method and confirmed by X-ray analysis.

Source and Purity of Materials:

Nothing specified.

Estimated Error:

Solubility: nothing specified; reading-out procedure ± 0.3 mass %.

Temperature: nothing specified.

Components:
(1) Samarium bromate; $\mathrm{Sm(BrO_3)_3}$; [28958-26-1]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. August 2003.

Critical Evaluation:

The Binary System

Solubilities of $\mathrm{Sm(BrO_3)_3}$ in pure water have been reported in 3 publications (1–3). James and Langelier (1) reported the solubility of 2.28 mol % or 1.29 mol kg^{-1} at 298 K. James et al. (2) studied the solubility over the temperature range of 273–318 K. The solubility increases monotonically with increasing the temperature. Serebrennikov et al. (3) measured the solubility at 298 K. The compilation of the latter solubility study (3) is in the previous volume (4) and the solubility value found is 1.32 mol kg^{-1}. The solubility of James et al. (2) equals with that of Serebrennikov et al. (3). The arithmetic mean of three reported values is 1.31 mol kg^{-1} and is designated as a recommended solubility at 298 K with the standard deviation ± 0.01 mol kg^{-1}. The solid phase was reported to be $\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$ (2, 3).

The temperature dependence of the solubility reported by James et al. (2) with the data of James and Langelier (1) and Serebrennikov et al. (3) is shown in Fig. 16. The $\mathrm{Sm(BrO_3)_3}$ solubility results reported by James et al. (2) were used to obtain the smoothing equation. The detailed information of the calculation procedure for the smoothing equation has been described in the Preface in this volume. The obtained equation is

$$Y_x = \frac{-225166.301}{(T/\mathrm{K})} - 1399.78455 \ln{(T/\mathrm{K})} + 8060.17656 + 2.24205433(T/\mathrm{K}), \tag{77}$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \tag{78}$$

$\sigma_Y = 0.0072$ and $\sigma_x = 3.5 \times 10^{-5}$.

The solubilities calculated from the fitting equation are given in Table 164. These values calculated from the smoothing equation are designated as tentative solubilities.

TABLE 164. Tentative solubility of $\mathrm{Sm(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	1.14	0.64
278	1.35	0.76

283	1.58	0.89
288	1.82	1.03
293	2.06	1.17
298	2.33	1.32
303	2.60	1.48
308	2.89	1.65
313	3.21	1.84
318	3.56	2.05

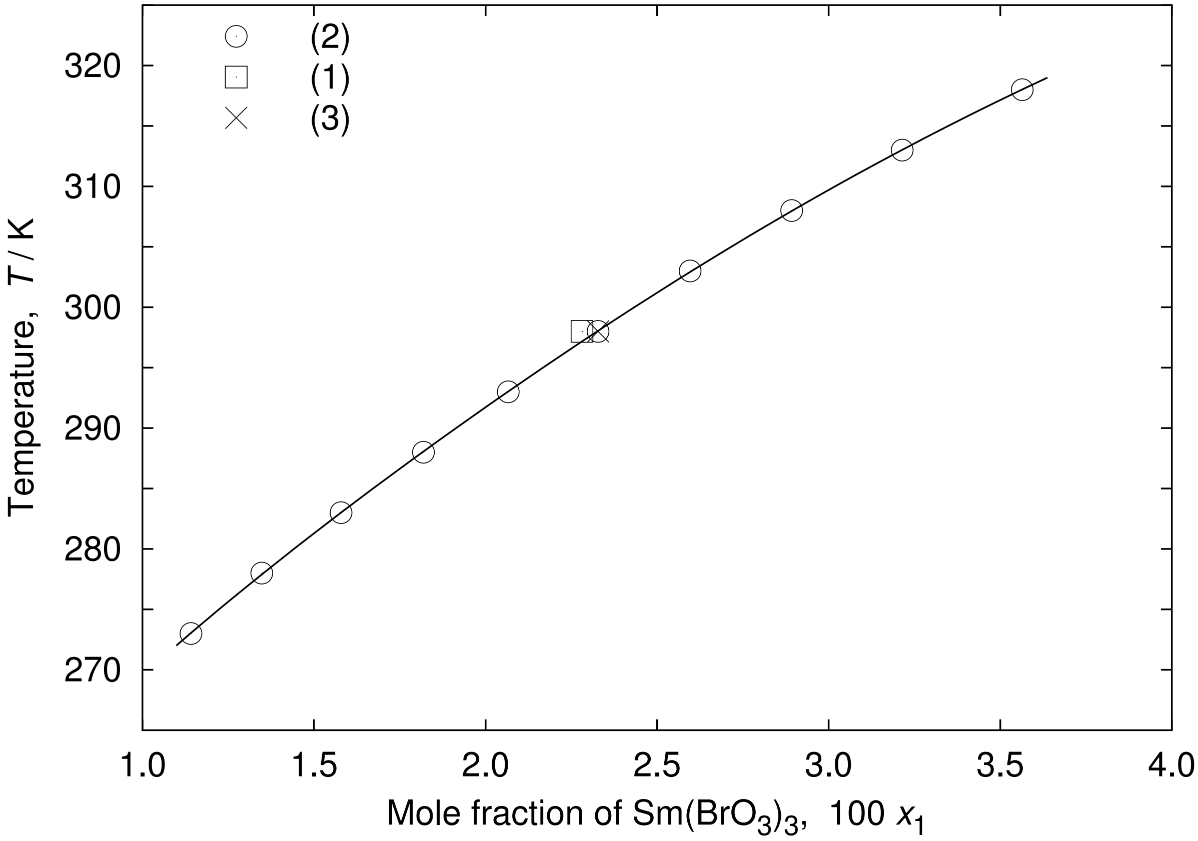

FIG. 16. Temperature dependence of solubility of $\mathrm{Sm(BrO_3)_3}$ in water

The Ternary System

Data for the solubilities in ternary $\mathrm{Sm(BrO_3)_3}$–$KBrO_3$–H_2O system have been reported by Serebrennikov et al. (3). The system is a simple eutonic type. The reader finds the compilation of this system in preceding volume (4).

References:

1. C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913 (1909).
2. C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132 (1927).
3. V. V. Serebrennikov, V. A. Batyreva, and I. S. Larionova, Zh. Neorg. Khim. **27**, 2959 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 1677 (1982).
4. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 241.

Components:
(1) Samarium bromate; $\mathrm{Sm(BrO_3)_3}$; [28958-26-1]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
C. James and W. F. Langelier, J. Am. Chem. Soc. **31**, 913–17 (1909).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The authors reported that 100 parts of water dissolve 114 parts of $\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$ at 25 $^\circ$C. The solubility of 40.9 mass %, 2.28 mol % or 1.29 mol kg^{-1} is calculated by the compiler.

When the nonahydrate salt was heated at 100 $^\circ$C, this salt transformed into $\mathrm{Sm(BrO_3)_3 \cdot 2H_2O}$. All water of crystallization was lost at 150 $^\circ$C while the salt decomposed at a higher temperature.

Auxiliary Information

Method/Apparatus/Procedure:
It seems that the authors used $\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$ for the solubility determination. The equilibration method and a method of the saturated solution analysis were not described.

Source and Purity of Materials:
The bromate nonahydrate was prepared by the double decomposition of samarium sulfate with barium bromate. The product was filtered, the filtrate boiled down and allowed to stand. The refiltered liquid was further concentrated and the samarium bromate crystallized. The crystals were then drained upon a Hirsch funnel at the filter pump and dried in the air upon filter paper. The analytical method for $\mathrm{Br_2O_5}$ and $\mathrm{Sm_2O_3}$ determinations were not specified. The analytical results of the product were as follows: found: $\mathrm{Br_2O_5}$–51.66; $\mathrm{Sm_2O_3}$–24.97; H_2O (difference)–23.37 mass %; calculated for $\mathrm{Sm(BrO_3)_3{\cdot}9H_2O}$ (original reported data): $\mathrm{Br_2O_5}$–51.69; $\mathrm{Sm_2O_3}$–25.04; H_2O (difference)–23.28 mass %. Because these values calculated by the authors were contained erroneous data the values recalculated by the compiler using 2003 IUPAC recommended atomic weights were as follows: $\mathrm{Br_2O_5}$–51.67; $\mathrm{Sm_2O_3}$–25.04; H_2O (difference)–23.29 mass %.

Estimated Error:
Solubility: nothing specified.
Temperature: nothing specified.

Components:

(1) Samarium bromate; $\mathrm{Sm(BrO_3)_3}$; [28958-26-1]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem Soc. **49**, 132–5 (1927).

Variables:

Temperature: 273–318 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 165. Experimental solubility of $\mathrm{Sm(BrO_3)_3}$ in water at various temperatures

	$\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$		$\mathrm{Sm(BrO_3)_3}$	
t / °C	$100w_B$ [a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	33.24	49.78	25.50	1.141
5	37.58	60.21	28.83	1.348
10	42.02	72.47	32.23	1.579
15	46.21	85.91	35.45	1.819
20	50.16	100.6	38.48	2.066
25	53.97	117.3	41.40	2.328
30	57.54	135.5	44.14	2.596
35	61.12	157.2	46.88	2.892
40	64.67	183.0	49.61	3.214
45	68.16	214.1	52.28	3.565

[a] B (elementary entities) in w_B is $\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$.

[b] Mass ratio is reported as the amount of parts of $\mathrm{Sm(BrO_3)_3 \cdot 9H_2O}$ per 100 parts of H_2O in the original paper.

[c] Cited in the Linke's book (1) and confirmed by the compilers.

[d] Calculated by the compilers from the mass % solubility of $\mathrm{Sm(BrO_3)_3}$.

Auxiliary Information

Method/Apparatus/Procedure:

Samarium bromate nonahydrate was used. The equilibrium was approached from both supersaturation and undersaturation. It seems that the authors used the method for Sm determination described in their previous paper (2). Bottles with oversaturated solutions were placed in a thermostat and rotated for more than 9 h. A sample of the saturated solution was withdrawn, weighed, diluted, precipitated with oxalic acid solution. The mixture was filtered after 12 h and the precipitate washed, ignited to oxide and weighed. All results are mean of duplicated analyses.

Source and Purity of Materials:

Samarium bromate nonahydrate was prepared by stirring the pure samarium sulfate with an excess of pure barium bromate suspended in water.

Estimated Error:

Solubility: precision within 1 %, but at higher temperatures within 1.5 %.
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1456.
[2] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Europium bromate; $Eu(BrO_3)_3$; [62667-63-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

The solubility of europium bromate in water at 25 °C is reported to be 1.21 mol kg^{-1}. This value is designated as a tentative solubility. The equilibrium solid phase was not defined but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $Eu(BrO_3)_3$ is 3.77 J.

Auxiliary Information

Method/Apparatus/Procedure:

A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified procedure, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:

The bromate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrate of a molar ratio of water/bromate > 9 was obtained.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

G. Gadolinium Bromate

Components:

(1) Gadolinium bromate; $Gd(BrO_3)_3$; [28958-27-2]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem. Soc. **49**, 132–5 (1927).

Variables:

Temperature: 273–318 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data and Critical Evaluation

Experimental and calculated solubilities of $Gd(BrO_3)_3$ in water at the selected temperatures are given in Tables 166 and 167, respectively.

TABLE 166. Experimental solubility of $Gd(BrO_3)_3$ in water at various temperatures

	$Gd(BrO_3)_3 \cdot 9H_2O$		$Gd(BrO_3)_3$	
t / °C	$100w_B$[a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	33.41	50.18	25.71	1.139
5	37.50	60.01	28.86	1.333
10	41.21	70.11	31.71	1.523
15	45.25	82.64	34.81	1.747
20	48.87	95.58	37.60	1.967
25	52.49	110.5	40.39	2.207
30	55.78	126.1	42.91	2.442
35	59.10	144.5	45.47	2.702
40	62.39	166.0	48.01	2.984
45	66.16	195.6	50.91	3.338

[a] B (elementary entities) in w_B is $Gd(BrO_3)_3 \cdot 9H_2O$.

[b] Mass ratio is reported as the value of parts of $Gd(BrO_3)_3 \cdot 9H_2O$ per 100 parts of H_2O in the original paper.

[c] Calculated by the evaluators from the experimental data.

[d] Calculated by the evaluators from the mass % solubility.

The composition of the solid phase was $\mathrm{Gd(BrO_3)_3 \cdot 9H_2O}$.

The mass % solubility of $\mathrm{Gd(BrO_3)_3}$ was used to obtain the smoothing equation. The detailed information of the calculation procedure for the smoothing equation was described in the Preface of this volume. The equation is:

$$Y_x = \frac{-225544.435}{(T/\mathrm{K})} - 1428.43491 \ln{(T/\mathrm{K})} + 8198.27282 + 2.32995991(T/\mathrm{K}), \tag{79}$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \tag{80}$$

$\sigma_Y = 0.016$ and $\sigma_x = 0.00011$.

The solubilities calculated from the fitting equation are given in Table 167. These values calculated from the smoothing equation are designated as tentative solubilities.

TABLE 167. Tentative solubility of $\mathrm{Gd(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	1.14	0.64
278	1.33	0.75
283	1.53	0.86
288	1.74	0.99
293	1.96	1.11
298	2.20	1.25
303	2.44	1.39
308	2.71	1.54
313	3.00	1.72
318	3.33	1.91

Temperature dependence of the solubility calculated from smoothing equation with experimental solubility of gadolinium bromate in water is shown in Fig. 17.

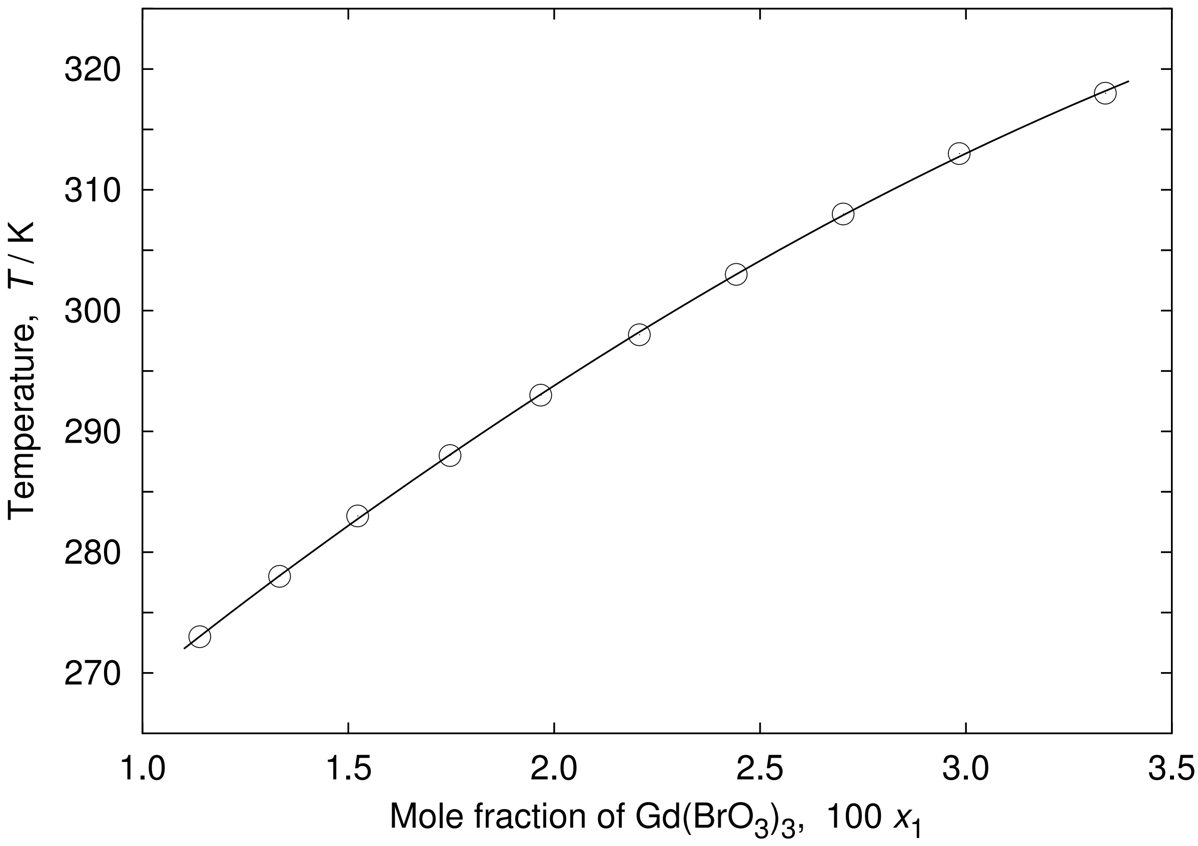

FIG. 17. Temperature dependence of solubility of $Gd(BrO_3)_3$ in water

Auxiliary Information

Method/Apparatus/Procedure:

Gadolinium bromate nonahydrate was used. The equilibrium was approached from both supersaturation and undersaturation. Bottles with oversaturated solutions were placed in a thermostat and rotated for 9 h or more. A sample of the saturated solution was withdrawn, weighed, diluted, precipitated with oxalic acid solution. The mixture was filtered after 12 h and the precipitate was washed, ignited to oxide and weighed. All results are mean of duplicate analyses.

Source and Purity of Materials:

Gadolinium bromate nonahydrate was prepared by stirring the pure gadolinium sulfate with an excess of pure barium bromate suspended in water.

Estimated Error:

Solubility: precision within 1 %, but at higher temperatures within 1.5 %.

Temperature: nothing specified.

References:

[1] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Terbium bromate; $\mathrm{Tb(BrO_3)_3}$; [not available]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. August 2002.

Critical Evaluation:

The solubility of $\mathrm{Tb(BrO_3)_3}$ in water has been reported in two publications (1, 2). James et al. (1) studied the solubility over a wide temperature range of 273–318 K, and Staveley et al. (2) measured the solubility at 298 K. The solid phase was not analyzed in both papers, but the evaluators assumed it to be the nonahydrate because James et al. (1) used it as the starting material.

The solubility values of 1.45 and 1.42 mol kg^{-1} at 298 K have been reported by James et al. (1) and Staveley et al. (2), respectively. The arithmetic mean of two results is 1.44 mol kg^{-1}, and the standard deviation is 0.01 mol kg^{-1}.

The temperature dependence of the solubility reported by James et al. (1) with Staveley et al. (2) was shown in Fig. 18. The detailed information of the calculation procedure for the smoothing equation has been described in the Preface in this volume. The $\mathrm{Tb(BrO_3)_3}$ solubility reported by James et al. (1) was used to obtain the smoothing equation. The equation is:

$$Y_x = \frac{-139442.297}{(T/\mathrm{K})} - 871.40816 \ln{(T/\mathrm{K})} + 5007.51611 + 1.42022714(T/\mathrm{K}), \qquad (81)$$

where equation (2) described in Preface was as follows:

$$Y_x = \ln\left(\frac{x^4(1-x)^9(4+9)^{4+9}}{9^9(1+x)^{4+9}}\right). \qquad (82)$$

$\sigma_Y = 0.0057$ and $\sigma_x = 3.9 \times 10^{-5}$. The solubilities calculated from the fitting equation are given in Table 168; these values are designated as tentative.

TABLE 168. Tentative values of the solubility of $\mathrm{Tb(BrO_3)_3}$ in water

T / K	$100x_1$	m_1 / mol kg^{-1}
273	1.45	0.82
278	1.65	0.93
283	1.86	1.05
288	2.08	1.18
293	2.30	1.31
298	2.54	1.45
303	2.79	1.60
308	3.06	1.75
313	3.36	1.93
318	3.68	2.12

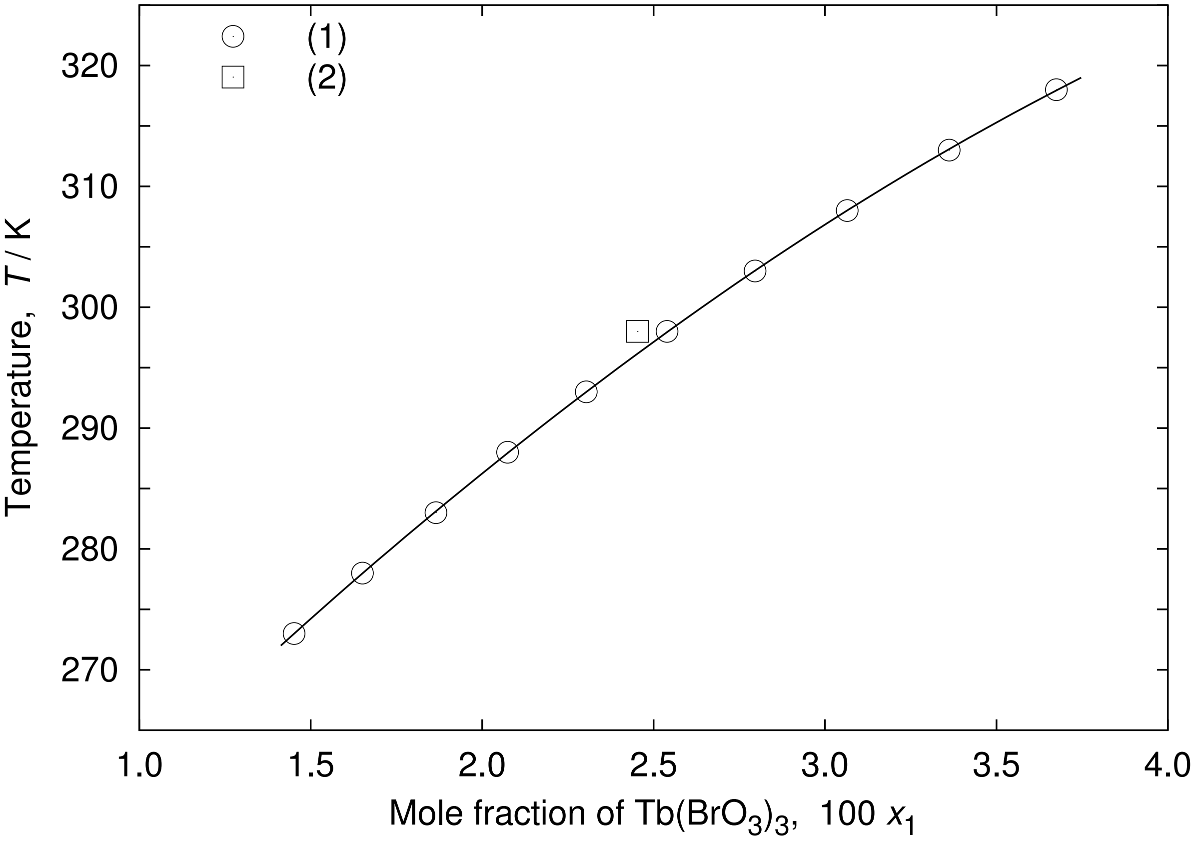

FIG. 18. Temperature dependence of solubility of $Tb(BrO_3)_3$ in water

References:

1. C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem. Soc. **49**, 132 (1927).
2. L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231 (1968).

Components:

(1) Terbium bromate; $\mathrm{Tb(BrO_3)_3}$; [not available]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

C. James, H. C. Fogg, B. W. McIntire, R. H. Evans, and J. E. Donovan, J. Am. Chem. Soc. **49**, 132–5 (1927).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 169. Experimental solubility of $\mathrm{Tb(BrO_3)_3}$ in water at various temperatures

	$\mathrm{Tb(BrO_3)_3 \cdot 9H_2O}$		$\mathrm{Tb(BrO_3)_3}$	
t / °C	$100w_B$ [a]	Mass ratio[b]	$100w_1$ [c]	$100x_1$ [d]
0	39.91	66.42	30.73	1.451
5	43.61	77.34	33.58	1.651
10	47.28	89.68	36.41	1.865
15	50.59	102.4	38.95	2.074
20	53.93	117.1	41.53	2.375
25	57.11	133.2	43.97	2.539
30	60.29	151.9	46.42	2.796
35	63.35	172.9	48.78	3.065
40	66.45	198.1	51.17	3.362
45	69.44	227.1	53.47	3.675

[a] B (elementary entities) in w_B is $\mathrm{Tb(BrO_3)_3 \cdot 9H_2O}$.

[b] Mass ratio is reported as the value of parts of $\mathrm{Tb(BrO_3)_3 \cdot 9H_2O}$ per 100 parts of H_2O in the original paper.

[c] Cited in the Linke's book (1) and confirmed by the compilers.

[d] Calculated by the compilers from the mass % solubility.

Auxiliary Information

Method/Apparatus/Procedure:

The terbium bromate nonahydrate was used. It seems that the authors used the method for Tb determination described in (2). Oversaturated solutions in bottles were placed in a thermostat and rotated for more than 9 h. A sample of the saturated solution was withdrawn, weighed, diluted, precipitated with oxalic acid solution. The mixture was filtered after 12 h and the precipitate was washed, ignited to oxide and weighed. All results are mean of duplicate analyses.

Source and Purity of Materials:

Terbium bromate nonahydrate was prepared by stirring the pure terbium sulfate with an excess of pure barium bromate suspended in water.

Estimated Error:

Solubility: precision within 1 %, but at higher temperatures within 1.5 %.
Temperature: nothing specified.

References:

[1] W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1527.
[2] M. D. Williams, H. C. Fogg, and C. James, J. Am. Chem. Soc. **47**, 297 (1925).

Components:

(1) Terbium bromate; $\mathrm{Tb(BrO_3)_3}$; [not available]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of terbium bromate in water at 25 °C is reported to be 1.42 mol kg^{-1}. The equilibrium solid phase was not defined but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $\mathrm{Tb(BrO_3)_3}$ is 10.32 J.

Auxiliary Information

Method/Apparatus/Procedure:

A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified method, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:

Terbium bromate nonahydrate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).
Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

Components:
(1) Dysprosium bromate; $\mathrm{Dy(BrO_3)_3}$; [28958-28-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

The solubility of dysprosium bromate in water at 25 $^\circ\mathrm{C}$ is reported to be 1.54 mol kg^{-1}. This value is designated as a tentative solubility. The equilibrium solid phase was not defined but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $\mathrm{Dy(BrO_3)_3}$ is 3.47 J.

Auxiliary Information

Method/Apparatus/Procedure:
A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified method, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:
Dysprosium bromate nonahydrate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:
Solubility: precision no better than $\pm$ 1 % (compilers).
Temperature: due to the calorimetric experiments the precision and accuracy should be better than $\pm$ 0.1 K (compilers).

Components:
(1) Erbium bromate; $\mathrm{Er(BrO_3)_3}$; [28958-30-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

The solubility of erbium bromate in water at 25 °C is reported to be 2.04 mol $\mathrm{kg^{-1}}$. This value is designated as tentative. The equilibrium solid phase was not defined but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol $\mathrm{dm^{-3}}$ $\mathrm{Er(BrO_3)_3}$ is 5.06 J.

Auxiliary Information

Method/Apparatus/Procedure:
A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified method, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:
Erbium bromate nonahydrate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol $\mathrm{dm^{-3}}$ bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:
Solubility: precision no better than ± 1 % (compilers).
Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

Components:

(1) Ytterbium bromate; $Yb(BrO_3)_3$; [28972-23-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan. August 2002.

Critical Evaluation:

The Binary System

Three values of solubility of $Yb(BrO_3)_3$ in pure water at 298 K have been reported (1–3). The value of Staveley et al. (1) was 2.14 mol kg^{-1}, and the values of groups of Serebrennikov et al. (2) and Batyreva et al. (3) were 1.62 and 1.63 mol kg^{-1}, respectively. The evaluator assumed that agreement between the value in (1) and those in (2, 3) fails. Redetermination is required to decide a recommended solubility. Reluctantly the arithmetic mean of the three values is 1.8 mol kg^{-1} and the standard deviation is 0.2. The mean is designated as a doubtful solubility at 298 K.

The Ternary System

Data for the solubilities of in the ternary $Nd(BrO_3)_3$–$Yb(BrO_3)_3$–H_2O system was reported by Serebrennikov et al. (2). The corresponding compilation was described in the $Nd(BrO_3)_3$–H_2O system in this volume. Two series of solid solution based on $Nd(BrO_3)_3$ and that based on $Yb(BrO_3)_3$ were found to exist in the system. Batyreva et al. (3) studied the ternary $La(BrO_3)_3$–$Yb(BrO_3)_3$–H_2O system at 298 K. In this system, two series of solid solution based on $La(BrO_3)_3$ and $Yb(BrO_3)_3$ were formed. The compilation of this system can be found in the $La(BrO_3)_3$–H_2O system section in this volume.

References:

1. L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231 (1968).
2. V. V. Serebrennikov, V. A. Batyreva, and T. N. Tsybukova, Zh. Neorg. Khim. **26**, 2837 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 1518 (1981).
3. V. A. Batyreva, O. V. Nikitina, and O. A. Loskutova, Zh. Neorg. Khim. **45**, 1740 (2000); Russ. J. Inorg. Chem. (Engl. Transl.) **45**, 1600 (2000).

Components:

(1) Ytterbium bromate; $\mathrm{Yb(BrO_3)_3}$; [28972-23-8]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of ytterbium bromate in water at 25 $^\circ\mathrm{C}$ is 2.14 mol kg^{-1}. The equilibrium solid composition was not reported but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $\mathrm{Yb(BrO_3)_3}$ is 4.02 J.

Auxiliary Information

Method/Apparatus/Procedure:

A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified method, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified KI and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:

Ytterbium bromate nonahydrate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crushed and sieved. The hydrate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:

Solubility: precision no better than ± 1 % (compilers).

Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

Components:
(1) Lutetium bromate; $\mathrm{Lu(BrO_3)_3}$; [28958-31-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
L. A. K. Staveley, D. R. Markham, and M. R. Jones, J. Inorg. Nucl. Chem., **30**, 231–40 (1968).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data and Critical Evaluation

The solubility of lutetium bromate in water at 25 $^\circ\mathrm{C}$ is reported to be 2.31 mol kg^{-1}. The solubility result is treated as a tentative value. The equilibrium solid phase was not defined but it may be assumed to be a nonahydrate by analogy to other lanthanides.

Heat of dilution of 1 g of the saturated solution to form $\sim$ 0.005 mol dm^{-3} $\mathrm{Lu(BrO_3)_3}$ is 6.15 J.

Auxiliary Information

Method/Apparatus/Procedure:
A differential calorimeter was used to determine the heat of dilution of the saturated solution. The saturated solution, obtained by an unspecified method, was diluted with known amount of water in the calorimeter. The diluted solution was analyzed by addition of acidified potassium iodide and titration of the liberated I_2 with a standard solution of sodium thiosulfate.

Source and Purity of Materials:
Lutetium bromate nonahydrate was prepared from the hydroxide (purity 99.95+ %) obtained from Johnson Matthey and a solution of 0.6 mol dm^{-3} bromic acid, freed from bromide ion. The bromic acid solution was allowed to react with a slight excess of a suspension of the hydroxide. After filtering off the excess of the hydroxide, the solution was concentrated until it deposited crystals on standing. The crystals were separated from the mother liquor by centrifuging and then crashed and sieved. The hydrate of a molar ratio of water/bromate $>$ 9 was obtained.

Estimated Error:
Solubility: precision no better than ± 1 % (compilers).
Temperature: due to the calorimetric experiments the precision and accuracy should be better than ± 0.1 K (compilers).

IX. SCANDIUM, YTTRIUM, LANTHANUM AND LANTHANIDE IODATES

A. Scandium Iodate

Components:

(1) Scandium iodate; $\mathrm{Sc(IO_3)_3}$; [42096-67-3]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

There is only one reported publication on $\mathrm{Sc(IO_3)_3}$ solubility. Vinogradov et al. (1) studied isothermally the solubility in pure water at 298 K. This study deals with the ternary $\mathrm{Sc(IO_3)_3}$–HIO_3–H_2O system and the binary system where is given in as one point on the phase diagram. The stable solid in equilibrium with the saturated solution is $\mathrm{Sc(IO_3)_3 \cdot 18H_2O}$. The solubility in water is 3.7 × 10^{-3} mol kg^{-1} at 298 K and it is designated as a tentative value.

The Ternary System

Vinogradov et al. (1) studied the ternary $\mathrm{Sc(IO_3)_3}$–HIO_3–H_2O system. The dominant feature of the ternary system with $\mathrm{Sc(IO_3)_3}$ is an existence of double salt $\mathrm{Sc(IO_3)_3 \cdot 4HIO_3 \cdot 18H_2O}$. The reader finds the detailed compilation in (2).

References:

1. E. E. Vinogradov, I. N. Lepeshkov, and G. N. Tarasova, Zh. Neorg. Khim. **22**, 2858 (1977); Russ. J. Inorg. Chem. (Engl. Transl.) **22**, 1552 (1977).
2. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 484.

Components:
(1) Yttrium iodate; $\mathrm{Y(IO_3)_3}$; [14723-99-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Solubility data for yttrium iodate in water at 298 K have been reported in 4 publications (1–4). The value of 1.92×10^{-3} mol dm^{-3} and that of 1.93×10^{-3} mol dm^{-3} were reported by Firsching and Paul (1) and by Bertha and Choppin (2), respectively. The solubility value reported in the book by Linke (3) was 0.53 g of $\mathrm{Y(IO_3)_3}$ in 100 g of H_2O, and the solubility of 8.6×10^{-4}mol kg^{-1} was computed by the evaluators. This solubility value is rejected, because there is a large difference between this and the values of (1) and (2); in addition, no information about temperature and method used is given. Pruitt et al. (4) have reported the solubility of $\mathrm{Y(IO_3)_3}$ on the solubility curve as one point on the quarternary $\mathrm{Y(IO_3)_3}$–KIO_3–HNO_3–H_2O system. Their results were presented as a figure and numerical data were read out by the compilers. Their $\mathrm{Y(IO_3)_3}$ solubility in pure water was found to be 2.17×10^{-3} mol dm^{-3} being 10 % higher than the results of (1) and (2). Vasil'ev and Serebrennikov (5) have reported the solubility of $\mathrm{Y(IO_3)_3}$ in acidic solution at pH 5.6–5.7; the value of 5.5×10^{-4} mol dm^{-3} found in their experiments is several times lower than in (1), (2) and (4). Authors of paper (4) showed that pH influence on the solubility is not essential. The arithmetic mean of two reasonable results (1, 2) is 1.93×10^{-3} mol dm^{-3} and it is designated as a tentative value at 298 K.

According to (1) and (5) unhydrated $\mathrm{Y(IO_3)_3}$ is in equilibrium with the saturated solution but this result should be confirmed.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. W. F. Linke (originally edited by A. Seidell), *Solubilities: Inorganic and Metal-Organic Compounds*, 4th ed. (American Chemical Society, Washington, D. C., 1965) Vol. 2, p. 1639.
4. M. E. Pruitt, R. R. Rickard, and E. I. Wyatt, Anal. Chem. **34**, 283 (1962).
5. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).

Components:

(1) Yttrium iodate; $\mathrm{Y(IO_3)_3}$; [14723-99-0]
(2) Potassium iodate; KIO_3; [7758-05-6]
(3) Nitric acid; HNO_3; [7697-37-2]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

M. E. Pruitt, R. R. Rickard, and E. I. Wyatt, Anal. Chem. **34**, 283–7 (1962).

Variables:

Composition of KIO_3 and HNO_3
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

Solubility curves for yttrium iodate in nitric acid–potassium iodate solutions at 25 °C were reported in a figure. They were read from the figure by compilers.

TABLE 170. Solubility of $\mathrm{Y(IO_3)_3}$ in HNO_3–KIO_3 solutions at 25 °C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	γ_1 / g dm^{-3}	$10^3 c_1$ / mol dm^{-3}
0	0	1.33	2.17
0.05	0	0.19	0.31
0.1	0	0.11	0.18
0.2	0	0.07	0.11
0.3	0	0.03	0.05
0	0.05	0.89	1.45
0.05	0.05	0.15	0.24
0.1	0.05	0.10	0.16
0.2	0.05	0.06	0.10
0.3	0.05	0.02	0.03
0	0.1	0.87	1.42
0.05	0.1	0.21	0.34
0.1	0.1	0.14	0.23
0.2	0.1	0.07	0.11
0.3	0.1	0.03	0.05
0	0.2	1.92	0.31
0.05	0.2	0.65	1.06
0.1	0.2	0.22	0.32
0.2	0.2	0.07	0.11
0.3	0.2	0.03	0.05
0	0.4	5.04	8.21
0.05	0.4	1.40	2.28
0.1	0.4	0.54	0.88
0.2	0.4	0.14	0.23
0.3	0.4	0.03	0.05

0.05	0.7	3.81	6.21
0.2	0.7	0.73	1.19
0.3	0.7	0.03	0.05
0.1	1.0	3.57	5.82
0.2	1.0	2.01	3.28
0.3	1.0	0.29	0.47

Composition of the equilibrium solid was not specified.

Auxiliary Information

Method/Apparatus/Procedure:

Yttrium iodate tagged with ^{91}Y was used. The solubility data of yttrium iodate were obtained by adding the tagged yttrium iodate to solution of nitric acid and iodate ion of various concentrations. The solutions were allowed to remain in contact with an excess of the iodate precipitate until a change in solubility was no longer observed. This equilibrium concentration was determined by counting aliquots of the supernatant liquid over a period of several weeks. An aliquot of the solution in contact with the tagged yttrium iodate was evaporated on a watch glass and was counted on a radioactivity counter. From the previously determined specific activity of the yttrium iodate, the counting rate of the supernatant liquid was direct measurement of solubility of yttrium iodate in each solution.

Source and Purity of Materials:

Yttrium iodate was prepared by adding reagent grade potassium iodate to a neutral solution of yttrium nitrate that contained 1 mCi of ^{91}Y. The precipitate was washed with water, and the suspension was filtered through a sintered-glass funnel. The yttrium iodate was washed with ethanol and with ether and was vacuum desiccated to constant weight.

Estimated Error:

Solubility: reading-out procedure $\pm 3 \times 10^{-5}$ mol dm^{-3}.
Temperature: nothing specified.

Components:

(1) Yttrium iodate; $\mathrm{Y(IO_3)_3}$; [14723-99-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Y(IO_3)_3}$ in water acidified to pH 5.6–5.7 at temperature of 25 $^\circ\mathrm{C}$ is given:

0.339 g $\mathrm{cm^{-3}}$	(the authors)
0.552×10^{-3} mol $\mathrm{dm^{-3}}$	(recalculated by the compilers)

Molar ratio of yttrium to iodate was found to be $1:3$. The number of hydrating water molecules in the salt was not reported. A formation of solid $\mathrm{Y_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of the salt was introduced into water in a 25 $\mathrm{cm^{-3}}$ pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and was shaken mechanically at selected temperature for 10–12 h until saturation (1). The water used for solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in two samples of the solution. The samples were titrated with thiosulfate standard solution in presence of starch as the indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:

Yttrium iodate was prepared from acidic solution of yttrium chloride (99.8+ % pure) and potassium iodate solution. Yttrium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.01 K (probably as in (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:
(1) Yttrium iodate; $\mathrm{Y(IO_3)_3}$; [14723-99-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414-6 (1966).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported the yttrium and iodate concentrations in the saturated solution at 25.0 °C:

$[\mathrm{Y^{3+}}] = (1.90 \pm 0.02) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO_3^-}] = 5.8 \times 10^{-3}$, 5.8×10^{-3}, 5.8×10^{-3}, and 5.6×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of solubility:

$$\text{Solubility of } \mathrm{Y(IO_3)_3} = 1.92 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product reported by the authors was:

$$K_s^\circ = 1.10 \times 10^{-10} \text{mol}^4 \text{dm}^{-12}.$$

The activity coefficients were calculated at the ionic strength of the equilibrium solution of 0.011 mol dm^{-3} and the ion size data of Kielland (1).

The composition of equilibrium solid was not specified.

Auxiliary Information

Method/Apparatus/Procedure:
Yttrium iodate and water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet at 25 °C for several days. Three slightly modified procedures were also used. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. Yttrium content was determined by an EDTA titration with methylthymol and xylenol orange indicator.

Source and Purity of Materials:
Yttrium iodate was prepared by dissolving its oxide (99.8 % purity) in an excess of HNO_3 solution. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of yttrium. The precipitated iodate was separated and then agitated with water at 80 °C for several hours to remove any soluble species.

Estimated Error:
Solubility: precision better than ± 2 %.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

Components:

(1) Yttrium iodate; $\mathrm{Y(IO_3)_3}$; [14723-99-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613-7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Y(IO_3)_3}$ in water at 25 °C is 1.93×10^{-3} mol dm^{-3}. The solubility product, K_{s}°, of $\mathrm{Y(IO_3)_3}$ in water at 25 °C at infinite dilution is 7.10×10^{-11}mol^{4}dm^{-12}. The solubility product for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation. The formation constant of $\mathrm{YIO_3^{2+}}$ ion was 18 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}. The enthalpy of solution of $\mathrm{Y(IO_3)_3}$ at infinite dilution was determined to be 9.2 kJ mol^{-1}. The free energy and entropy of solutions for $\mathrm{Y(IO_3)_3}$ were 57.0 kJ and -0.164 J mol^{-1} K^{-1}, respectively.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Y(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made with each sample of the iodate. The heat of solution of the salt was measured in a calorimeter.

Source and Purity of Materials:

Yttrium iodate was prepared from vigorously stirred dilute solution of $\mathrm{Y(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water ($HClO_4$, pH 3.25). Thermal balance curves for the product indicated that there were no water of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. Yttrium perchlorate solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solution was evaporated under a heat lamp to remove excess of the acid and then the $\mathrm{Y(ClO_4)_3}$ solution was adjusted to pH 3.0–3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of lanthanum iodate in water have been reported in 11 publications (1–11). Four studies deal solely with the binary system (1, 8, 9, 11). One study has reported the solubility in water together with the solubilities in aqueous–organic solvent mixtures (7). The remaining studies deal with ternary or multicomponent systems (2–6, 10), and then the solubility in pure water has been reported as one point. Most studies are based on isothermal measurements with chemical analysis, and some of them deals with conductivity measurements in order to determine the solubility of the lanthanum iodate in pure water.

The most typical method for preparation of lanthanum iodate is the reaction between aqueous solution of lanthanum nitrate and aqueous solution of alkali metal iodate (2–5, 8). Frequently the syntheses were performed in dilute nitric acid solution. Iodic acid was alternatively used in place of alkali metal iodates (1). Although, lanthanum iodate prepared by these methods was obtained as finely crystalline compound, in number of cases a compound of amorphous type was precipitated with varying quantities of bounded water. The compositions of lanthanum iodate hydrates obtained in the studies of thermal decomposition are given in Table 171.

TABLE 171. Composition of lanthanum iodate hydrates

Composition	Registry number	Reference
$\mathrm{La(IO_3)_3}$	[13870-19-4]	(2), (9)
$\mathrm{La(IO_3)_3 \cdot 1/2H_2O}$	[56491-56-6]	(15), (16)
$\mathrm{La(IO_3)_3 \cdot 3/2H_2O}$	[not available]	(1)
$\mathrm{La(IO_3)_3 \cdot 5/2H_2O}$	[not available]	(12), (14)
$\mathrm{La(IO_3)_3 \cdot 3H_2O}$	[40854-11-3]	(10)
$\mathrm{La(IO_3)_3 \cdot 3H_2O}$ (amorphous)	[not available]	(21)
$\mathrm{La(IO_3)_3 \cdot 5H_2O}$	[24859-35-6]	(15), (17)
$\mathrm{La(IO_3)_3 \cdot 6H_2O}$	[56491-55-5]	(15)

According to (17) there is a partial dehydration of $\mathrm{La(IO_3)_3 \cdot 5H_2O}$ at $\sim$ 400 K which starts at 329 K and $\mathrm{La(IO_3)_3}$ decomposition starts at $\sim$ 750 K. Some phase transitions were observed at $\sim$ 600 and 650 K. For solubility measurements, Harkins and Pearce (2) reported clearly the use of the anhydrous salt, but no information of hydration number of the iodate was reported in the remaining studies (3–8, 11), and composition of the equilibrium solid phase ought to be clarified in further experiments.

TABLE 172. Experimental solubility data of $\mathrm{La(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	$10^3 m_1$ / mol kg^{-1}	Analytical method	Reference
298.2	0.6634	0.6653	Iodometry	(5)
298	0.883	–	Iodometry	(7)
298	0.893	–	Iodometry	(6)
298	0.912	–	Iodometry	(11)
298	0.9127	–	Iodometry	(3)
298	0.939	–	Iodometry	(4)
298.2	1.02	–	Complexometry (La^{3+}), Iodometry (IO_3^-)	(8)
298	1.031	–	Gravimetry (La^{3+}), Iodometry (IO_3^-)	(2)
298	1.07	–	Iodometry	(9)
298.2	2.533	–	Chemical method	(1)
298	2.819	–	Conductivity	(1)
298	–	1.6	Complexometry (La^{3+}), Iodometry (IO_3^-)	(10)

La Mer and Goldman (3) reported 6 solubility values of lanthanum iodate in water at 298 K using 6 different samples of the salt obtained the same way. The arithmetic mean of their 6 solubility values is cited in Table 172. The solubility value in water acidified to pH 5.6–5.7 has been reported by Vasil'ev and Serebrennikov (11). This result agrees very well with that of La Mer and Goldman (3). The pH values of the solutions were not reported, except the paper of Vasil'ev and Serebrennikov as well as Bertha and Choppin (9), but it seems that all of them were slightly acidic. Monk (6) stated that the difference between the results of La Mer and Goldman (3) and of Pearce and Oelke (5) appears to be related to the solute particle size. The sample used in the Monk's solubility measurements (6) showed a solubility of 8.93×10^{-4} mol dm^{-3} at 298 K, but in another paper from the same laboratory it was reported as 8.83×10^{-4} mol dm^{-3}.

The scatter of experimental results collected in Table 172 may be explained not only by the different hydration degree of $\mathrm{La(IO_3)_3}$, different particle size of the crystals being in equilibrium but also by phenomenology of the $\mathrm{La(IO_3)_3}$ precipitation process. It was found by Herak et al. (20) that although the precipitation is fast, sedimentation of the crystals is relatively slow (even at 2.5 h after mixing). Moreover, these authors suggested that soluble polynuclear complexes may be formed in the solution what may increase both ionic concentrations determined by chemical analyses. The experimental results of (20) have been presented in a figure. The solubility value of 0.85×10^{-3} mol dm^{-3} at 293 K (as read from the figure by the evaluators) fits very well to the suggested solubility value at 298 K, given below. However, an origin of this value is not clear and no method of such determination was described therein. Therefore, this result is not taken into account in the further evaluation.

In the first step of this evaluation, the values of Reimbach and Schubert (1) and of Pearce and Oelke (5) were rejected because the former is drastically too high and the latter is too low. The arithmetic mean of the remaining 8 values (2–4, 6–9, 11) is calculated, and the solubility value of 0.96×10^{-3} mol dm^{-3} and the standard deviation is 0.03×10^{-3} was obtained. In a next step, the value of Bertha and Choppin (9) is rejected because their result is higher than the 4σ. The evaluators have calculated again the arithmetic mean of the remaining 7 values, and obtained 0.94×10^{-3} and with the standard deviation of 0.06×10^{-3}. This value is designated as a tentative solubility at 298 K.

The solubility values based on mol kg^{-1} units at 298 K have been reported in (5) and (10). According to the density measurements of saturated solution in (2) and the difference between the solubility results given in molal and molar units in (5) one should not expect a difference of molality and molarity higher than 0.3 %, therefore the results of both 0.6653×10^{-3} (5) and 1.6×10^{-3} mol kg^{-1} (10) are unacceptably different from the tentative value in molar scale and are rejected.

The Ternary System

Solubilities in aqueous electrolyte solutions: Solubility studies for lanthanum iodate in aqueous electrolyte solutions of chlorides, nitrates, sulfates, and iodates have been reported in 4 publications (2–5). A summary of the solubility studies is given in Table 173.

TABLE 173. Summary for solubility studies of $\mathrm{La(IO_3)_3}$ in aqueous solutions of various inorganic salts at 298 K

Ternary system	Concentration of added salt mol dm^{-3}	Solubility range of $La(IO_3)_3$ $10^3 c_1$ / mol dm^{-3}	Reference
$La(IO_3)_3$–NaCl–H_2O	0–0.100	0.9426–1.627	(3)
$La(IO_3)_3$–KCl–H_2O	0–2.00	0.89006–3.7828	(3)
$La(IO_3)_3$–KCl–H_2O	0–3.500	0.6634–3.898 [a]	(5)
$La(IO_3)_3$–$MgCl_2$–H_2O	0–0.005	0.939–1.768	(4)
$La(IO_3)_3$–$MgCl_2$–H_2O	0–0.05	0.9426–1.739	(3)
$La(IO_3)_3$–$MgCl_2$–H_2O	0–3.00	0.6634–4.639 [a]	(5)
$La(IO_3)_3$–$CdCl_2$–H_2O	0–0.05	0.939–1.689	(4)
$La(IO_3)_3$–$LaCl_3$–H_2O	0–0.03333	0.9426–0.9233	(3)
$La(IO_3)_3$–$NaNO_3$–H_2O	0–0.0032	1.0301–4.5657	(2)
$La(IO_3)_3$–$NaNO_3$–H_2O	0–0.100	0.9426–1.654	(3)
$La(IO_3)_3$–KNO_3–H_2O	0–0.1	0.89006–1.679	(3)
$La(IO_3)_3$–$Mg(NO_3)_2$–H_2O	0–0.05	0.9426–1.766	(3)
$La(IO_3)_3$–$La(NO_3)_3$–H_2O	0–0.03333	0.9426–0.9398	(3)
$La(IO_3)_3$–$La(NO_3)_3$–H_2O	0–0.06684	1.0301–1.1195	(2)
$La(IO_3)_3$–$La(NH_4)_2(NO_3)_5$–H_2O	0–0.5249	1.0301–2.8968	(2)
$La(IO_3)_3$–Na_2SO_4–H_2O	0–0.1000	0.9426–3.326	(3)
$La(IO_3)_3$–K_2SO_4–H_2O	0–0.050	0.89006–3.7828	(3)
$La(IO_3)_3$–K_2SO_4–H_2O	0–0.050	0.6634–2.568 [a]	(5)
$La(IO_3)_3$–$MgSO_4$–H_2O	0–0.005	0.939–3.010	(4)
$La(IO_3)_3$–$MgSO_4$–H_2O	0–0.05	0.9426–2.997	(2)
$La(IO_3)_3$–$MgSO_4$–H_2O	0–2.000	0.634–10.558 [a]	(5)
$La(IO_3)_3$–$CdSO_4$–H_2O	0–0.005	0.939–3.205	(4)
$La(IO_3)_3$–$La_2(SO_4)_3$–H_2O	0–0.03333	0.9426–1.830	(3)
$La(IO_3)_3$–$NaIO_3$–H_2O	0–0.0019828	1.0301–0.1468	(2)
$La(IO_3)_3$–KIO_3–H_2O	0–0.0019828	1.0301–0.5632	(2)

[a] $10^3 m_1$ / mol kg^{-1}

The dependences of the solubility of lanthanum iodate on the concentration of the added salts are classified in three groups. In the first group are chlorides, nitrates and sulfates of alkali and alkaline earth metals having no common ion with lanthanum iodate. In this group, the solubility of lanthanum iodate increases monotonously with an increase of concentration of the added salts. In the second group, a decrease of the solubility is observed in case of sodium and potassium iodate addition. In the third group a use of lanthanum nitrate or sulfate as added salt resulted in an initial decrease of the solubility and then increase of the solubility with an increase of the concentration of the added salt. These phenomena were discussed concerning the effect of non-common ions, common univalent and common trivalent ions (2–5).

Monk (6) measured the solubility of lanthanum iodate in aqueous solutions of glycine, alanine and glycylglycine. The solubility increases with increasing the concentration of the amino acids. Amino acids have weakly complexing features for lanthanide ions (19). The solubility results were discussed in terms of the Debye–Hückel theory and dipole–ion modification of the Kirkwood's theory (18). The solubility products have also been calculated but it is not clear how Monk (6) did calculate the products when lanthanum ion concentration was not determined. Monk also reported the solubility in aqueous amino acid solutions containing potassium iodate. The addition of potassium iodate resulted in a decrease of the lanthanum iodate solubility.

Monk (7) has measured the solubilities of the iodate in the mixtures of organic solvent and water. Eight organic solvents were used to prepare the solvent mixtures. Monk stated that the chemical character of the solvents is the major cause of the decrease in solubility with the decrease in the dielectric constant of the solvent mixtures.

Two solubility studies for the aqueous ternary system, $La(IO_3)_3$–HIO_3–H_2O, have been reported in (10) and (12). Vinogradov and Tarasova (10) constructed the isothermal solubility diagram. The crystallization fields of this system in the presence of both components at 298 K have been determined. Two new incongruently soluble double salts: $2La(IO_3)_3 \cdot 3HIO_3 \cdot 6H_2O$ and $3La(IO_3)_3 \cdot 2HIO_3 \cdot 6H_2O$ are formed. Lyalina and Soboleva (12) studied same system at 298 K, and the corresponding phase diagram has been reported. Numerical data of the HIO_3 concentrations were only reported but those of $La(IO_3)_3$ concentration were below their detection limit. The detailed compilation of (12) has already been presented by Miyamoto and Salomon in (13).

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Components:
(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. Rimbach and A. Schubert, Z. Phys. Chem. **67**, 183–200 (1909).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 174. Solubility of $\mathrm{La(IO_3)_3}$ in water at 25.0 °C

Analytical method	γ_1 / (g / 100 cm^3) [a]	$10^3 c_1$ / mol dm^{-3} [b]
Conductivity	0.1871	2.819
Chemical analysis	0.1681	2.553

[a] Observed.

[b] Calculated by the compilers.

The equilibrium solid phase was specified to be $\mathrm{2La(IO_3)_3 \cdot 3H_2O}$. Degree of the salt dissociation in the solution is 76 %.

Auxiliary Information

Method/Apparatus/Procedure:
Conductivity method:
The iodate was mixed with water at room temperature for several hours and the specific conductivity measured. It was corrected to 25.0 °C by use of an experimentally determined temperature coefficient of conductivity. The solubility was calculated from the specific conductivities of the saturated solution and water.
Chemical method:
An excess of the iodate was placed with water in a large flask, and stirred in a thermostat at the selected temperature for 6 h. After settling, lanthanum ion in the saturated solution was determined by oxalate precipitation. The solution containing acidified iodate was added to potassium iodide solution, and the liberated iodine was determined by iodometric titration.

Source and Purity of Materials:
Lanthanum iodate was prepared from solution of an unspecified lanthanum salt and iodic acid. Stoichiometry of $\mathrm{2La(IO_3)_3 \cdot 3H_2O}$ was confirmed; the salt should contain 55.14 % and analyses gave 53.15 and 55.00 % of iodine. Conductivity water was used.

Estimated Error:
Solubility: precision no better than ± 0.5 % (compilers).
Temperature: precision ± 0.1 K.

Components:

(1) Lanthanum iodate; $La(IO_3)_3$; [13870-19-4]
(2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Sodium iodate; $NaIO_3$; [7681-55-2]
or (2) Potassium iodate; KIO_3; [7758-05-6]
or (2) Lanthanum nitrate; $La(NO_3)_3$; [10099-59-9]
or (2) Nitric acid, ammonium lanthanum(3+) salt(5:2:1) (lanthanum diammonium pentanitrate); $La(NH_4)_2(NO_3)_5$; [13566-21-7]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

W. D. Harkins and W. T. Pearce, J. Am. Chem. Soc. **38**, 2679–709 (1916).

Variables:

Concentration of inorganic salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 175. Solubility of $La(IO_3)_3$ in aqueous solutions of $NaNO_3$, $NaIO_3$ and KIO_3 at 25 °C

Added salt	c_2 / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}	γ_1 / g dm^{-3}	ρ / g cm^{-3}
None	—	1.0301	0.6841 (0.6842[b])	0.99825
$NaNO_3$	0.02500	1.3092	0.96901	1.00250
	0.05000	1.4921	0.99040	1.00385
	0.10000	1.7481	1.1603	1.00742
	0.20000	2.0873	1.3852[a]	1.01290
	0.40000	2.4657	1.6363[a]	1.02422
	0.80000	3.2487	2.1559[a]	1.046775
	1.60000	4.3114	2.8611[a]	1.09005
	3.20000	4.5657	3.0299[a]	1.17243
$NaIO_3$	0.0913	0.9572	0.63538	1.00060
	0.4560	0.8507	0.56466	1.00059
	0.9130	0.7658	0.60835	1.00065
	1.8260	0.6016	0.39938	1.00065
	3.6530	0.2973	0.19736	1.00069
	4.5326	0.2017	0.13393	1.00083
	6.7989	0.1468	0.09733	0.00130
KIO_3	0.0990	0.9476	0.6290	1.00030
	0.4957	0.8488	0.5633	1.00027
	0.9914	0.7488	0.4970	1.00030
	1.9828	0.5632	0.3738	1.00031

[a] Calculated by the compilers using IUPAC 1987 atomic weights.
[b] This value is mol kg^{-1} scale.

TABLE 176. Solubility of $\mathrm{La(IO_3)_3}$ in aqueous solutions of $\mathrm{La(NO_3)_3}$ and $\mathrm{La(NH_4)_2(NO_3)_5}$ at 25 °C

Added salt	$3 \times 10^3 c_2$ / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}	γ_1 / g dm^{-3} [a]	ρ / g cm^{-3}
None	—	1.0301	0.6841 (0.6842[b])	0.99825
$\mathrm{La(NO_3)_3}$	2.00	0.8430	0.5595	0.99732
	5.00	0.7968	0.5288	0.99807
	10.00	0.7825	0.5194	0.99859
	50.00	0.8320	0.5522	1.00212
	100.00	0.9362	0.6214	1.00661
	200.52	1.1195	0.7431	1.01533
$\mathrm{La(NH_4)_2(NO_3)_5}$	0.02634	0.9510	0.6311[a]	1.00112
	0.05268	1.0156	0.6740[a]	1.00355
	0.10536	1.1367	0.7543[a]	1.00971
	0.15804	1.2303	0.8164[a]	1.01608
	0.19683	1.3061	0.8667[a]	1.02183
	0.39367	1.6016	1.0628	1.04343
	0.78735	2.0551	1.3638	1.08286
	1.57470	2.8968	1.9224	1.16652

[a] Calculated by the compilers using IUPAC 1987 atomic weights.

[b] This value is mol kg^{-1} scale.

Compositions of the equilibrium solid phases were not specified.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility was determined by rotating crystals of the purified lanthanum iodate salt with water or the solution of the second salt in a glass-stoppered bottle. For each determination, saturation was approached from both undersaturation and supersaturation. The saturated solutions were filtered. The first portion of the solution was rejected and then remainder of the filtrate was used for analyses. The iodate content was determined iodometrically. Two methods were used for analysis of lanthanum. (1) A weighed quantity of the solution was evaporated in a small weighed platinum dish or crucible in an air bath and ignited to the oxide. (2) The sample solution was diluted and the lanthanum precipitated as the oxalate. The oxalic acid was liberated after a decomposition with a strong acid and treated in the usual (but unspecified) way.

Source and Purity of Materials:

Hot dilute solutions containing lanthanum nitrate and potassium iodate were brought together, the former being in excess. The solution was stirred, and then quickly cooled with ice. The precipitated salt was filtered, washed and recrystallized from water. The slightly soluble salt was recrystallized in large evaporating dishes heated on steam baths. Anhydrous $\mathrm{La(IO_3)_3}$ crystals were obtained. No other rare earths were spectrographically detected therein.

Chemically pure sodium nitrate, and sodium and potassium iodates were recrystallized. Crystals of lanthanum diammonium pentanitrate (from Walsbach Burner and Light Co.) were recrystallized. A solution of the salt was prepared and the lanthanum precipitated as oxalate, thoroughly washed and ignited in Pt dish to the oxide. The lanthanum nitrate was then made by adding an excess of the oxide to nitric acid and crystallizing the salt from the clear solution. The salt was recrystallized. Conductivity water was used.

Estimated Error:

Solubility: precision better than ± 0.1 % (compilers).

Temperature: precision ± 0.01 K.

Components:

(1) Lanthanum iodate; $La(IO_3)_3$; [13870-19-4]
(2) Sodium chloride; NaCl; [7647-14-5]
or (2) Sodium nitrate; $NaNO_3$; [7631-99-4]
or (2) Sodium sulfate; Na_2SO_4; [7757-82-6]
or (2) Potassium chloride; KCl; [7447-40-7]
or (2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
or (2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Magnesium nitrate; $Mg(NO_3)_2$; [10377-60-3]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
or (2) Lanthanum chloride; $LaCl_3$; [10099-58-8]
or (2) Lanthanum nitrate; $La(NO_3)_3$; [10099-59-9]
or (2) Lanthanum sulfate; $La_2(SO_4)_3$; [10099-60-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632–45 (1929).

Variables:

Concentration of added salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 177. Solubility and its index of $La(IO_3)_3$ in water at 25 °C

Index[a]	Number of detn.	$10^3 c_1$ / mol dm^{-3}
a	11	0.89006
b	Not given	0.8967
c	Not given	0.8989
d	Not given	0.9000
e	5	0.9426
f	7	0.9482[b]
		(av.) 0.9127

[a] Different lots of $La(IO_3)_3$ salt were used. These indexes were cited in Table 178.

[b] This solubility value was taken from Table 178.

TABLE 178. Solubility of $\mathrm{La(IO_3)_3}$ in aqueous solutions of various added salts at 25 °C

Added salt	c_2 / mol dm^{-3}	No. of detns.	$10^3 c_1$ / mol dm^{-3}	Index[a]
NaCl	0.1	2	1.627	e
$\mathrm{NaNO_3}$	0.1	3	1.654	e
$\mathrm{Na_2SO_4}$	0.05	4	3.326	e
KCl	0.001	2	0.91128	a
	0.005	5	0.97490	a
	0.01	4	1.0322	a
	0.05	4	1.3331	a
	0.1	2	1.5713	c
	0.1	2	1.648	e
	0.2	2	1.8776	a
	0.5	2	2.5641	d
	1.0027	4	3.0547	d
	2.000	4	3.7828	d
$\mathrm{KNO_3}$	0.1	4	1.679	e
$\mathrm{K_2SO_4}$	0.0002	6	0.91666	a
	0.0005	6	0.97463	a
	0.001	4	1.0564	a
	0.002	4	1.2153	a
	0.007	4	1.6852	a
	0.0125	4	2.0272	a
	0.002	4	2.3675	a
	0.05	4	3.1871	b
	0.05	4	3.370	e
$\mathrm{MgCl_2}$	0.05	4	1.739	e
$\mathrm{Mg(NO_3)_2}$	0.05	4	1.766	e
$\mathrm{MgSO_4}$	0.05	2	2.997	e
$\mathrm{LaCl_3}$	0.03333	4	0.9233	e
$\mathrm{La_2(SO_4)_3}$	0.01667	2	1.532	e
	0.03333	2	1.830	e

Added salt	c_2 / mol dm^{-3}	No. of detns.	$\log \sqrt[4]{\frac{K_{\mathrm{sp}}}{K_{0\mathrm{sp}}}}$ [b]	Index[a]
$\mathrm{La(NO_3)_3}$	0	7	0.0009482	f
	0.001667	7	0.0008347	f
	0.003333	4	0.0008101	f
	0.01667	2	0.0008696	f
	0.03333	3	0.0009398	e

[a] The compilers suppose that different lots of $\mathrm{La(IO_3)_3}$ salt shown by the index in Table 177 were used to determine the solubility in various aqueous solutions of added salts.

[b] It is necessary to consider the ratio of the stoichiometric solubility product constants rather than the solubilities; see the original paper for detailed information.

The number of hydrating water molecules in the product was not given.

Auxiliary Information

Method/Apparatus/Procedure:

The products obtained from 6 glass containers for preparation of lanthanum iodate were used for the study of the lanthanum iodate solubility. The saturating column method was used as described in (1). The solute contained in a glass tube was dissolved in water slowly flowing from a bulb container for 24 h. The saturated solution was filtered through a cotton plug and collected for further analysis. The iodate content in the saturated solution was determined by titration with sodium thiosulfate solution standardized with potassium permanganate. Prior to the titration, 3 g of KI and 2 cm^3 of 9 mol dm^{-3} sulfuric acid were added to 25 cm^3 aliquot of the solution. Duplicate determinations for each ternary system were performed.

Source and Purity of Materials:

Lanthanum iodate was prepared by adding cerium-free lanthanum nitrate solution and potassium iodate solution to a large amount of water. The mixture was agitated for 3–4 d. The precipitate was washed by decantation 4 times, and then allowed to digest for 2 weeks in water at 70 °C with stirring. The salt was sedimented in a tall narrow cylinder several times and the finer material remaining at the top each time was rejected. Six products of lanthanum iodate were obtained by the same method as described above.

Chemically pure grade sodium chloride, nitrate and sulfate, and potassium chloride, nitrate and sulfate were twice recrystallized from distilled water, dried in an oven at 120 °C, ground and dried again at 120 °C for 24 h. Magnesium chloride for analysis was from Kahlbam; 1 dm^{-3} of 0.05 mol dm^{-3} $MgCl_2$ solution contained only 0.5 mg of NH_3. Chemically pure grade magnesium sulfate was twice recrystallized, dried in an oven at 50 °C for 3d, and then kept in a desiccator over phosphorus pentoxide for a week. It was kept in glass stoppered bottles sealed with paraffin. The source of magnesium nitrate was not reported. Chemically pure lanthanum chloride, nitrate, and sulfate were twice recrystallized from water, dried at 120 °C, ground and dried again at 120 °C for 24 h. Distilled water was used.

Estimated Error:

Solubility: precision of analysis within 0.1 %.

Temperature: stability ± 0.005 K (as in (2)).

References:

[1] J. N. Brönsted and V. K. La Mer, J. Am. Chem. Soc. **46**, 555 (1924).

[2] V. K. La Mer, C. V. King, and C. F. Mason, J. Am. Chem. Soc. **49**, 363 (1927).

Components:

(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
or (2) Cadmium chloride; $CdCl_2$; [10108-64-2]
or (2) Cadmium sulfate; $CdSO_4$; [10124-36-4]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. B. Friedman and V. K. La Mer, J. Am. Chem. Soc. **53**, 103–5 (1931).

Variables:

Addition of salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 179. Solubility of $\mathrm{La(IO_3)_3}$ in aqueous solutions of magnesium and cadmium salts at 25 °C

Added salt	c_2 / mol dm^{-3}	No. of detn.	$10^3 c_1$ / mol dm^{-3}
None	—	5	0.939 ± 0.0012
$MgCl_2$	0.05	6	1.768 ± 0.007
$MgSO_4$	0.05	4	3.010 ± 0.004
$CdCl_2$	0.05	4	1.689 ± 0.006
$CdSO_4$	0.05	5	3.205 ± 0.009

The number of hydrating water molecules in the solute was not given.

Auxiliary Information

Method/Apparatus/Procedure:

The saturating column method was used as described in (1). The solute contained in a glass tube was dissolved in water slowly flowing from a bulb container for 24 h. The saturated solution was filtered through a cotton plug and collected for further analysis. The iodate content in the saturated solution was determined by iodometric titration using sodium arsenate as the titrant (2).

Source and Purity of Materials:

Lanthanum iodate was prepared by adding cerium-free lanthanum nitrate solution and potassium iodate solution to a large volume of water. The precipitate was washed by decantation 4 times and then allowed to digest for 2 weeks in water at 70 °C with stirring. The salt was sedimented in a tall narrow cylinder several times and the finer material remaining at the top each time was rejected.

Magnesium sulfate was recrystallized twice and dried to the anhydrous state. Chemically pure grade cadmium sulfate (from J. T. Baker) was recrystallized and the monohydrate was obtained. The source of magnesium and cadmium chlorides was not reported. Redistilled water was used.

Estimated Error:

Solubility: standard deviation between 0.1 and 0.4 %.

Temperature: stability ± 0.005 K (as in (2)).

References:

[1] J. N. Brönsted and V. K. La Mer, J. Am. Chem. Soc. **46**, 555 (1924).

[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Lanthanum iodate; $La(IO_3)_3$; [13870-19-4]
(2) Potassium chloride; KCl; [7447-40-7]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
or (2) Magnesium chloride; $MgCl_2$; [7786-30-3]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

J. N. Pearce and W. C. Oelke, J. Phys. Chem. **42**, 95–106 (1938).

Variables:

Concentration of the second salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto, and Cezary Guminski

Experimental Data

TABLE 180. Solubility of $La(IO_3)_3$ in aqueous solution of KCl, K_2SO_4, $MgCl_2$ and $MgSO_4$ at 25 °C

Added salt	m_2 / mol kg^{-1}	10^3c_1 / mol dm^{-3}	γ_1 / g cm^{-3}	10^3m_1 / mol kg^{-1}
None	—	0.6634	0.99749	0.6653
KCl	0.0010	0.684	0.99750	0.686
	0.005	0.737	0.99776	0.739
	0.010	0.786	0.99801	0.789
	0.050	1.014	1.00000	1.019
	0.100	1.195	1.00243	1.202
	0.500	1.919	1.02090	1.952
	1.000	2.452	1.04277	2.531
	2.000	3.167	1.08268	3.368
	3.500	3.898	1.13787	4.329
K_2SO_4	0.0010	0.824	0.99790	0.826
	0.005	1.215	0.99847	1.219
	0.010	1.488	0.99952	1.493
	0.050	2.568	1.00554	2.581
$MgCl_2$	0.0010	0.702	0.99757	0.704
	0.005	0.817	0.99798	0.820
	0.010	0.908	0.99841	0.911
	0.050	1.306	1.00180	1.311
	0.100	1.601	1.00589	1.608
	0.500	2.782	1.03666	2.817
	1.000	3.508	1.07255	3.590
	2.000	4.209	1.13805	4.413
	3.000	4.639	1.19716	4.995
$MgSO_4$	0.0010	0.846	0.99766	0.848
	0.005	1.209	0.99839	1.213

0.010	1.448	0.99921	1.452
0.050	2.384	1.00496	2.390
0.100	3.021	1.01167	3.028
0.500	5.863	1.06116	5.879
1.000	8.198	1.12180	8.227
1.500	9.690	1.17865	9.759
2.000	10.558	1.23098	10.702

Auxiliary Information

Method/Apparatus/Procedure:

A generous portion of the lanthanum iodate paste was washed several times with portions of the solvent solution and finally transferred to duplicate oil-sample bottles with the bulk of the solution. The bottles were clamped to the shaker and rotated. After a minimum of 2 weeks of rotation at the selected temperature, the bottles suspended near the surface of the water in the constant-temperature bath. The suspension was allowed to settle overnight and the clear solution siphoned into similar bottles held in the bath. After at least 3 h more of setting, the supernatant liquid was again transferred to dry flasks. The solutions were allowed to settle until they were optically clear. The analyses of the saturated solutions for iodate ion were made in triplicate, by titration with standard sodium thiosulfate solution. Carefully calibrated pipets and Normax volume burets were used. All titrations were carried out under artificial light.

Source and Purity of Materials:

Lanthanum iodate was prepared, as in (1), by adding cerium-free lanthanum nitrate solution and potassium iodate solution to a large amount of water. The mixture was agitated for 3–4 d. The precipitate was washed by decantaion 4 times, and then allowed to digest for 2 weeks in water at 70 °C with stirring. The salt was sedimented in a tall narrow cylinder several times and the finer material remaining at the top each time was rejected. Potassium chloride and sulfate, and magnesium chloride and sulfate were recrystallized at least twice. Conductivity water was used.

Estimated Error:

Solubility: precision ± 0.2 % or better.

Temperature: stability ± 0.01 K.

References:

[1] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Alanine; $\mathrm{C_3H_7NO_2}$; [6898-94-8]
or (2) Glycine (amicoacetic acid); $\mathrm{C_2H_5NO_2}$; [56-40-6]
or (2) Glycine, *N*-glycyl- (*N*-glycylglycine); $\mathrm{C_4H_8N_2O_3}$; [556-50-3]
(3) Potassium iodate; $\mathrm{KIO_3}$; [7758-05-6]
(4) Water; $\mathrm{H_2O}$; [7732-18-5]

Original Measurements:

C. B. Monk, Trans. Faraday Soc. **47**, 1233–40 (1951).

Variables:

Concentration of alanine: 0–0.2 mol dm^{-3}
or concentration of glycine: 0–0.21 mol dm^{-3}
or concentration of *N*-glycylglycine: 0–0.09 mol dm^{-3}
or concentration of $\mathrm{KIO_3}$: 0–0.002 mol dm^{-3}
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto, Ryo Miyamoto and Cezary Guminski

Experimental Data

TABLE 181. Solubility of $\mathrm{La(IO_3)_3}$ in aqueous solutions of alanine, glycine, *N*-glycylglycine, and potassium iodate at 25 °C

Amino acid	$10^3 c_3$ / mol dm^{-3}	c_2 / mol dm^{-3}	$10^3 c_1$ / mol dm^{-3}	$-\log K_s^\circ$ [a]
None	0	—	0.893	11.168
Alanine[b]	0	0.06466	1.010	10.965
	0	0.14860	1.183	10.708
	0	0.21160	1.313	10.534
Glycine	0	0.05610	1.007	10.972
	1.024	0.05610	0.753	10.975
	1.775	0.05610	0.589	10.975
	0	0.09660	1.095	10.834
	0.996	0.09660	0.848	10.835
	0	0.21330	1.394	10.443
	1.574	0.21330	1.005	10.446
	1.929	0.21330	0.926	10.448
N-Glycylglycine	0	0.4883	1.063	10.873
	0	0.5804	1.119	10.788
	0	0.6569	1.144	10.749
	0	0.8884	1.300	10.539

[a] $K_s^\circ = [\mathrm{La^{3+}}][\mathrm{IO_3^-}]^3\gamma_+\gamma_-^3$ was calculated from equation (83), where γ is the activity coefficient.

$$\log K_s^\circ = \log([\mathrm{La^{3+}}][\mathrm{IO_3^-}]^3) - 6\left(\frac{78.54}{\epsilon}\right)^{\frac{3}{2}}\left(\frac{\sqrt{I}}{1+\sqrt{I}} - 0.2\,I\right) \tag{83}$$

where K_s° is the activity solubility product, ϵ is the solvent dielectric constant and I is the ionic strength.
[b]Information about both optical activity and structural isomer was not described by the author.

The composition of equilibrium solid phase was not specified.

Auxiliary Information

Method/Apparatus/Procedure:
A modified saturating column method was used as described in (1). A glass bulb and column of the saturating salt were connected by capillary tubing to a second, parallel, arm in which the saturated liquid was collected. Slowly flowing water, containing alanine, glycine, or glycylglycine, was saturated with the iodate in a glass column. The solution was filtered by a capillary and collected for analysis. The apparatus was immersed in a thermostat. A prolonged washing of the sample was applied. The iodate content in the saturated solution withdrawn from the saturators was determined by titration with sodium thiosulfate solution standardized with potassium permanganate solution. Prior to the titration, excess of potassium iodide solution was added to the solution acidified with sulfuric acid. Duplicated determinations were performed.

Source and Purity of Materials:
Lanthanum iodate was prepared according to (2) by adding aqueous potassium iodate solution to aqueous lanthanum chloride solution. The latter was prepared from "Spec-pure" lanthanum oxide. $\mathrm{La(IO_3)_3}$ crystals were increased by prolonged boiling in water. Laboratory grade alanine was recrystallized from aqueous alcohol, and dried to constant weight in a vacuum oven at 80 °C. Analytical reagent grade glycine was dried to constant weight in a vacuum oven at 80 °C. Glycylglycine (from Roche Product) was dried to constant weight in a vacuum oven at 80 °C.

Estimated Error:
Solubility: averaged deviation within ± 0.1 % (assumed by compilers).
Temperature: stability ± 0.03 K.

References:
[1] R. W. Money and C. W. Davies, J. Chem. Soc. **1934**, 440.
[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:

(1) Lanthanum iodate; $La(IO_3)_3$; [13870-19-4]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
or (2) 1-Propanol; C_3H_8O; [71-23-8]
or (2) 1,2-Ethanediol (ethylene glycol); $C_2H_6O_2$; [107-21-1]
or (2) 1,2,3-Propanetriol (glycerol); $C_3H_8O_3$; [56-81-5]
or (2) 2-Propanone (acetone); C_3H_6O; [67-64-1]
or (2) 1,4-Dioxane; $C_4H_8O_2$; [123-91-1]
or (2) Acetic acid, ethyl ester (ethyl acetate); $C_4H_8O_2$; [141-78-6]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

C. B. Monk, J. Chem. Soc. **1951**, 2723–6.

Variables:

Concentration of various organic solvents
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 182. Solubility of $La(IO_3)_3$ in the mixtures of water and organic solvents at 25 °C

Organic solvent	$100w_2$	$100x_2$ [a]	10^4c_1 / mol dm^{-3}	$-\log K_s^\circ$ [b]	$10^{12}K_s^\circ$ [a]
None	—	—	8.83	11.186	6.52
Methanol	4.72	2.71	5.78	11.867	1.36
	9.53	5.59	3.99	12.469	0.340
	14.43	8.66	2.70	13.109	0.0778
Ethanol	3.82	1.53	5.95	11.821	1.51
	7.67	3.15	4.20	12.387	0.410
	11.59	4.88	2.89	12.999	0.100
1-Propanol	4.16	1.28	5.92	11.833	1.47
	8.40	2.68	4.08	12.441	0.362
	12.71	4.18	2.84	13.039	0.0914
1,2-Ethanediol	5.62	1.70	7.50	11.452	3.53
	11.24	3.55	6.69	11.641	2.29
	16.85	5.56	5.91	11.847	1.42
1,2,3-Propanetriol	6.31	1.30	8.22	11.316	4.83
	12.44	2.70	7.89	11.387	4.10
	18.43	4.23	7.61	11.456	3.50
2-Propanone	4.09	1.31	6.01	11.805	1.57
	8.25	2.71	4.28	12.356	0.441
	12.46	4.23	3.01	13.065	0.0861
1,4-Dioxane	2.2	0.46	7.69	11.416	3.84
	4.7	1.00	6.63	11.663	2.17

	9.4	2.08	4.88	12.175	0.668
Ethyl acetate	3.8	0.80	6.55	11.673	2.12
	6.1	1.31	5.59	11.936	1.16

[a] Calculated by the compilers.

[b] $K_{\mathrm{s}}^{\circ} = [\mathrm{La}^{3+}][\mathrm{IO}_3^-]^3\gamma_+\gamma_-^3$ was calculated from equation (84), where γ is the activity coefficient.

$$\log K_{\mathrm{s}}^{\circ} = \log([\mathrm{La}^{3+}][\mathrm{IO}_3^-]^3) - 6\left(\frac{78.54}{\epsilon}\right)^{\frac{3}{2}}\left(\frac{\sqrt{I}}{1+\sqrt{I}} - 0.2\,I\right) \tag{84}$$

where K_{s}° is the activity solubility product, ϵ is the solvent dielectric constant and I is the ionic strength.

The number of hydrating water molecule in the product was not given.

Auxiliary Information

Method/Apparatus/Procedure:

A saturating column method was used; see (1) for details. The iodate content in the saturated solutions was determined by titration with sodium thiosulfate solution standardized with potassium permanganate solution, prior to the titration excess potassium iodate was added and the solution acidified with sulfuric acid. Solutions containing dioxane were corrected for peroxides by back titrations. Densities of the solutions were determined.

Source and Purity of Materials:

Lanthanum iodate was prepared by adding aqueous solutions of potassium iodate and lanthanum chloride to a large volume of water. The lanthanum chloride was prepared from “Spec-Pure” lanthanum oxide (2). Methanol, ethanol, 1-propanol, ethylene glycol, glycerol, acetone, and ethyl acetate were of laboratory grade, except in the cases of acetone and 1,4-dioxane (Anala R).

Estimated Error:

Solubility: precision better than ± 0.5 %.

Temperature: precision ± 0.03 K.

References:

[1] R. W. Money and C. W. Davies, J. Chem. Soc. **1934**, 440.
[2] V. K. La Mer and F. H. Goldman, J. Am. Chem. Soc. **51**, 2632 (1929).

Components:
(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{La(IO_3)_3}$ at 25 $^\circ\mathrm{C}$ in water acidified to pH 5.6–5.7 is given below:

0.605 g dm^{-3}	(authors)
0.912×10^{-3} mol dm^{-3}	(compilers).

The number of hydrating water was not reported, but La:IO_3 mol ratio was found to be 1:3. A formation of $\mathrm{La_2(IO_3)_6}$ as equilibrium solid was suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:
A weighed sample of lanthanum iodate and water were taken in 25 cm^3 pycnometer with a ground glass stopper which was sealed externally with a paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at selected temperature for 10–12 h until saturation (1). Water used for solubility determinations was acidified to pH 5.6–7 before use. The iodate content was determined iodometrically in two 10 cm^3 portions of the saturated solution. The portions were acidified by $\mathrm{H_2SO_4}$, proper amount of KI added and the solutions titrated with 0.01 mol dm^{-3} $\mathrm{Na_2S_2O_3}$ in presence of starch.

Source and Purity of Materials:
Lanthanum iodate was prepared from acidic solution of lanthanum chloride (99.8–99.9 % pure according to (1)) and potassium iodate solution. Lanthanum iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:
Solubility: nothing specified.
Temperature: precision probably ± 0.01 K (authors of (1)).

References:
[1] G. I. Vasil'ev and V. V Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:
(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported the lanthanum and iodate concentrations in the saturated solution at 25.0 °C.

$[\mathrm{La^{3+}}] = (1.00 \pm 0.02) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO_3^-}] = 3.1 \times 10^{-3}, 3.1 \times 10^{-3}, 3.0 \times 10^{-3}$, and 3.1×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility.

$$\text{Solubility of } \mathrm{La(IO_3)_3} = 1.02 \times 10^{-3} \text{ mol dm}^{-3}$$

The activity solubility product was reported by the authors.

$$K_s^\circ = 12 \times 10^{-12} \text{ mol}^4 \text{ dm}^{-12}$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 6.2 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules was reported.

Auxiliary Information

Method/Apparatus/Procedure:
About 10 g of lanthanum iodate and 15 cm^3 of water were placed in a 50 cm^3 Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to 25°C with agitation for 24 h and then placed in a constant temperature cabinet at the selected temperature for several days. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The lanthanum cation was analyzed by EDTA titration with methyl thymol and xylenol orange indicators.

Source and Purity of Materials:
Lanthanum iodate was prepared by dissolving about 10 g lanthanum oxide (99.8 and 99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of (recrystallized reagent grade) KIO_3 was slowly poured, with stirring, into the nitric acid solution of lanthanum. The precipitated iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

Components:

(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{La(IO_3)_3}$ in water at 25 °C is 1.07×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{La(IO_3)_3}$ in water at 25 °C is 1.02×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{La(IO_3)_3}$ at infinite dilution was determined to be 28.9 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{La(IO_3)_3}$ calculated by the authors were 62.7 kJ and -0.114 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{LaIO_3^{2+}}$ ion was 17 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{La(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry was performed. Three or 4 measurements were made of each sample of lanthanum iodate. Heat of solution was determined in a calorimeter.

Source and Purity of Materials:

$\mathrm{La(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{La(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves indicated that there was no water of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{La(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{La(ClO_4)_3}$ solution was adjusted to pH 3.0–3.3. Distilled water was used.

Estimated Error:

Nothing specified.

Components:

(1) Lanthanum iodate; $\mathrm{La(IO_3)_3}$; [13870-19-4]
(2) Iodic acid; HIO_3; [7782-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

E. E. Vinogradov and G. N. Tarasova, Zh. Neorg. Khim. **30**, 1542–7 (1985); Russ. J. Inorg. Chem. (Engl. Transl.) **30**, 879–82 (1985).

Variables:

Concentration of the salt and the acid
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 183. Composition of saturated solutions in the ternary $\mathrm{La(IO_3)_3}$–HIO_3–H_2O system at 25 °C

$\mathrm{La(IO_3)_3}$		HIO_3		H_2O		Solid phase[b]
$100w_1$	$100x_1$[a]	$100w_2$	$100x_2$[a]	$100w_3$	$100x_3$[a]	
0.11[c]	0.003	–	–	99.89	99.997	A
0.10	0.003	0.01	0.001	99.89	99.996	A+B
0.09	0.002	0.01	0.001	99.90	99.997	A+B
0.06	0.002	0.23	0.024	99.71	99.975	B
0.06	0.002	2.87	0.302	97.07	99.696	B
0.05	0.001	4.39	0.468	99.56	95.630	B
< 0.01	< 0.001	5.46	0.588	94.53	99.41	B
< 0.01	< 0.001	8.28	0.92	91.72	99.08	B
< 0.01	< 0.001	8.51	0.94	91.49	99.06	B
< 0.01	< 0.001	12.51	1.44	87.49	98.56	B
< 0.01	< 0.001	14.68	1.73	85.32	98.27	B
< 0.01	< 0.001	16.43	1.97	83.57	98.03	B
< 0.01	< 0.001	19.52	2.42	80.48	97.58	B+C
< 0.01	< 0.001	19.61	2.44	80.39	97.56	B+C
< 0.01	< 0.001	19.53	2.43	80.47	97.57	B+C
< 0.01	< 0.001	25.67	3.42	74.33	96.58	C
< 0.01	< 0.001	30.84	4.37	69.16	95.63	C
< 0.01	< 0.001	34.48	5.11	65.52	94.89	C
< 0.01	< 0.001	39.62	6.30	60.38	93.70	C
< 0.01	< 0.001	45.45	7.86	54.55	92.14	C
< 0.01	< 0.001	46.35	8.13	53.65	91.87	C
< 0.01	< 0.001	49.44	9.10	50.56	90.90	C
< 0.01	< 0.001	56.75	11.85	43.25	88.15	C
< 0.01	< 0.001	58.81	12.76	41.19	87.24	C
< 0.01	< 0.001	65.39	16.21	34.61	83.79	C
< 0.01	< 0.001	70.51	19.67	29.49	80.33	C+D

< 0.01	< 0.001	70.46	19.63	29.54	80.37	C+D
< 0.01	< 0.001	70.51	19.67	29.49	80.33	C+D
< 0.01	< 0.001	70.38	19.57	29.62	80.43	C+D
–	–	75.62[c]	24.11	24.38	75.89	D

[a] Calculated by the compiler.

[b] The solid phases are: A = $La(IO_3)_3 \cdot 3H_2O$; B = $3La(IO_3)_3 \cdot 2HIO_3 \cdot 6H_2O$; C = $2La(IO_3)_3 \cdot 3HIO_3 \cdot 6H_2O$ (eutonic composition); D = HIO_3

[c] For the binary systems the compiler computed the following values:

Solubility of $La(IO_3)_3$ = 1.6×10^{-3} mol kg^{-1}
Solubility of HIO_3 = 17.63 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The experiment was performed in an electrically heated water thermostat. Equilibrium was established at the selected temperature in 18–25 d with continuous stirring. The liquid phases and solid residues were analyzed for the iodate ion by titration with sodium thiosulfate in the presence of sulfuric acid and potassium iodide. Lanthanum ion was determined by complexometric titration in the presence of hexamethylenetetramine using methylthymol blue as indicator. The composition of the solid phases crystallizing in the system were determined by the Schreinemakers' residues method. The samples were identified by X-ray diffraction. Thermal analysis of selected composition samples was also performed.

Source and Purity of Materials:

"Chemically pure" grade iodic acid was used. Lanthanum iodate was obtained from lanthanum oxide and iodic acid.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.1 K.

D. Cerium(III) Iodate

Components:
(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of cerium(III) iodate in water at 298 K have been reported in 7 publications (1–7). The reported solubilities are summarized in Table 184.

TABLE 184. Experimental solubility data of $\mathrm{Ce(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method	Reference
298	0.889	Iodometry	(7)
298	1.15	Iodometry	(4)
298.15	1.963	Iodometry	(5, 6)
298.2	2.190	Chemical analysis	(1)
298.2	2.461	Conductivity	(1)
298.15	2.6045	Iodometry	(3)

The reported solubilities at 298 K are much dispersed between 0.889×10^{-3} and 2.6045×10^{-3} mol dm^{-3}. Although the individual precision limits are very narrow. Chloupek et al. (2, 3) have reported the solubility data in 2 publications. The solubility values reported in (2) are identical with those in (3) but units reported in (2) and (3) are g kg^{-1} and g dm^{-3}, respectively. By comparison with the $\mathrm{La(IO_3)_3}$ saturated solutions the results should not differ more than 0.3% (see the $\mathrm{La(IO_3)_3}$–H_2O system). Unit in the remaining papers (1, 4–6) is molarity. The reported solubility value in water by Miyamoto et al. in (5) and (6) can be treated as a single experimental measurement. Vasil'ev and Serebrennikov (7) measured the solubility in water of pH 5.6–5.7, and Bertha and Choppin (4) have reported the solubility in water of pH 3.25. The pH values of the solvents were not reported by the other investigators (1, 2, 3, 5, 6). Rimbach and Schubert (1), Chloupek et al. (2, 3), and Miyamoto et al. (5, 6) used the dihydrate salt for the solubility measurements. Bertha and Choppin (4) used the anhydrous salt, but the equilibrium solute was not analyzed. Since preparation of $\mathrm{Ce(IO_3)_3 \cdot 2H_2O}$ was performed in vicinity of room temperature we may conclude that the equilibrium solid phase is also the dihydrated salt, however further investigation of the equilibrium solute composition is suggested.

Designation of a tentative solubility value was done by the evaluators by statistical treatment. The arithmetic mean value is calculated from the two values 1.963×10^{-3} (5, 6) and 2.190×10^{-3} mol dm^{-3} (1). The mean of 2.1×10^{-3} mol dm^{-3} is designated as a tentative solubility of cerium(III) iodate in water at 298 K.

The Ternary System

Chloupek et al. (2, 3) studied the solubilities of cerium(III) iodate in aqueous KNO_3, K_2SO_4, $MgSO_4$, and $MgCl_2$ solutions at 298.15 K. The solubility of the iodate increases with an increase of the concentration of each added salt. Miyamoto et al. measured the solubilities of the iodate in 3 organic solvent–water mixtures; methanol (5), ethanol (5) and dimethyl sulfoxide (6) were used as the organic co-solvents. In each system, the logarithm of the solubility of the iodate increases almost linearly with the reciprocal dielectric constant of the solvent mixtures.

The Multicomponent System

Kopyrin and Shokin (8) studied the solubilities of cerium(III) iodate in solutions containing lithium perchlorate and nitrate over a wide range of ionic strength at 298 K. Beside the solubility , the stability constants for Ce^{3+}–NO_3^- complexes were also calculated from the dependence of the solubility on nitrate ion concentration.

References:

1. E. Rimbach and A. Schubert, Z. Phys. Chem. **67**, 183 (1909).
2. J. B. Chloupek, V. Z. Danesh, and B. A. Daneshova, Collect. Czech. Chem. Commun. **4**, 473 (1932).
3. J. B. Chloupek, V. Z. Danesh, and B. A. Daneshova, Chem. Listy **26**, 531 (1932).
4. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
5. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
6. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
7. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).
8. A. A. Kopyrin and V. V. Shokin, Issled. Khim. Tekhnol. Primen. Radioakt. Veshchestv **1**, 77 (1977); Chem. Abstr. **93**, 5511 (1980).

Components:
(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
E. Rimbach and A. Schubert, Z. Phys. Chem. **67**, 183–200 (1909).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 185. Solubility of $\mathrm{Ce(IO_3)_3}$ in water at 25.0 °C

Solubility determination method	γ_1 / (g / 100 cm^3)	$10^3 c_1$ / mol dm^{-3} [a]
Conductivity method	0.1636	2.416
Chemical analysis	0.1456	2.190

[a] Calculated by the compilers.

The equilibrium solid phase was found to be $\mathrm{Ce(IO_3)_3 \cdot 2H_2O}$. Degree of the salt dissociation in the solution is 77 %.

Auxiliary Information

Method/Apparatus/Procedure:
Conductivity method: The iodate was mixed with water at room temperature for several hours and the specific conductivity measured. It was corrected to 25.0 °C by use of an experimentally determined temperature coefficient. The solubility was calculated from the specific conductivities of the saturated solution and water.
Chemical analysis: An excess of the iodate was placed with water in a large flask, and stirred in a thermostat at the selected temperature for 6 h. After setting, cerium was determined in the saturated solution by oxalate precipitation. The solution containing iodate was added to acidified potassium iodide solution, and the liberated iodine was determined by iodometric titration.

Source and Purity of Materials:
Cerium iodate was prepared from solutions of cerium ammonium nitrate solution and iodic acid. Stoichiometry of $\mathrm{Ce(IO_3)_3 \cdot 2H_2O}$ obtained was confirmed; the salt should contain 54.32 mass % of iodine and analyses gave 54.16 and 54.11 mass %. Conductivity water was used.

Estimated Error:
Solubility: precision no better than ± 0.5 % (compilers).
Temperature: precision ± 0.1 K.

Components:

(1) Cerium(III) iodate; $Ce(IO_3)_3$; [24216-72-6]
(2) Potassium nitrate; KNO_3; [7757-79-1]
or (2) Potassium sulfate; K_2SO_4; [7778-80-5]
or (2) Magnesium sulfate; $MgSO_4$; [7487-88-9]
or (2) Magnesium chloride; $MgCl_2$; [7786-30-3]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

J. B. Chloupek, V. Z. Danesh, and B. A. Daneshova, Chem. Listy **26**, 531–3 (1932); Collect. Czech. Chem. Commun. **4**, 473–9 (1932).

Variables:

Concentration of the added salt
One temperature: 298.15 K

Prepared By:

Jitka Eysseltova, Hiroshi Miyamoto, and Cezary Guminski

Experimental Data

TABLE 186. Solubility of $Ce(IO_3)_3$ in aqueous solution of KNO_3, K_2SO_4, $MgCl_2$ and $MgSO_4$ at 25.00 °C

Added salt	m_2 / mol kg^{-1}	10^3c_1 / mol dm^{-3} ^{a}	γ_1 / g dm^{-3} ^{a}	Total ionic strength
None	—	2.6045	1.732	0.01563
KNO_3	0.005	2.7767	1.846	0.02166
	0.01	2.9047	1.932	0.02743
	0.02	3.1540	2.097	0.03892
	0.05	3.6367	2.418	0.07182
	0.10	4.1155	2.736	0.12382
	0.20	4.6922	3.120	0.22820
	0.50	5.9800	3.976	0.53588
K_2SO_4	0.002	3.0271	2.013	0.02416
	0.005	3.5635	2.370	0.03638
	0.010	4.2392	2.819	0.05574
	0.020	5.2785	3.510	0.09167
	0.050	6.8114	4.529	0.19087
	0.100	8.5120	5.660	0.40107
$MgSO_4$	0.002	3.0920	2.056	0.02655
	0.005	3.6716	2.442	0.04203
	0.010	4.3870	2.917	0.06632
	0.020	5.3666	3.568	0.11220
	0.050	7.0610	4.695	0.24237
	0.100	9.0610	6.025	0.45437
$MgCl_2$	0.002	2.813	1.870	0.02288
	0.005	2.9980	1.993	0.03299

0.010	3.2617	2.169	0.04957
0.020	3.6505	2.427	0.08190
0.050	4.4868	2.983	0.17692
0.100	5.3139	3.533	0.33188

[a] mol dm^{-3} and g dm^{-3} units in Chem. Listy and mol kg^{-1} and g kg^{-1} in Collect. Czech. Chem. Commun.

Dihydrate of cerium iodate was the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:

This samples were mixed in grounded glass vessels embedded by paraffin. The vessels were shaken with speed 30 r.p.m. for 48–72 h. The samples were equilibrated in a water thermostat with toluene temperature controller (1). The temperature stability was checked by a Beckmann thermometer and its exact value was established by two especially calibrated thermometers. The equilibrium was reached from both directions overheating and undercooling. After sedimentation of the mixture in the thermostat, the samples were filtered through fine porcelain filter crucibles and the contents of iodate in the solutions were determined by the titration with 0.025 mol dm^{-3} thiosulfate using gravimetrical burets.

Source and Purity of Materials:

CeO_2 (Merck, reagent grade) was taken as starting substance and was transformed to the solution in form of $CeCl_3$ or $Ce(NO_3)_3$. Potassium iodate solution was slowly added to $CeCl_3$ or $Ce(NO_3)_3$ solution and vigorously mixed. The precipitate was decantated 10 times with water, taking special care to remove fine particles. The precipitate was filtered through a Jena filtration funnel and dried at 105 °C. An analysis showed: Ce–21.15 and IO_3–78.70 mass %. Calculated composition for $Ce(IO_3)_3 \cdot 2H_2O$: Ce–21.075 and IO_3–78.925 mass %. Reagent grade KNO_3, K_2SO_4, $MgSO_4$ and $MgCl_2$ from Merck were used. Three times distilled water for conductivity measurements was used.

Estimated Error:

Solubility: precision better than ± 0.1 %.

Temperature: precision ± 0.005 K; accuracy ± 0.02 K.

References:

[1] J. B. Chloupek and V. Z. Danesh, Collect. Czech. Chem. Commun. **4**, 8 (1932).

Components:
(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Ce(IO_3)_3}$ at 25 °C in water acidified to pH 5.6–5.7 is given below:

0.591 g $\mathrm{dm^{-3}}$	(authors)
0.889×10^{-3} mol $\mathrm{dm^{-3}}$	(recalculated by the compilers)

The number of hydrating water molecules was not reported, but Ce:IO_3 ratio was found to be 1:3. A formation of solid $\mathrm{Ce_2(IO_3)_6}$ was suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:
A weighed sample of cerium iodate and water were taken into a 25 $\mathrm{cm^3}$ pycnometer with a ground–glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation. Water used for the solubility determinations was acidified to pH 5.6–5.7. The iodate content was determined iodometrically in two portions of the saturated solution. The solutions were acidified by $\mathrm{H_2SO_4}$, KI excess added and titrated with 0.01 mol $\mathrm{dm^{-3}}$ $\mathrm{Na_2S_2O_3}$ in presence of starch.

Source and Purity of Materials:
Cerium iodate was prepared from acidic solution of cerium chloride (99.8–99.9 % pure according to (1)) and potassium iodate solution. Cerium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:
Solubility: nothing specified.
Temperature: precision probably ± 0.01 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:
(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Ce(IO_3)_3}$ in water at 25 °C is 1.15×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Ce(IO_3)_3}$ in water at 25 °C is 1.37×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Ce(IO_3)_3}$ at infinite dilution was determined to be 28.5 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Ce(IO_3)_3}$ calculated by the authors were 60.8 kJ and -0.109 J mol^{-1} K^{-1}, respectively. The formation constant of $CeIO_3^{2+}$ ion was 16 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
A sample of $\mathrm{Ce(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentrations by iodometry was performed. Three or 4 measurements were made of each sample of cerium iodate. Enthalpy of solution was determined in a calorimeter.

Source and Purity of Materials:
$\mathrm{Ce(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Ce(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$, pH 3.25). Thermal balance curves indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. $\mathrm{Ce(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Ce(ClO_4)_3}$ solution was adjusted to pH 3.0–3.3. Distilled water was used.

Estimated Error:
Nothing specified.

Components:

(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_5O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass%
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 187. Solubility of $\mathrm{Ce(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.963 ± 0.001
Methanol	4.99	2.87	1.227 ± 0.001
	9.95	5.85	0.848 ± 0.0002
	20.00	12.32	0.386 ± 0.0002
	40.04	27.30	0.109 ± 0.0003
Ethanol	5.01	2.02	1.161 ± 0.001
	10.04	4.18	0.754 ± 0.0002
	20.01	8.91	0.338 ± 0.001
	40.03	20.70	0.066 ± 0.0001

[a] Calculated by the compiler.

The equilibrium solid phases were not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The cerium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12h. The temperature was lowered to 25.00 °C and the bottles were continuously rotated for 48 h. After setting in the thermostat at the selected level for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat for 1 h. Aliquots were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Cerium iodate was prepared by dropwise addition of both acidic solution of $Ce(NO_3)_3$ and aqueous KIO_3 solution into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The products were filtered, dried under reduced pressure at room temperature, and stored in dark. The cerium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Ce–19.94; IO_3–74.69; H_2O–5.0 mass %, calculated composition for $Ce(IO_3)_3 \cdot 2H_2O$: Ce–19.99; IO_3–74.87; H_2O–5.1 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$ and redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: precision between ± 0.05 and ± 1.5 %; see above.

Temperature: precision ± 0.05 K.

Components:

(1) Cerium(III) iodate; $\mathrm{Ce(IO_3)_3}$; [24216-72-6]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 188. Solubility of $\mathrm{Ce(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.963 ± 0.001
5.03	1.21	1.495 ± 0.003
10.03	2.51	1.261 ± 0.002
20.09	5.48	0.905 ± 0.001
40.03	13.34	0.428 ± 0.001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to 25.00 °C and the bottles were continuously rotated for 48 h. After settling in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h, aliquots were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Cerium iodate was prepared by a dropwise addition of both acidic solution of cerium nitrate and aqueous solution of potassium iodate. The agreement between the chemically found and the theoretically calculated contents of elements in cerium iodate dihydrate salt were within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemicals Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between 0.1 and 0.5 %; given precisely, in the table above.
Temperature: precision ± 0.05 K.

Components:
(1) Cerium(IV) iodate; $\mathrm{Ce(IO_3)_4}$; [13813-99-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Cezary Guminski, University of Warsaw, Warsaw, Poland, and Hiroshi Miyamoto, Niigata University, Niigata, Japan. September 2001.

Critical Evaluation:

The solubility studies of cerium(IV) iodate in solutions containing nitric acid and alkali metal iodates or iodic acid have been reported in 3 publications (1–3). Shvedov and Musaev (1) measured the solubility of cerium(IV) iodate in solution containing fixed amount of HNO_3 and KIO_3 at 293 K. Fardy and Weaver (2) determined the solubility at 298 K in 3 different solutions of HNO_3 concentration containing iodic acid. Sodium bromate was added to the solutions to inhibit reduction of Ce(IV). Posvol'skii et al. (3) measured the solubilities of cerium(IV) iodate in aqueous $NaIO_3$ solution at an ionic strength 5.6 at 296 K. Nitric acid and perchloric acid were added to adjust the ionic strength and to avoid a hydrolysis process; in some cases an adjustment of the ionic strength was done by using only HNO_3. In all experiment the cerium content in the saturated solution was determined by a radioactive method using ^{144}Ce. The equilibrium solid phase was cerium(IV) iodate monohydrate (3).

The results of all solubility studies are summarized in Table 189.

TABLE 189. Summary for solubility study of $\mathrm{Ce(IO_3)_4}$ in HNO_3 solutions

T / K	Concentration of HNO_3, mol dm^{-3}	Concentration of $NaBrO_3$, mol dm^{-3}	Concentration of HIO_3 or MIO_3, mol dm^{-3}	Solubility of $\mathrm{Ce(IO_3)_4}$, mol dm^{-3}	Reference
298	1	0.02	0.057	7.3×10^{-11}	(2)
298	2	0.02	0.057	8.1×10^{-9}	(2)
298	3	0.02	0.057	3.2×10^{-7}	(2)
293	5	nothing specified	0.23^{a}	2.4×10^{-6}	(1)
296	5.60	nothing specified	0.00170^{b}	2.67×10^{-5}	(3)
296	5.60	nothing specified	0.00162^{b}	3.34×10^{-5}	(3)
296	5.60	nothing specified	0.00147^{b}	4.94×10^{-5}	(3)
296	5.60	nothing specified	0.00123^{b}	8.46×10^{-5}	(3)
296	5.60	nothing specified	0.00101^{b}	1.62×10^{-4}	(3)
296	5.60	nothing specified	0.00078^{b}	5.20×10^{-4}	(3)
298	1	0.02	0	3.1×10^{-4}	(2)
298	2	0.02	0	8.1×10^{-4}	(2)
298	3	0.02	0	1.7×10^{-3}	(2)

[a] The concentration of potassium iodate (M = K).

[b] The concentration of sodium iodate (M = Na).

The results of 3 laboratories may be only qualitatively compared. The respective solubility data from (1), (2) and (3) differ orders of magnitude at almost similar temperature (293–298 K). The solubility of cerium(IV) iodate decreases with an decrease of the HNO_3 concentration and the increase of alkali metal iodate concentration. It is obvious from (3) that nitrate ions complexate cerium(IV) increasing its concentration in the solution. This effect is in a different degree compensated by increase of iodate ion concentration intensifying the $Ce(IO_3)_4$ precipitation.

Fardy and Weaver (2) reported the solubility products of $Ce(IO_3)_4$ of 7.7×10^{-18}–3.4×10^{-12} $mol^5\ dm^{-15}$ at 298 K depending on the HNO_3 concentration (1–3 mol dm^{-3}). Lur'e (4) reported a value of solubility product of $Ce(IO_3)_4$ of 5×10^{-17} $mol^5\ dm^{-15}$ although no composition of the solution, temperature and method of measurement was specified.

Although no any recommendation of the $Ce(IO_3)_4$ solubility may be given the results of (1), (2) and (3) do not seem to be contradictory.

References:

1. V. P. Shvedov and Sh. A. Musaev, Izv. Vyssh. Uchebn. Zaved., Khim. Khim. Tekhnol. **4**, 727 (1961).

2. J. J. Fardy and B. Weaver, Anal. Chem. **41**, 1299 (1969).

3. M. V. Posvol'skii, G. S. Markov, and F. P. Fedorov, Radiokhimiya **16**, 906 (1974); Sov. Radiochem. (Engl. Transl.) **16**, 882 (1974).

4. Yu. Yu. Lur'e, *Spravochnik po Analiticheskoi Khimii (Handbook for Analytical Chemistry)*, (Khimiya, Moskva, 1965), p. 96.

Components:

(1) Cerium(IV) iodate; $\mathrm{Ce(IO_3)_4}$; [13813-99-5]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Potassium iodate; KIO_3; [7758-05-6]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

V. P. Shvedov and Sh. A. Musaev, Izv. Vyssh. Uchebn. Zaved., Khim. Khim. Tekhnol. **4**, 727–33 (1961).

Variables:

Fixed concentration of HNO_3 and KIO_3
One temperature: 293 K

Prepared By:

Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Solubility of $\mathrm{Ce(IO_3)_4}$ in 5 mol dm^{-3} HNO_3 and 0.23 mol dm^{-3} KIO_3 solution at 20 $^\circ\mathrm{C}$ was found to be 2.1×10^{-6} g cm^{-3} or 2.4×10^{-6} mol dm^{-3} (calculated by the compilers).

An analysis of the equilibrium solid phase showed potassium content changing from 0.20 to 2.16 mass % and iodate to cerium ratio changing between 4.15 and 4.60. The solid phase composition depends on the precipitation method, washing reagents concentrations and number of recrystallization steps. Double salts containing cerium and potassium were not found.

Auxiliary Information

Method/Apparatus/Procedure:

Only freshly prepared solutions were used in this investigation. The solubility of $\mathrm{Ce(IO_3)_4}$ was determined by radiometric analysis, however the corresponding details were not specified.

Source and Purity of Materials:

$\mathrm{Ce(IO_3)_4}$ was prepared by 3 methods: mixing (acidified by HNO_3) solutions of (i) KIO_3 with $\mathrm{(NH_4)_2Ce(NO_3)_6}$, (ii) $KBrO_3$ with $\mathrm{Ce(NO_3)_3}$ and KIO_3, (iii) KIO_3 with $\mathrm{Ce(NO_3)_3}$ and $KBrO_3$. The $\mathrm{Ce(IO_3)_4}$ precipitate was washed and separated from the saturated solution by centrifuging.

Estimated Error:

Nothing specified.

Components:

(1) Cerium(IV) iodate; $\mathrm{Ce(IO_3)_4}$; [13813-99-5]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Sodium bromate; $NaBrO_3$; [7789-38-0]
(4) Iodic acid; HIO_3; [7782-68-5]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

J. J. Fardy and B. Weaver, Anal. Chem. **41**, 1299–302 (1969).

Variables:

Concentration of HNO_3: 1–3 mol dm^{-3}
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 190. Solubility of $\mathrm{Ce(IO_3)_4}$ in HNO_3 containing $NaBrO_3$ and HIO_3 at 25 °C

c_2 / mol dm^{-3}	γ_3 / mg cm^{-3}	c_3 / mol dm^{-3}	c_4 / mol dm^{-3}	γ_1 / g dm^{-3}	c_1 / mol dm^{-3}	K_s / mol^5 dm^{-15}
1	3	0.02^a	0.057	6.1×10^{-8} ^{b}	7.3×10^{-11}	—
2	3	0.02^a	0.057	6.8×10^{-6} ^{b}	8.1×10^{-9}	—
3	3	0.02^a	0.057	2.7×10^{-4} ^{b}	3.2×10^{-7}	—
1	3	0.02^a	0	0.263	3.1×10^{-4} ^{c}	7.7×10^{-16} ^{d}
2	3	0.02^a	0	0.68	8.1×10^{-4} ^{c}	8.5×10^{-14} ^{d}
3	3	0.02^a	0	1.41	1.7×10^{-3} ^{c}	3.4×10^{-12} ^{d}

[a] Calculated by the compilers from the γ_3 value.
[b] Calculated by the compilers from the c_1 value.
[c] Calculated by the compilers from the γ_1 value.
[d] Calculated by the authors from K_s = $[\mathrm{Ce^{4+}}][\mathrm{IO_3^-}]^4$ using c_1 value.

Method of activity coefficient estimation for both ions used for K_s calculation is unknown. The equilibrium solid phase was not specified.

Auxiliary Information

Method/Apparatus/Procedure:

The solubilities of $Ce(IO_3)_4$ in various concentrations of HNO_3 were determined by agitating acid–washed precipitates from ^{144}Ce-spiked solutions with HNO_3, and some protective amount of $NaBrO_3$, and assaying the solution. Only freshly prepared solutions were used in this investigation. The solubility of $Ce(IO_3)_4$ was determined by a radiometric analysis, however the corresponding details were not specified.

Source and Purity of Materials:

The precipitation of cerium(IV) iodate from homogeneous solution have been reported by Willard and Yu (1). Cerium(III) nitrate, iodic acid, nitric acid and sodium bromate were of analytical reagent grade (2). Cerium(III) was satisfactorily oxidized to cerium(IV) by bromate. $NaBrO_3$ was added from a stock solution to prescribed mixture of $Ce(NO_3)_3$, HIO_3 and HNO_3. $Ce(IO_3)_4$ precipitate was washed by a solution of iodic acid while stirring. The contents were transfered to a glass-stoppered centrifuge tube, centrifuged and sampled.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 1 K.

References:

[1] H. Willard and S. T. Yu, Anal. Chem. **25**, 1754 (1953).

[2] B. Weaver, Anal. Chem. **40**, 1894 (1968).

Components:

(1) Cerium(IV) iodate; $\mathrm{Ce(IO_3)_4}$; [13813-99-5]
(2) Nitric acid; HNO_3; [7697-37-2]
(3) Perchloric acid; $HClO_4$; [7601-90-3]
(4) Sodium iodate; $NaIO_3$; [7681-55-2]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

M. V. Posvol'skii, G. S. Markov, and F. P. Fedorov, Radiokhimiya **16**, 906–9 (1974); Sov. Radiochem. (Engl. Transl.) **16**, 882–5 (1974).

Variables:

Concentration of the solutions
One temperature: 296 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 191. Solubility of $\mathrm{Ce(IO_3)_4}$ in aqueous solutions containing $NaIO_3$, HNO_3 and $HClO_4$ at ionic strength of 5.6 at 23 °C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	pH	10^3c_4 / mol dm^{-3}	10^3c_1 / mol dm^{-3}
2.72	2.88	−1.65	0.373	0.0059
			0.315	0.0092
			0.310	0.0106
			0.245	0.0190
			0.180	0.0519
			0.116	0.373
			0.0832	1.317
3.68	1.92	−1.50	0.499	0.0156
			0.415	0.0270
			0.328	0.0925
			0.241	0.245
			0.165	0.952
			0.155	1.14
			0.111	2.85
4.64	0.96	−1.47	0.645	0.0173
			0.550	0.0302
			0.457	0.0615
			0.355	0.209
			0.262	0.389
5.60	0	−1.06	1.70	0.0267
			1.62	0.0331
			1.47	0.0494
			1.23	0.0846
			1.01	0.162

0.78	0.520

TABLE 192. Solubility product of $\mathrm{Ce(IO_3)_4}$ in water at 296 K

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	$10^{17}K_\mathrm{s}$ / mol^5 dm^{-15}
2.72	2.88	0.3
3.68	1.92	0.6
4.64	0.96	0.4
5.60	0	4.3

The authors of this paper reported a value of solubility product of $\mathrm{Ce(IO_3)_4}$ of 5×10^{-17} mol^5 dm^{-15} from a handbook (1) where no details of measurements were reported. $\mathrm{Ce(IO_3)_4 \cdot H_2O}$ was found as the equilibrium solid phase.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of $\mathrm{Ce(IO_3)_4}$ was determined in the mother solutions obtained after precipitating and separating the cerium(IV) iodate. Given concentrations of nitric and perchloric acids, and sodium iodate and dichromate were added to solution of cerium(III) nitrate. Then with continuous stirring, a solution of sodium iodate and arsenate was added to the previous solution resulting in precipitation of cerium(IV) iodate. Stirring was continued for 1.5–2.0 h. The precipitate was separated from the solution by filtration through a glass filter G3. To eliminate an error associated with the incomplete oxidation of cerium(III) and partial reduction of the cerium(IV) in the mother solutions, solutions of thorium nitrate and additional amount of sodium iodate were added to the solution after separation of the precipitate of cerium iodate. After stirring for 15 min, the $\mathrm{Th(IO_3)_4}$ precipitate was separated by filtration on a glass filter. The cerium content in the solution was determined by a radiometric method with the use of the isotope ^{144}Ce. The measurements were made through the γ activity of ^{144}Pr.

Source and Purity of Materials:

The preparation of cerium(IV) iodate is described under "Methods/Apparatus/Procedure". Analysis of the precipitate showed that its composition corresponded to cerium(IV) iodate monohydrate.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 1 K.

References:

[1] Yu. Yu. Lur'e, *Spravochnik po Analiticheskoi Khimii*, (Khimiya, Moskva, 1965), p. 96.

Components:
(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Cezary Guminski, University of Warsaw, Warsaw, Poland, and Hiroshi Miyamoto, Niigata University, Niigata, Japan. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of praseodymium iodate in water at 298 K have been reported in 5 publications (1–5). Only the study of Firsching and Paul (1) deals solely with the binary system, and the solubility product of the iodate was also reported. Miyamoto et al. (3, 4) have reported the solubility in water together with the solubilities in aqueous–organic solvent mixtures.

Praseodymium chloride (5), perchlorate (2) and nitrate (1, 3, 4) with alkali metal iodates were used to prepare praseodymium iodate. Bertha and Choppin (2) used the anhydrous iodate and Miyamoto et al. (3, 4) used its dihydrate for the solubility measurements. Firsching and Paul (1), and Vasil'ev and Serebrennikov (5) did not report the number of hydrating waters in the equilibrium solid. One may assume that $\mathrm{Pr(IO_3)_3\cdot 2H_2O}$ phase is in equilibrium with the saturated solution because such salt was obtained during its preparation at very similar temperature (3, 4).

Experimental data of the praseodymium iodate solubility in water are summarized in Table 193.

TABLE 193. Experimental solubility data of $\mathrm{Pr(IO_3)_3}$ in water

T / K	10^3c_1 / mol dm^{-3}	Analytical method	Reference
298	0.798	Iodometry	(5)
298.15	1.098	Iodometry	(3, 4)
298.2	1.11	Spectrophotometry ($\mathrm{Pr^{3+}}$), Iodometry ($\mathrm{IO_3^-}$)	(1)
298	1.13	Iodometry	(2)

The solubility result in water of pH 3.25 reported by Bertha and Choppin (2) is in good agreement with that of Firsching and Paul (1) and Miyamoto et al. (3, 4). The value in water of pH 5.6–5.7 reported by Vasil'ev and Serebrennikov (5) is rejected because this value is distinctly different from other reported data. The arithmetic mean of these 3 reasonable values from (1), (2) and (3, 4) is 1.11×10^{-3} mol dm^{-3}, and standard deviation is 0.02×10^{-3} mol dm^{-3}. The mean value is designated as the recommended solubility at 298 K.

The Ternary System

Miyamoto et al. reported the solubilities of praseodymium iodate in mixtures of organic solvents and water at 298.15 K. Methanol (3), ethanol (3) and dimethyl sulfoxide (4) were used as organic co-solvents, and the concentrations of organic solvents were between 0–40 mass %. In each solvent mixture, the solubility decreases with increasing the concentration of the organic solvent component.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
5. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).

Components:

(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Pr(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 $^\circ\mathrm{C}$ is given below:

0.525 g dm^{-3}	(authors)
0.789×10^{-3} mol dm^{-3}	(recalculated by the compilers).

The number of hydrating water molecules in the solid was not reported, but ratio of praseodymium to iodate was found to be 1:3. A formation of solid $\mathrm{Pr_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of the salt was introduced into water in a 25 cm^3 pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). The water used for solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in two samples of the solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:

Praseodymium iodate was prepared from acidic solution of praseodymium chloride (99.8+ % pure) and potassium iodate solution. The praseodymium iodate precipitate was separated from the solution by filtration and washed with water (1). Distilled water was used.

Estimated Error:

Solubility: nothing specified.

Temperature: precision ± 0.01 K; (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:

(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported the praseodymium and iodate concentrations in the saturated solution at 25.0 °C:

[Pr^{3+}] = $(1.10 \pm 0.02) \times 10^{-3}$ mol dm^{-3}
[$\mathrm{IO_3^-}$] = 3.4×10^{-3}, 3.4×10^{-3}, 3.3×10^{-3}, and 3.4×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Pr(IO_3)_3} = 1.11 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_{\mathrm{s}}^{\circ} = 1.7 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 6.6 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of praseodymium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Another three slightly modified procedures were also used. The saturated solution was centrifuged to remove any suspended particles.

The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The praseodymium cation was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium praseodymium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

The praseodymium nitrate was prepared by dissolving about 10 g praseodymium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized potassium iodate (reagent grade) was slowly poured, with stirring, into the nitric acid solution of praseodymium. The precipitated praseodymium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision better than ± 2 %.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:

(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Pr(IO_3)_3}$ in water at 25 °C is 1.13×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Pr(IO_3)_3}$ in water at 25 °C is 1.28×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Pr(IO_3)_3}$ at infinite dilution was determined to be 28.0 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Pr(IO_3)_3}$ calculated by the authors were 62.2 kJ and -0.113 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{PrIO_3^{2+}}$ ion was 15 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Pr(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made for each sample of the praseodymium iodate solution. The enthalpy of solution of the salt was measured in a calorimeter.

Source and Purity of Materials:

$\mathrm{Pr(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Pr(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Pr(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Pr(ClO_4)_3}$ solution was adjusted to pH 3.0–3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:
(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:
Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 194. Solubility of $\mathrm{Pr(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.098 ± 0.001
Methanol	4.99	2.87	0.745 ± 0.002
	9.95	5.85	0.494 ± 0.0003
	20.00	12.32	0.229 ± 0.0004
	40.04	27.30	0.049 ± 0.0004
Ethanol	5.01	2.02	0.701 ± 0.0002
	10.04	4.18	0.453 ± 0.004
	20.01	8.91	0.178 ± 0.001
	40.03	20.70	0.035 ± 0.001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The praseodymium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept further in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

The praseodymium iodate was prepared by dropwise addition of both the acidic solution of praseodymium nitrate and the aqueous solution of KIO_3 into the KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The praseodymium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Pr–20.02; IO_3–74.43; H_2O–5.0 mass %; calculated for $\mathrm{Pr(IO_3)_3 \cdot 2H_2O}$: Pr–20.08; IO_3–74.78; H_2O–5.1 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 3 %.
Temperature: precision ± 0.05 K.

Components:
(1) Praseodymium iodate; $\mathrm{Pr(IO_3)_3}$; [14945-15-4]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:
Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 195. Solubility of $\mathrm{Pr(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.098 ± 0.001
5.03	1.21	0.881 ± 0.006
10.03	2.51	0.733 ± 0.003
20.09	5.48	0.493 ± 0.001
40.03	13.34	0.226 ± 0.008

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified praseodymium iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 $^\circ\mathrm{C}$ for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Praseodymium iodate was prepared by a dropwise addition of both the acidic solution of praseodymium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution.
The agreement between the analytically found and the theoretically calculated contents of elements in $\mathrm{Pr(IO_3)_3 \cdot 2H_2O}$ were within ± 0.5 %. Analytical grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS $\mathrm{cm^{-1}}$ was used.

Estimated Error:

Solubility: standard deviation between 0.1 and 3 %.
Temperature: precision ± 0.05 K.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, Ryo Miyamoto, Hirosaki University, Hirosaki, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of neodymium iodate in water have been reported in 11 publications (1–11). Three solubility studies (1, 2, 11) deal solely with the water–$\mathrm{Nd(IO_3)_3}$ binary system. The solubility in water with the solubilities in aqueous–organic solvent mixtures has been reported in 2 publications (6, 7). The solubility in pure water was given as one point on phase diagrams of 5 ternary systems of $\mathrm{Nd(IO_3)_3}$ with different salts (3–5, 8). The pH values of the saturated $\mathrm{Nd(IO_3)_3}$ solutions have been reported in the studies of Bertha and Chopin (2) and of Vasil'ev and Serebrennikov (9). Pruitt and Rickard (10) reported the solubility in water as one point on the solubility curve for neodymium iodate in nitric acid, but these results were presented in a graphical form. All solubility measurements were performed at 298 K and their results in water are summarized in the following Table 196.

TABLE 196. Experimental solubility data of $\mathrm{Nd(IO_3)_3}$ in water at 298 K

10^3c_1 / mol dm^{-3}	10^3m_1 / mol kg^{-1}	Equilibrium solid phase	Analytical method	Reference
0.734	—	—	Iodometry	(9)
1.01	—	—	Iodometry, Spectrophotometry ($\mathrm{Nd^{3+}}$)	(1)
1.03	—	$\mathrm{Nd(IO_3)_3}$ [a]	Iodometry	(2)
1.043	1.046	$\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$	Iodometry	(11)
1.044	—	$\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$ [a]	Iodometry	(6, 7)
—	1.047	$\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$	Iodometry, Spectrophotometry ($\mathrm{Nd^{3+}}$)	(8)
—	2.2	$\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$	Iodometry, Spectrophotometry ($\mathrm{Nd^{3+}}$)	(3, 4)
—	2.2	$\mathrm{Nd(IO_3)_3}$	Iodometry, Spectrophotometry ($\mathrm{Nd^{3+}}$)	(5)

[a] Salt used for the study

The solubility of neodymium iodate expressed in mol dm^{-3} units was reported 6 publications (1, 2, 6, 7, 9, 11). The result of Vasil'ev and Serebrennikov (9) is distinctly different from those obtained by other investigators (1, 2, 6, 7, 11). Their value of 0.73×10^{-3} mol dm^{-3} is not taken into account. The mean value of remaining 5 results (1, 2, 6, 7, 11) is 1.03×10^{-3} mol dm^{-3} with the standard deviation of 0.01×10^{-3} mol dm^{-3}. The mean value is designated as a recommended solubility at 298 K. Three groups of investigators (3–5, 8, 11) reported the solubility based on mol kg^{-1} units and these results are very divergent. The solubilities of crystalline neodymium iodate dihydrate and amorphous neodymium anhydride in water at 298 K were studied by Miyamoto and Arimoto (11). Anhydrous iodate of an amorphous type was prepared by thermal dehydration of the iodate dihydrate. Composition of the starting material and the solid phases equilibrated with the solutions were checked by powder X-ray diffraction analysis and IR measurements. The solubility measurements were performed using supersaturation method. During the measurements the amorphous iodate recrystallized into crystalline iodate dihydrate. Thus, Miyamoto and Arimoto stated that the solubility result obtained agreed very well with that of the crystalline iodate dihydrate. The result of Tarasova's group (3–5) is twice larger than from other investigators (8, 11). This difference can probably be attributed to the use of a contaminated iodate by (3–5) as a source material or the equilibrium was not achieved. The mean of two reasonable values of (8) and (11) is 1.05×10^{-3} mol kg^{-1}, and this is designated as the recommended solubility at 298 K.

The Ternary System

Miyamoto et al. (6, 7) have reported the solubilities of neodymium iodate in mixtures of organic solvents and water. Methanol (6), ethanol (6) and dimethyl sulfoxide (7) were use as the co-solvents. The logarithm of the solubility of neodymium iodate decreases almost linearly with the reciprocal dielectric constant of the mixtures as it is expected from the Born equation. Pruitt et al. (10) graphically reported the dependence of the solubility of iodate on the concentration of nitric acid. The solubility increased with the increase of nitric acid concentration.

The solubilities in the ternary $\mathrm{Nd(IO_3)_3}$–$\mathrm{KIO_3}$–$\mathrm{H_2O}$ (3), $\mathrm{Nd(IO_3)_3}$–$\mathrm{RbIO_3}$–$\mathrm{H_2O}$ (3), $\mathrm{Nd(IO_3)_3}$–$\mathrm{LiIO_3}$–$\mathrm{H_2O}$ (4) and $\mathrm{Nd(IO_3)_3}$–$\mathrm{HIO_3}$–$\mathrm{H_2O}$ (5) systems have been studied. The corresponding compilations have been previously reported in (12). Shklovskaya et al. (8) have studied the solubilities in the aqueous ternary $\mathrm{Nd(IO_3)_3}$–$\mathrm{Al(IO_3)_3}$–$\mathrm{H_2O}$ system, and the corresponding compilation is presented in this volume together with the aluminum iodate solubility.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2412 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613, (1969).
3. G. N. Tarasova, E. E. Vinogradov, and I. B. Kudinov, Zh. Neorg. Khim. **26**, 2841 (1981); Russ. J. Inorg. Chem. (Engl. Transl.) **26**, 1520 (1981).
4. E. E. Vinogradov and G. N. Tarasova, Zh. Neorg. Khim. **27**, 269 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 153 (1982).
5. G. N. Tarasova, E. E. Vinogradov, and I. B. Kudinov, Zh. Neorg. Khim, **27**, 505 (1982); Russ. J. Inorg. Chem. (Engl. Transl.) **27**, 287 (1982).

6. H. Miyamoto, H. Shimura, and K, Sasaki, J. Solution Chem. **14**, 485 (1985).
7. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
8. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, L. A. Kolobkova, and N. G. Dubovenko, Zh. Neorg. Khim. **33**, 3217 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 1859 (1988).
9. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).
10. M. E. Pruitt, R. R. Rickard, and E. I. Myatt, Anal. Chem. **34**, 283 (1962).
11. H. Miyamoto and K. Arimoto, Nippon Kagaku Kaishi **1993**, 623; 5th International Symposium on Solubility Phenomena, Abstract PS4, (Moscow, 1992).
12. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, UK, 1987), p. 331, 404, 442, 487.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]
(2) Potassium iodate; KIO_3; [7758-05-6]
(3) Nitric acid; HNO_3; [7697-37-2]
(4) Water; H_2O; [7732-18-5]

Original Measurements:

M. E. Pruitt, R. R. Rickard, and E. I. Myatt, Anal. Chem. **34**, 283–7 (1962).

Variables:

Concentration of KIO_3 and HNO_3
One temperature: 298 K

Prepared By:

Cezary Guminski and Hiroshi Miyamoto

Experimental Data

Solubility curves for neodymium iodate in nitric acid containing potassium iodate solutions at 25°C were reported in a figure. The solubility data were read from the figure by the compilers.

TABLE 197. Solubility of $\mathrm{Nd(IO_3)_3}$ in HNO_3–KIO_3 solutions at 25°C

c_2 / mol dm^{-3}	c_3 / mol dm^{-3}	γ_1 / mg cm^{-3}	10^3c_1 / mol dm^{-3}
0	0	0.26	0.39
0.05	0	0.15	0.22
0.1	0	0.11	0.16
0.2	0	0.04	0.06
0.3	0	< 0.03	< 0.05
0	0.05	3.49	5.22
0.05	0.05	0.13	0.19
0.1	0.05	0.07	0.10
0.2	0.05	< 0.03	< 0.05
0	0.1	4.38	6.55
0.05	0.1	0.16	0.24
0.1	0.1	0.08	0.12
0.2	0.1	< 0.03	< 0.05
0	0.2	5.79	8.66
0.05	0.2	0.52	0.78
0.1	0.2	0.12	0.18
0.2	0.2	< 0.03	< 0.05
0.05	0.4	1.80	2.71
0.1	0.4	0.30	0.45
0.2	0.4	0.13	0.19
0.3	0.4	< 0.03	< 0.05
0.05	0.7	2.36	3.53
0.1	0.7	1.32	1.97
0.2	0.7	0.18	0.27
0.3	0.7	0.03	0.05

0.05	1.0	4.01	5.99
0.1	1.0	3.04	4.54
0.2	1.0	0.51	0.76
0.3	1.0	0.09	0.13

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of neodymium iodate was determined by the same procedure as it was described for the $Y(IO_3)_3$–KIO_3–HNO_3–H_2O system. Neodymium iodate that was tagged with ^{147}Nd was used. The solubility data of neodymium iodate were obtained by adding the tagged neodymium iodate to solution of nitric acid and iodate ion of various concentrations. The solutions were allowed to remain in contact with an excess of the iodate precipitate until a change in solubility was no longer observed. This equilibrium concentration was determined by counting aliquots of the supernatant tagged neodymium iodate. It was evaporated on a watch glass. The ^{147}Nd was counted by a well-type gamma scintillation counter. Specific activity of the neodymium iodate was previously determined. The counting rate of the supernatant liquid was a direct measurement of solubility of neodymium iodate in each solution.

Source and Purity of Materials:

Neodymium iodate was prepared by adding reagent grade potassium iodate to a neutral solution of neodymium nitrate. ^{147}Nd was used as a tracer. The product was amorphous, and the neodymium iodate was a mixture of $Nd(OH)(IO_3)_2 \cdot xH_2O$ and $Nd(OH)_2IO_3 \cdot xH_2O$.

Estimated Error:

Nothing specified.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Nd(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 $^\circ\mathrm{C}$ is given below:

0.491 g dm^{-3}	(authors)
0.734×10^{-3} mol dm^{-3}	(recalculated by the compilers)

The number of hydrating water molecules in the solid was not reported, but molar ratio of neodymium to iodate was found to be 1:3. A formation of solid $\mathrm{Nd_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of neodymium iodate was introduced into water in a 25 cm^3 pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation. The water used for solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in two samples of the solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:

Neodymium iodate was prepared from acidic solution of neodymium chloride (99.8+ % pure) and potassium iodate solution. The neodymium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ±0.01 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported neodymium and iodate concentrations in the saturated solution at 25.0 $^\circ\mathrm{C}$:

[$\mathrm{Nd^{3+}}$] = $(0.98 \pm 0.02) \times 10^{-3}$ mol dm^{-3}
[$\mathrm{IO_3^-}$] = 3.1×10^{-3}, 3.1×10^{-3}, 3.0×10^{-3}, and 3.1×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Nd(IO_3)_3} = 1.01 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_{\mathrm{s}}^{\circ} = 1.2 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution $(I = 6.2 \times 10^{-3})$ and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of neodymium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 $^\circ\mathrm{C}$ for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. Slightly different procedures were used for other 3 determinations. The neodymium cation was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium neodymium iodate solution was compared with spectra of the known solution of its pure neodymium perchlorate.

Source and Purity of Materials:

The $\mathrm{Nd(IO_3)_3}$ was prepared by dissolving about 10 g neodymium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of $\mathrm{HNO_3}$. A solution containing a slight stoichiometric excess of recrystallized $\mathrm{KIO_3}$ (reagent grade) was slowly poured, with stirring, into the nitric acid solution of neodymium. The precipitated neodymium iodate was separated and then agitated with 15 cm^3 of water at 80 $^\circ\mathrm{C}$ for several hours to remove any soluble species.

Estimated Error:

Solubility: precision better than ± 2 %.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Nd(IO_3)_3}$ in water at 25 °C is 1.03×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Nd(IO_3)_3}$ in water at 25 °C is 9.56×10^{-12} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Nd(IO_3)_3}$ at infinite dilution was determined to be 26.8 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Nd(IO_3)_3}$ calculated by the authors were 63.0 kJ and -0.121 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{NdIO_3^{2+}}$ ion was 14 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of $\mathrm{Nd(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made for each sample of the neodymium iodate. The enthalpy of solution of $\mathrm{Nd(IO_3)_3}$ was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Nd(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Nd(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. $\mathrm{Nd(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Nd(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentrations of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 198. Solubility of $\mathrm{Nd(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.044 ± 0.001
Methanol	4.99	2.87	0.708 ± 0.0004
	9.95	5.65	0.480 ± 0.001
	20.00	12.32	0.221 ± 0.001
	40.04	27.30	0.046 ± 0.001
Ethanol	5.01	2.02	0.670 ± 0.002
	10.04	4.18	0.443 ± 0.0003
	20.01	8.91	0.173 ± 0.0002
	40.03	20.70	0.029 ± 0.0002

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified neodymium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

The neodymium iodate was prepared by dropwise addition of both the acidic solution of neodymium nitrate and the aqueous solution of KIO_3 into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The products were filtered, dried under reduced pressure at room temperature, and stored in the dark. The neodymium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product: Nd–20.40; IO_3–74.50; H_2O–5.4 mass %, calculated composition for $\mathrm{Nd(IO_3)_3 \cdot 2H_2O}$: Nd–20.46; IO_3–74.43; H_2O–5.1 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and then distilled. The product was treated with Ca metal and distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: precision between ± 0.1 and ± 2 %.

Temperature: precision ± 0.05 K.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 199. Solubility of $\mathrm{Nd(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.044 ± 0.001
5.03	1.21	0.855 ± 0.001
10.03	2.51	0.693 ± 0.001
20.09	5.48	0.476 ± 0.001
40.03	13.34	0.209 ± 0.001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Neodymium iodate was prepared by a dropwise addition of both the acidic solution of neodymium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated composition values for the dihydrate salt was within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co.) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 0.5 %.

Temperature: precision ± 0.05 K.

Components:

(1) Neodymium iodate; $\mathrm{Nd(IO_3)_3}$; [14732-16-2]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto and K. Arimoto, Nippon Kagaku Kaishi **1993**, 623; 5th International Symposium on Solubility Phenomena, Moscow (1992), Abstract PS4.

Variables:

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of crystalline neodymium iodate in water at 298.15 K is given as 1.043×10^{-3} mol dm^{-3} and 1.048×10^{-3} mol kg^{-1}. The equilibrium solid phase was the dihydrate.

Anhydrous neodymium iodate of an amorphous type was prepared by thermal dehydration of the purified neodymium iodate dihydrate. During the equilibrium process the amorphous sample recrystallized to the crystalline iodate. Thus, the obtained solubility value agreed well with the value for the crystalline iodate.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of crystalline neodymium iodate in water was determined by using supersaturation method. The prepared neodymium iodate dihydrate and water were placed into glass stoppered bottles. The bottles were rotated in a thermostat at 27 °C for 24 h and then the temperature was slowly lowered to the selected temperature and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically. The equilibrium method for the amorphous sample was similar to that of the crystalline dihydrate. The sample solutions were shaken in the thermostat at the selected temperature (25 °C) for a long time. After shaking for 25 h at 25 °C, the sampling of the solution was performed at the interval of 25–50 h and the iodate concentration of the solutions was determined iodometrically. The composition of the equilibrium solid was identified by chemical analysis, and powder X-ray diffraction pattern and IR spectra measurements.

Source and Purity of Materials:

The crystalline neodymium iodate dihydrate was prepared by a dropwise addition of both the acidic solution of neodymium nitrate and aqueous solution of potassium iodate into KNO_3 solution. The agreement between the analytically found and the theoretically calculated values for the crystalline dihydrate salt was within ± 0.5 %. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used. Anhydrous neodymium iodate of an amorphous type was prepared by thermal dehydration of the crystalline iodate dihydrate. The iodate dihydrate was heated at about 260 °C for about 30 h under about 0.1 Pa (1×10^{-3} torr) in vacuum line. The iodate content of the product was determined iodometrically. The chemically found value of the product agrees with the theoretical value calculated as the iodate anhydride. The amorphous iodate anhydride was identified by powder X-ray diffraction and IR spectra.

Estimated Error:

Solubility: precision ± 0.1 % (authors of (1)).
Temperature: precision ± 0.02 K.

References:

[1] H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).

Components:

(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of samarium iodate in water at 298 K have been reported in 6 publications (1–6). Papers of Firsching and Paul (1) and Bertha and Choppin (2) deal solely with the binary system, and the authors also reported the solubility product. The solubility in water with that in aqueous–organic solvent mixtures has been reported in 2 publications (4, 5), and the reported values in water have been treated as single measurements. A study of Shklovskaya et al. (3) deal with the ternary system and the solubility of samarium iodate in the binary system was given as one point at the corresponding phase diagram. Vasil'ev and Serebrennikov (6) have reported the solubility of the iodate in water acidified to pH 5.6–5.7 and this solubility value is smaller than the other values. No information of the reagent used to adjust the pH was given therein. Experimental data for samarium iodate in water are summarized in Table 200.

Shklovskaya et al. (3) have reported that the solid phase equilibrated with the saturated solution was samarium iodate dihydrate [56491-69-1] and it is the equilibrium solid phase assumed in this evaluation. Miyamoto et al. (4, 5) used the dihydrate salt for solubility measurements. Bertha and Chopppin (2) used iodate which had no water of crystallization.

TABLE 200. Experimental solubility data of $\mathrm{Sm(IO_3)_3}$ in water at 298 K

T / K	10^3c_1 / mol dm^{-3}	10^3m_1 / mol kg^{-1}	Analytical method	Reference
298.15	0.771		Iodometry	(4, 5)
298.2	0.83		Iodometry (IO_3^-), Spectrophotometry (Sm^{3+})	(1)
298	0.86		Iodometry	(2)
298		0.34	Complexometry (Sm^{3+})	(3), compiled in (7)
298	0.701		Iodometry	(6)

The arithmetic mean of the solubility results from (1), (2) and (4, 5) in water at 298 K is 0.81×10^{-3} mol dm^{-3}, and the standard deviation is 0.03×10^{-3} mol dm^{-3}. The mean value is designated as a tentative value. The solubility based on mol kg^{-1} scale is 0.34×10^{-3} (3), but this value is in complete disagreement with the other data and may be rejected.

The Ternary System

Miyamoto et al. (4, 5) reported the solubilities of samarium iodate in mixtures of organic solvents and water. Methanol (4), ethanol (4) and dimethyl sulfoxide (5) were used as co-solvents. In every case the logarithm of the solubility decreases almost linearly with the reciprocal dielectric constant of the solvent mixtures, as expected from the Born's equation.

Shklovskaya et al. (3) studied the solubility in the ternary $\mathrm{Sm(IO_3)_3}$–$\mathrm{LiIO_3}$–$\mathrm{H_2O}$ system. The compilation of this paper was reported in (7).

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. R. M. Shklovskaya, S. M. Arkhipov, B. I. Kidyarov, and L. P. Zherdienko, Zh. Neorg. Khim. **22**, 1139 (1977); Russ. J. Inorg. Chem. (Engl. Transl.) **22**, 624 (1977).
4. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
5. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
6. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).
7. H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*, IUPAC Solubility Data Series, Vol. 30, (Pergamon Press, Oxford, 1987), p. 312.

Components:

(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Sm(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 °C is given below:

0.473 g dm^{-3}	(authors)
0.701×10^{-3} mol dm^{-3}	(recalculated by the compilers)

The number of hydrating water molecules in the solid was not reported but ratio of samarium to iodate was found to be 1:3. A formation of solid $\mathrm{Sm_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of samarium iodate was introduced into water in a 25 cm^3 pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). Water used for the solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in 2 samples of the solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:

Samarium iodate was prepared from acidic solution of samarium chloride (99.8+ % pure) and potassium iodate solution. Samarium iodate precipitate was separated from the solution by filtration and further washed with water. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.01 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:

(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported samarium and iodate concentrations in the saturated solution at 25.0 °C:

$[\mathrm{Sm}^{3+}] = (0.81 \pm 0.05) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO}_3^-]$ = 2.6×10^{-3}, 2.6×10^{-3}, 2.5×10^{-3}, and 2.6×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Sm(IO_3)_3} = 0.83 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 6.4 \times 10^{-12} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 5.2 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of samarium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of IO_3^- determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The samarium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium samarium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

The samarium nitrate was prepared by dissolving about 10 g samarium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of samarium. The precipitated samarium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision better than ± 2 %.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:

(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Sm(IO_3)_3}$ in water at 25 °C is 0.86×10^{-3} mol $\mathrm{dm^{-3}}$. The solubility product, K_s°, of $\mathrm{Sm(IO_3)_3}$ in water at 25 °C is 5.07×10^{-12} $\mathrm{mol^4\ dm^{-12}}$. The cKs for a standard state of infinite dilution was calculated by employing mean activity coefficient from Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Sm(IO_3)_3}$ at infinite dilution was determined to be 24.3 kJ $\mathrm{mol^{-1}}$. The free energy and entropy of the solution for $\mathrm{Sm(IO_3)_3}$ calculated by the authors were 64.5 kJ and -0.136 J $\mathrm{mol^{-1}\ K^{-1}}$, respectively. The formation constant of $\mathrm{SmIO_3^{2+}}$ ion was 13 $\mathrm{mol^{-1}\ dm^3}$ at ionic strength of 0.10 mol $\mathrm{dm^{-3}}$.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Sm(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected level for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements were made with each sample of the samarium iodate. The heat of solution of the salt in water was measured in a calorimeter.

Source and Purity of Materials:

$\mathrm{Sm(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Sm(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$, to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of its iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Sm(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol $\mathrm{dm^{-3}}$ $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Sm(ClO_4)_3}$ solution was adjusted to pH 3.0–3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.

Temperature: nothing specified.

Components:

(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 201. Solubility of $\mathrm{Sm(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	0.771 ± 0.002
Methanol	4.99	2.87	0.532 ± 0.001
	9.95	5.85	0.356 ± 0.0001
	20.00	12.32	0.165 ± 0.0004
	40.04	27.30	0.034 ± 0.0001
Ethanol	5.01	2.02	0.484 ± 0.001
	10.04	4.18	0.322 ± 0.003
	20.01	8.91	0.129 ± 0.001
	40.03	20.70	0.024 ± 0.0003

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified samarium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

The samarium iodate was prepared by dropwise addition of both acidic solution of samarium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The samarium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Sm–21.09; IO_3–73.35; H_2O–5.0 mass %; calculated for $\mathrm{Sm(IO_3)_3 \cdot 2H_2O}$: Sm–21.14; IO_3–73.79; H_2O–5.1 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was collected. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.3 and ± 3 %.
Temperature: precision ± 0.05 K.

Components:
(1) Samarium iodate; $\mathrm{Sm(IO_3)_3}$; [14732-17-3]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:
Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 202. Solubility of $\mathrm{Sm(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	0.771 ± 0.002
5.03	1.21	0.631 ± 0.001
10.03	2.51	0.518 ± 0.001
20.09	5.48	0.359 ± 0.001
40.03	13.34	0.164 ± 0.0003

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:
The supersaturation method was used. The purified samarium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:
Samarium iodate was prepared by a dropwise addition of both the acidic solution of samarium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated contents of elements in $\mathrm{Sm(IO_3)_3 \cdot 2H_2O}$ were within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:
Solubility: standard deviation between ± 0.2 and ± 0.3 %.
Temperature: precision ± 0.05 K.

I. Europium Iodate

Components:

(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. September 2001.

Critical Evaluation:

The Binary System

Data for the solubility of europium iodate in water at 298 K have been reported in 5 publications (1–5). The study of Firsching and Paul (2) deals solely with the binary system, and the solubility product was also reported. The solubility in pure water with the solubilities in aqueous potassium chloride solution have been reported by Laurie and Monk (1). Measurements in aqueous–organic solvent mixtures have been performed by Miyamoto et al. (4, 5), and the value reported in water was treated as a single measurement. The solubility of the iodate in water at pH 3.25 has also been determined by Bertha and Choppin (3).

Europium iodate was prepared from solutions of europium nitrate or perchlorate and alkali metal iodates. Bertha and Chopin (3) used the anhydrate salt for solubility measurements, and Miyamoto et al. (4, 5) used the dihydrate. Laurie and Monk (1) as well as Firsching and Paul (2) did not report the number of hydrating water molecules in the solute. Since the synthesis of $\mathrm{Eu(IO_3)_3 \cdot 2H_2O}$ and the determination of its solubility were carried out in (4, 5) at similar temperatures, we may conclude that $\mathrm{Eu(IO_3)_3 \cdot 2H_2O}$ is the equilibrium solid phase.

Experimental solubility data of europium iodate in water are summarized in Table 203.

TABLE 203. Experimental solubility data of $\mathrm{Eu(IO_3)_3}$ in water

T / K	10^3c_1 / mol dm^{-3}	Analytical method	Reference
298.15	0.782	Iodometry	(4, 5)
298.2	0.80	Iodometry (IO_3^-), Spectrophotometry (Eu^{3+})	(2)
298	0.80	Iodometry	(3)
298	0.817	Radiometry	(1)

The solubility result in water of pH 3.25 reported by (3) is in good agreement with that of (2), (4, 5) and (1). The arithmetic mean of these 4 reasonable values at 298 K is 0.80×10^{-3} mol dm^{-3}, and the standard deviation is 0.01×10^{-3} mol dm^{-3}. The mean value is designated as the recommended solubility at 298 K.

The Ternary System

Laurie and Monk (1) measured the solubilities of the iodate in aqueous solution of potassium chloride at 298 K. The solubility increases with the increasing KCl concentration. The results were used to determine the activity coefficients of the iodate.

Miyamoto et al. (4, 5) measured the solubilities of the iodate in mixtures of organic solvents and water at 298.15 K. Methanol (4), ethanol (4) and dimethyl sulfoxide (5) were used as organic co-solvents. The solubilities decreased with increasing the organic solvent concentration, and confirmed the theory of the solvation effect.

References:

1. S. H. Laurie and C. B. Monk, J. Chem. Soc. **1963**, 3343.
2. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
3. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
4. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
5. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).

Components:

(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]
(2) Potassium chloride; KCl; [7447-40-7]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

S. H. Laurie and C. B. Monk, J. Chem. Soc. **1963**, 3343–7.

Variables:

Concentration of KCl: 0–0.051 mol $\mathrm{dm^{-3}}$
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 204. Solubility of $\mathrm{Eu(IO_3)_3}$ in aqueous solution of potassium chloride at 25 °C

10^2c_2 / mol $\mathrm{dm^{-3}}$	10^3c_1 / mol $\mathrm{dm^{-3}}$	$-\log K_\mathrm{s}^\circ$	$10^{12}K_\mathrm{s}^\circ$ / $\mathrm{mol^4}$ $\mathrm{dm^{-12}}$ [a]
0	0.817	11.305	4.95
2.046	1.076	11.265	5.43
3.580	1.180	11.270	5.37
5.115	1.252	11.295	5.07

[a] Calculated by the compiler from $\log K_\mathrm{s}$.

The activity solubility product, K_s°, was calculated from the following equation:

$$K_s^\circ = \log([\mathrm{Eu}^{3+}][\mathrm{IO}_3^-]^3) - \frac{6\sqrt{I}}{1+\sqrt{I}} - 0.2\,I \qquad (85)$$

The authors reported the activity solubility product:

$$K_s^\circ = 5.188 \times 10^{-12}\ \mathrm{mol}^4\ \mathrm{dm}^{-12}.$$

Auxiliary Information

Method/Apparatus/Procedure:

A set of glass tubes of about 50 cm^3 capacity, and having narrow necks fitted with rubber bungs, was used. A few grams of $\mathrm{Eu(IO_3)_3}$ crystals were put into each tube, washed a few times with saturating solutions, then rocked overnight in a shaker built into a water-bath kept at the selected temperature. Samples were drawn off with a warm pipet with a plastic tube containing a small plug of cotton wool. For the radioactivity assays, 5 cm^3 sample of the solution was used with addition of a little sodium ethylenediaminetetraacetate. The counting equipment was a scintillation counter fitted with a well-type crystal. The reference solubilities in water were found by thiosulfate titration.

Source and Purity of Materials:

Europium iodate crystals were made from the oxide (from Johnson, Mattey and Co.) dissolved in a slight excess of warm perchloric acid. $^{152}\mathrm{Eu}$ and $^{154}\mathrm{Eu}$ were then added as chloride followed by the hot saturated potassium iodate. Part of the resulting flocculent precipitate was dissolved in water in a large dish and was evaporated on a water-bath, till crystals were formed. Further portions of the hot saturated solution were added and good crystals were obtained. “AnalaR” grade potassium chloride was used.

Estimated Error:

Radioactivity: accuracy ± 0.2 %.
Solubility product: average deviation ± 6 %.
Temperature: precision ± 0.1 K.

Components:

(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported europium and iodate concentrations in the saturated solution at 25.0 °C:

[$\mathrm{Eu^{3+}}$] = $(0.80 \pm 0.06) \times 10^{-3}$ mol dm^{-3}
[$\mathrm{IO_3^-}$] = 2.4×10^{-3}, 2.4×10^{-3}, 2.3×10^{-3}, and 2.4×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value:

$$\text{Solubility of } \mathrm{Eu(IO_3)_3} = 0.80 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product reported by the authors was

$$K_s^\circ = 4.8 \times 10^{-12} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution $(I = 4.8 \times 10^{-3})$ and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium salt was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of europium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of $\mathrm{IO_3^-}$ determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The europium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium europium iodate solution was compared with spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

Europium nitrate was prepared by dissolving about 10 g europium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of $\mathrm{HNO_3}$. A solution containing a slight stoichiometric excess of recrystallized $\mathrm{KIO_3}$ (reagent grade) was slowly poured, with stirring, into the nitric acid solution of europium. The precipitated europium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision ± 2 % or less.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Eu(IO_3)_3}$ in water at 25 °C is 0.80×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Eu(IO_3)_3}$ in water at 25 °C is 3.89×10^{-12} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Eu(IO_3)_3}$ at infinite dilution was determined to be 21.3 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Eu(IO_3)_3}$ calculated by the authors were 65.2 kJ and -0.147 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{EuIO_3^{2+}}$ ion was 14 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of $\mathrm{Eu(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis of iodate ion concentration by iodometry. Three or 4 measurements of the concentration were carried out for each sample of the europium iodate. The enthalpy of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Eu(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Eu(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to its oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. $\mathrm{Eu(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Eu(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 205. Solubility of $\mathrm{Eu(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	0.782 ± 0.003
Methanol	4.99	2.87	0.539 ± 0.002
	9.95	5.85	0.367 ± 0.001
	20.00	12.32	0.169 ± 0.0004
	40.04	27.30	0.036 ± 0.0002
Ethanol	5.01	2.02	0.502 ± 0.0003
	10.04	4.18	0.321 ± 0.0001
	20.01	8.91	0.135 ± 0.0004
	40.03	20.70	0.024 ± 0.0004

[a] Calculated by the compiler.

Composition of the equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The europium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously were rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Europium iodate was prepared by dropwise addition of both acidic solution of europium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The europium iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Eu–21.22; IO_3–73.56; H_2O–5.2 mass %, calculated for $\mathrm{Eu(IO_3)_3 \cdot 2H_2O}$: Eu–21.32; IO_3–73.62; H_2O–5.0 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: precision between 0.3 and 1.5 %.

Temperature: precision ± 0.05 K.

Components:

(1) Europium iodate; $\mathrm{Eu(IO_3)_3}$; [14732-18-4]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 206. Solubility of $\mathrm{Eu(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	0.782 ± 0.003
5.03	1.21	0.647 ± 0.003
10.03	2.51	0.534 ± 0.0002
20.09	5.48	0.369 ± 0.0002
40.03	13.34	0.166 ± 0.001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Europium iodate was prepared by a dropwise addition of both the acidic solution of europium nitrate and aqueous solution of potassium iodate into the $\mathrm{KNO_3}$ solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.5 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.04% and ± 0.6%.

Temperature: precision ± 0.05 K.

J. Gadolinium Iodate

Components:
(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility of gadolinium iodate in water at 298 K have been reported in 5 publications (1–5). The solubility in water together with the solubilities in aqueous–organic solvent mixtures has been reported in 2 publications (4, 5), and the reported value in water, in these studies, has been treated as a single independent measurement.

Gadolinium iodate was prepared from gadolinium chloride (1), perchlorate (3) or nitrate (2, 4, 5), and alkali metal iodates. The iodate used for the solubility measurements by Bertha and Choppin (3), and Miyamoto et al. (4, 5) was the anhydrate and the dihydrate, respectively. Vasil'ev and Serebrennikov (1), as well as Firsching and Paul (2) did not report the number of hydrating water molecules in the equilibrium solid phase. Since the $\mathrm{Gd(IO_3)_3 \cdot 2H_2O}$ preparation and its solubility determinations were performed at very similar temperatures one may deduce that the solute at 298 K has the composition $\mathrm{Gd(IO_3)_3 \cdot 2H_2O}$.

The solubility result in water of pH 3.25 reported by (3) is in good agreement with that of (2) and (4, 5). The value of solubility in (1) is rejected because this value is distinctly different from other data (2–5). Experimental data of gadolinium iodate solubility in water at 298 K are summarized in Table 207.

TABLE 207. Experimental solubility data of $\mathrm{Gd(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method	Reference
298	0.488	Iodometry	(1)
298	0.89	Iodometry (IO_3^-)	(3)
298.15	0.893	Iodometry	(4, 5)
298.2	0.90	Iodometry (IO_3^-), Spectrophotometry (Gd^{3+})	(2)

The arithmetic mean of the 3 reasonable values from (2–5) is 0.90×10^{-3} mol dm^{-3}, and standard deviation is 0.01×10^{-3} mol dm^{-3}. The mean result is designated as a recommended solubility at 298 K.

The Ternary System

Miyamoto et al. (4, 5) measured the solubilities of gadolinium iodate in mixtures of organic solvents and water at 298.15 K. Methanol (4), ethanol (4) and dimethyl sulfoxide (5) were used as the organic co-solvents. In each mixture, the solubility of the iodate decreased with an increase of the concentration of the organic component in the mixed solvent.

References:

1. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).
2. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **23**, 2414 (1966).
3. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
4. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
5. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).

Components:

(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Gd(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 $^\circ\mathrm{C}$ is given below:

0.333 g $\mathrm{dm^{-3}}$	(authors)
0.488×10^{-3} mol $\mathrm{dm^{-3}}$	(recalculated by the compilers)

The number of hydrating water molecules in the solute was not reported but ratio of gadolinium to iodate was found to be 1:3. A formation of solid $\mathrm{Gd_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of gadolinium iodate was introduced into water in a 25 $\mathrm{cm^3}$ pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). Water used for the solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in 2 samples of the saturated solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodate and sulfuric acid.

Source and Purity of Materials:

Gadolinium iodate was prepared from acidic solution of gadolinium chloride (99.8+ % pure) and potassium iodate solution. Gadolinium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.1 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:

(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The authors reported gadolinium and iodate concentrations in the saturated solution at 25.0 °C:

$[\mathrm{Gd}^{3+}] = (0.91 \pm 0.05) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO_3^-}] = 2.7 \times 10^{-3}$, 2.7×10^{-3}, 2.6×10^{-3}, and 2.7×10^{-3} mol dm^{-3}
(from different procedures).

The compiler computes the following value of the solubility:

$$\text{Solubility of } \mathrm{Gd(IO_3)_3} = 0.90 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 7.4 \times 10^{-12} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 5.4 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the solute was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of gadolinium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in the constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of $\mathrm{IO_3^-}$ determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The gadolinium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium gadolinium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

Gadolinium iodate was prepared by dissolving about 10 g gadolinium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of gadolinium. The precipitated gadolinium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision better than ± 2 %.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Gd(IO_3)_3}$ in water at 25 °C is 0.83×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Gd(IO_3)_3}$ in water at 25 °C is 4.29×10^{-12} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Gd(IO_3)_3}$ at infinite dilution was determined to be 17.6 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Gd(IO_3)_3}$ calculated by the authors were 64.9 kJ and -0.156 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{GdIO_3^{2+}}$ ion was 15 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of $\mathrm{Gd(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were carried out for each sample of gadolinium iodate. The heat of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Gd(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Gd(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of its iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until pH of 3.25 was obtained. $\mathrm{Gd(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Gd(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3 was obtained. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 %.
Temperature: nothing specified.

Components:

(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; $\mathrm{C_2H_6O}$; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 208. Solubility of $\mathrm{Gd(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	0.893 ± 0.002
Methanol	5.03	2.89	0.622 ± 0.002
	9.88	5.81	0.431 ± 0.002
	19.76	12.16	0.205 ± 0.0004
	40.11	27.35	0.042 ± 0.001
Ethanol	4.84	1.95	0.611 ± 0.001
	10.03	4.18	0.390 ± 0.001
	19.84	8.82	0.162 ± 0.0004
	40.01	20.69	0.030 ± 0.001

[a] Calculated by the compiler.

The equilibrium solid phases was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified gadolinium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept further in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Gadolinium iodate was prepared by dropwise addition of both the acidic solution of gadolinium nitrate and the aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The gadolinium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Gd–21.79; IO_3–72.92; H_2O–5.0 mass %; calculated for $\mathrm{Gd(IO_3)_3 \cdot 2H_2O}$: Gd–21.90; IO_3–73.09; H_2O–5.0 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was collected. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was collected. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.2 and ± 3 %.
Temperature: precision ± 0.05 K.

Components:
(1) Gadolinium iodate; $\mathrm{Gd(IO_3)_3}$; [14732-19-5]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:
Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 209. Solubility of $\mathrm{Gd(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	0.893 ± 0.002
5.03	1.21	0.747 ± 0.002
10.03	2.51	0.614 ± 0.0004
20.09	5.48	0.422 ± 0.0004
40.03	13.34	0.185 ± 0.0003

Composition of the equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:
The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:
Gadolinium iodate was prepared by a dropwise addition of both the acidic solution of gadolinium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytical grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:
Solubility: standard deviation between ± 0.06% and ± 0.2 %.
Temperature: precision ± 0.05 K.

Components:
(1) Terbium iodate; $\mathrm{Tb(IO_3)_3}$; [14732-20-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubilities of terbium iodate in water have been reported in 4 publications (1–4). One study (1) deals solely with the binary system, and the solubility product was also determined therein. The solubility in pure water with the solubilities in aqueous–organic solvent mixtures has been reported in 2 publications (3, 4), and the reported value for pure water in these studies was treated as a single independent measurement. The solubility of the iodate in water of pH 3.25 has also been reported by Bertha and Choppin (2).

Terbium iodate was prepared from a solution of terbium nitrate or perchlorate and alkali metal iodates. Bertha and Choppin (2) used the anhydrous iodate for the solubility measurements, and Miyamoto et al. (3, 4) used the dihydrate. Firsching and Paul (1) did not report the amount of hydrating water molecule in the salt used.

Experimental data of terbium iodate solubility in water at 298 K are summarized in Table 210.

TABLE 210. Experimental solubility data of $\mathrm{Tb(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method	Reference
298.2	0.91	Spectrophotometry ($\mathrm{Tb^{3+}}$), Iodometry ($\mathrm{IO_3^-}$)	(1)
298	0.93	Iodometry	(2)
298.15	0.933	Iodometry	(3, 4)

We assume that the equilibrium solid is $\mathrm{Tb(IO_3)_3 \cdot 2H_2O}$ because this salt was obtained and investigated in (3, 4) at very similar temperatures.

The arithmetic mean of the 3 reported values (1–4) is 0.93×10^{-3} mol dm^{-3}, and the standard deviation is 0.01×10^{-3}. The mean result is designated as a recommended solubility at 298 K.

The Ternary System

Miyamoto et al. (3, 4) have reported the solubilities of terbium iodate in mixtures of organic solvents and water at 298.15 K. Methanol (3), ethanol (3) and dimethyl sulfoxide (4) were used as the organic co-solvents, the concentration of organic components was between 0–40 mass $\%$. In each mixture, the solubility decreases with increasing the concentration of each organic component.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).

Components:

(1) Terbium iodate; $\mathrm{Tb(IO_3)_3}$; [14732-20-8]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported terbium and iodate concentrations in the saturated solution at 25.0 °C:

[Tb^{3+}] = $(0.90 \pm 0.01) \times 10^{-3}$ mol dm^{-3}

[IO_3^-] = 2.8×10^{-3}, 2.8×10^{-3}, 2.7×10^{-3}, and 2.8×10^{-3} mol dm^{-3} (from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Tb(IO_3)_3} = 0.91 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_\mathrm{s}^\circ = 7.7 \times 10^{-12} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution $(I = 5.6 \times 10^{-3})$ and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of terbium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of IO_3^- determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The terbium cation was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium terbium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

Terbium iodate was prepared by dissolving about 10 g of terbium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of terbium. The precipitated terbium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision better than ± 2 %.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:

(1) Terbium iodate; $\mathrm{Tb(IO_3)_3}$; [14732-20-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Tb(IO_3)_3}$ in water at 25 °C is 0.93×10^{-3} mol $\mathrm{dm^{-3}}$. The solubility product, K_s°, of $\mathrm{Tb(IO_3)_3}$ in water at 25 °C is 6.44×10^{-12} $\mathrm{mol^4\ dm^{-12}}$. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Tb(IO_3)_3}$ at infinite dilution was determined to be 17.1 kJ $\mathrm{mol^{-1}}$. The free energy and entropy of solution for $\mathrm{Tb(IO_3)_3}$ calculated by the authors were 63.4 kJ and -0.157 J $\mathrm{mol^{-1}}$ $\mathrm{K^{-1}}$, respectively. The formation constant of $\mathrm{TbIO_3^{2+}}$ ion was 16 $\mathrm{mol^{-1}}$ $\mathrm{dm^3}$ at ionic strength of 0.10 mol $\mathrm{dm^{-3}}$.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Tb(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were further kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the cocentration were made with each sample of the terbium iodate. The heat of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:

$\mathrm{Tb(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Tb(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Tb(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol $\mathrm{dm^{-3}}$ $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Tb(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Terbium iodate; $\mathrm{Tb(IO_3)_3}$; [14732-20-8]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 211. Solubility of $\mathrm{Tb(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	0.933 ± 0.001
Methanol	5.03	2.89	0.637 ± 0.001
	9.88	5.81	0.435 ± 0.0001
	19.76	12.76	0.204 ± 0.0002
	40.11	27.35	0.040 ± 0.0002
Ethanol	4.84	1.95	0.613 ± 0.001
	10.03	4.18	0.390 ± 0.001
	19.84	8.82	0.167 ± 0.002
	40.01	20.69	0.031 ± 0.001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The terbium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Terbium iodate was prepared by dropwise addition of both acidic solution of terbium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The products were filtered, dried under reduced pressure at room temperature, and stored in the dark. The terbium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analyses of the product showed: Tb–22.06; IO_3–73.13; H_2O–5.2 mass %, while calculated composition for $\mathrm{Tb(IO_3)_3 \cdot 2H_2O}$ was Tb–22.08; IO_3–72.91; H_2O–5.0 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours, and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 3 %.
Temperature: precision ± 0.05 K.

Components:

(1) Terbium iodate; $\mathrm{Tb(IO_3)_3}$; [14732-20-8]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 212. Solubility of $\mathrm{Tb(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	0.933 ± 0.001
5.03	1.21	0.776 ± 0.001
10.03	2.51	0.627 ± 0.001
20.09	5.48	0.427 ± 0.001
40.03	13.34	0.177 ± 0.001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Terbium iodate was prepared by a dropwise addition of both the acidic solution of terbium nitrate and aqueous solution of potassium iodate into $\mathrm{KNO_3}$ solution. The agreement between the chemically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytical grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure three times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 0.5%.

Temperature: precision ± 0.05 K.

L. Dysprosium Iodate

Components:

(1) Dysprosium iodate; $\mathrm{Dy(IO_3)_3}$; [14732-21-9]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubilities of dysprosium iodate in water at 298 K have been reported in 6 publications (1–6). One study (2) deals solely with the binary system, and the solubility product was also determined therein. The solubility in pure water with that in aqueous–organic solvent mixtures has been reported in 2 publications (4, 5), and the reported value for pure water was treated as a single independent measurement. The solubility of the iodate in water of pH 5.6 (1) and of pH 3.25 (3) has also been reported. The study (6) deals with the ternary $\mathrm{Dy(IO_3)_3}$–$LiIO_3$–H_2O system, and the solubility based on mass % units was given as one point on the phase diagram.

Dysprosium chloride (1), perchlorate (3) or nitrate (2, 4, 5) with alkali metal iodates or iodic acid were used to prepare the dysprosium iodate. Some preparations (2, 4, 5) were performed in acidic solutions.

Shklovskaya et al. (6) have reported that the solid phase equilibrated with the saturated solution at 298 K was the tetrahydrate salt. Miyamoto et al. (4, 5) used the dihydrate for the solubility measurements, and Bertha and Choppin (3) used the anhydrous salt. Vasil'ev and Serebrennikov (1), and Firsching and Paul (2) did not report the number of hydrating water molecules in the salt used. By an analogy to other lanthanide iodates (4, 5), it seems that dysprosium iodate dihydrate is the equilibrium solid phase at 298 K, because the solute was also prepared in vicinity of this temperature.

The experimental data of dysprosium iodate solubility in water at 298 K are summarized in Table 213.

TABLE 213. Experimental solubility data of $\mathrm{Dy(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	$10^3 m_1$ / mol kg^{-1}	Analytical method	Reference
298	0.589	—	Iodometry	(1)
298	—	1.00	Complexometry (Dy^{3+}) Iodometry (IO_3^-)	(6)
298.15	1.010	—	Iodometry	(4), (5)
298.2	1.01	—	Spectrophotometry (Dy^{3+}) Iodometry (IO_3^-)	(2)
298	1.03	—	Iodometry	(3)

The solubility result in water of pH 3.25 reported by Bertha and Choppin (3) agrees acceptably with that of Firsching and Paul (2) and Miyamoto et al. (4, 5). The solubility at pH 5.6–5.7 reported by Vasil'ev and Serebrennikov (1) is distinctly different from all other determinations, and this value is rejected. The arithmetic mean of the 3 reasonable results (2–5) is 1.02×10^{-3} mol dm^{-3} with the standard deviation of 0.01×10^{-3} mol dm^{-3}. The mean value is designated as the recommended solubility at 298 K.

The result based on mol kg^{-1} units has been reported by Shklovskaya et al. (6), and the value of 1.00×10^{-3} mol kg^{-1} is designated as the tentative solubility at 298 K. By a comparison with the solubility of $\mathrm{La(IO_3)_3}$ expressed in mol kg^{-1} and that in mol dm^{-3} units, the difference between the solubilities of $\mathrm{Dy(IO_3)_3}$ expressed by these units should be smaller than 1 %.

The Ternary System

Miyamoto et al. (4, 5) measured the solubilities of dysprosium iodate in mixtures of organic solvents and water at 298 K. Methanol (4), ethanol (4), and dimethyl sulfoxide (5) were used as organic co-solvents, and concentration of the organic solvents was changed between 0–40 mass %. In each mixture, the solubility of the iodate decreased with the increase of the concentration of the organic component.

Shklovskaya et al. (6) studied the solubilities in the ternary $\mathrm{Dy(IO_3)_3}$–$\mathrm{LiIO_3}$–$\mathrm{H_2O}$ system at 298 K. A restricted solid solution based on dysprosium iodate is formed as the equilibrium solid phase in these conditions.

References:

1. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).
2. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
3. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
4. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
5. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
6. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, N. L. Svetlichnaya, and V. A. Kuzina, Zh. Neorg. Khim. **34**, 258 (1989); Russ. J. Inorg. Chem. (Engl. Transl.) **34**, 146 (1989).

Components:

(1) Dysprosium iodate; $Dy(IO_3)_3$; [14732-21-9]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $Dy(IO_3)_3$ in water acidified to pH 5.6–5.7 at 25 °C is given below:

0.405 g dm^{-3}	(authors)
0.589×10^{-3} mol dm^{-3}	(recalculated by the compilers).

The number of hydrating water molecules in the solid was not reported. Molar ratio of dysprosium to iodate was found to be 1:3. A formation of solid $Dy_2(IO_3)_6$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:

A weighed sample of dysprosium iodate and water was taken into a 25 cm^3 pycnometer with a ground–glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). The distilled water used for the solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in 2 samples of the saturated solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid. $Na_2S_2O_3$ in presence of starch.

Source and Purity of Materials:

Dysprosium iodate was prepared from acidic solution of dysprosium chloride (99.8+ % pure) and potassium iodate solution. The dysprosium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision ± 0.1 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:
(1) Dysprosium iodate; $\mathrm{Dy(IO_3)_3}$; [14732-21-9]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported dysprosium and iodate concentrations in the saturated solution at 25.0 °C:

$[\mathrm{Dy}^{3+}] = (1.00 \pm 0.06) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO_3^-}] = 3.1 \times 10^{-3}$, 3.1×10^{-3}, 3.0×10^{-3}, and 3.1×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Dy(IO_3)_3} = 1.01 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 1.2 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution $(I = 6.2 \times 10^{-3})$ and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:
About 10 g of dysprosium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air–cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of $\mathrm{IO_3^-}$ determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The dysprosium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium dysprosium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:
Dysprosium nitrate was prepared by dissolving about 10 g dysprosium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of dysprosium. The precipitated dysprosium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision of 2 % or less.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:

(1) Dysprosium iodate; $\mathrm{Dy(IO_3)_3}$; [14732-21-9]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Dy(IO_3)_3}$ in water at 25 °C is 1.03×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Dy(IO_3)_3}$ in water at 25 °C is 9.03×10^{-12} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of solution of $\mathrm{Dy(IO_3)_3}$ at infinite dilution was determined to be 15.1 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Dy(IO_3)_3}$ calculated by the authors were 63.9 kJ and -0.162 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{DyIO_3^{2+}}$ ion was 17 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Dy(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the iodate content were made for each sample of the dysprosium iodate. The heat of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:

$\mathrm{Dy(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Dy(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Dy(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Dy(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.

Temperature: nothing specified.

Components:

(1) Dysprosium iodate; $\mathrm{Dy(IO_3)_3}$; [14732-21-9]

(2) Methanol; CH_4O; [67-56-1]

or (2) Ethanol; C_2H_6O; [64-17-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 214. Solubility of $\mathrm{Dy(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.010 ± 0.001
Methanol	5.03	2.89	0.688 ± 0.002
	9.88	5.81	0.472 ± 0.003
	19.76	12.16	0.219 ± 0.0002
	40.11	27.35	0.048 ± 0.001
Ethanol	4.84	1.95	0.668 ± 0.001
	10.03	4.18	0.414 ± 0.0004
	19.84	8.82	0.175 ± 0.0004
	40.01	20.69	0.034 ± 0.0002

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified dysprosium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Dysprosium iodate was prepared by dropwise addition of both acidic solution of dysprosium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The products were filtered, dried under reduced pressure at room temperature, and stored in the dark. The dysprosium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Dy–22.45; IO_3–72.34; H_2O–5.2 mass %, while calculated for $Dy(IO_3)_3 \cdot 2H_2O$: Dy–22.47; IO_3–72.55; H_2O–5.0 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 2 %.

Temperature: precision ± 0.05 K.

Components:

(1) Dysprosium iodate; $\mathrm{Dy(IO_3)_3}$; [14732-21-9]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 215. Solubility of $\mathrm{Dy(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.010 ± 0.001
5.03	1.21	0.836 ± 0.004
10.03	2.51	0.687 ± 0.001
20.09	5.48	0.453 ± 0.001
40.03	13.34	0.181 ± 0.0003

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Dysprosium iodate was prepared by a dropwise addition of both the acidic solution of dysprosium nitrate and aqueous solution of potassium iodate into the $\mathrm{KNO_3}$ solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 % and ± 0.5 %.
Temperature: precision ± 0.05 K.

Components:

(1) Dysprosium iodate; $Dy(IO_3)_3$; [14732-21-9]
(2) Lithium iodate; $LiIO_3$; [13765-03-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, N. L. Svetlichnaya, and V. A. Kuzina, Zh. Neorg. Khim. **34**, 258–60 (1989); Russ. J. Inorg. Chem. (Engl. Transl.) **34**, 146–8 (1989).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 216. Composition of saturated solutions in the ternary $Dy(IO_3)_3$–$LiIO_3$–H_2O system at 25 °C

$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	Solid phase[b]
—	—	0.069[d]	18×10^{-4}	A
4.01	0.41	< 0.001	0.27×10^{-4}	C
7.28	0.77	0.001	0.28×10^{-4}	C
14.04	1.59	0.001	0.30×10^{-4}	C
21.91	2.70	0.002	0.65×10^{-4}	C
25.78	3.33	0.002	0.68×10^{-4}	C
30.58	4.18	0.003	1.1×10^{-4}	C
34.26	4.91	0.006	2.3×10^{-4}	C
37.63	5.64	0.007	2.8×10^{-4}	C
42.19	6.74	0.011	4.7×10^{-4}	C
43.30[c]	7.03	0.013[c]	5.6×10^{-4}	B+C[c]
43.82[d]	7.17	—	—	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $Dy(IO_3)_3 \cdot 4H_2O$; B = $LiIO_3$; C = solid solution based on $Dy(IO_3)_3 \cdot 4H_2O$.

[c] The results are duplicated in the original paper.

[d] For the binary systems the compilers computed the following values:

Solubility of $Dy(IO_3)_3$ = 1.00×10^{-3} mol kg^{-1}
Solubility of $LiIO_3$ = 4.29 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied by the isothermal method. The equilibration time was 25–30 d. The liquid and solid phases of the system were analyzed for the iodate and dysprosium ions. The iodate content was determined iodometrically. Dysprosium in the solid “residues” and in the liquid was determined by photometry using arsenazo III after reducing the iodate ion with hydroxylamine. Lithium concentration in the samples was found by difference and determined by flame emission spectroscopy.

Source and Purity of Materials:

Lithium iodate of high purity was used. Dysprosium iodate tetrahydrate was obtained by the reaction of dysprosium nitrate and iodic acid.

Estimated Error:

Solubility: precision of iodate and dysprosium contents ± (10–15) %.
Temperature: precision ± 0.1 K.

Components:
(1) Holmium iodate; $\mathrm{Ho(IO_3)_3}$; [23340-47-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility of holmium iodate in water at 298 K have been reported in 4 publications (1–4). Firsching and Paul (1) and Bertha and Choppin (2) deal solely with the binary system, and they also reported the activity solubility product. The solubility of the iodate in water with that in aqueous–organic solvent mixtures has been reported in 2 publications (3, 4): the reported solubility in water has been treated as a single measurement.

Miyamoto et al. (3, 4) used the dihydrate of holmium iodate for solubility measurements, and Bertha and Choppin stated that the iodate used had no water of crystallization. On the other hand, Firsching and Paul (1) did not report the hydration number of the salt being in equilibrium with its saturated solution.

Experimental data of solubility for holmium iodate in water at 298 K are summarized in Table 217.

TABLE 217. Experimental solubility data of $\mathrm{Ho(IO_3)_3}$ in water

T / K	10^3c_1 / mol dm^{-3}	Analytical method	Reference
298.2	1.16	Iodometry (IO_3^-), Spectrophotometry (Ho^{3+})	(1)
298.15	1.162	Iodometry	(3, 4)
298	1.17	Iodometry	(2)

We assume that composition of the equilibrium solid phase $\mathrm{Ho(IO_3)_3 \cdot 2H_2O}$ was the same either in preparation or the solubility determination because they were performed at almost the same temperature.

The results of Firsching and Paul (1), Bertha and Choppin (2) and Miyamoto et al. (3, 4) agree within the experimental errors. The arithmetic mean value is 1.16×10^{-3} mol dm^{-3}, and it is designated as a recommended value at 298.2 K.

The Ternary System

Miyamoto et al. (3, 4) measured the solubilities of the holmium iodate in mixtures of organic solvents and water at 298.15 K. Methanol (3), ethanol (3) and dimethyl sulfoxide (4) were used as the organic co-solvents. The solubility of the iodate decreases with an increase of the organic solvent concentration in the mixtures.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).

Components:

(1) Holmium iodate; $\mathrm{Ho(IO_3)_3}$; [23340-47-8]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported holmium and iodate concentrations in the saturated solution at 25.0 °C:

$[\mathrm{Ho^{3+}}] = (1.15 \pm 0.03) \times 10^{-3}$ mol dm^{-3}
$[\mathrm{IO_3^-}] = 3.5 \times 10^{-3}$, 3.5×10^{-3}, 3.4×10^{-3}, and 3.5×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Ho(IO_3)_3} = 1.15 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 2.0 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 7.0 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the solute was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of holmium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of $\mathrm{IO_3^-}$ determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. The holmium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium holmium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

Holmium nitrate was prepared by dissolving about 10 g holmium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of holmium. The precipitated holmium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision ± 2 % or less.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Holmium iodate; $\mathrm{Ho(IO_3)_3}$; [23340-47-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Ho(IO_3)_3}$ in water at 25 °C is 1.17×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Ho(IO_3)_3}$ in water at 25 °C is 1.34×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Ho(IO_3)_3}$ at infinite dilution was determined to be 13.4 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Ho(IO_3)_3}$ calculated by the authors were 62.1 kJ and -0.163 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{HoIO_3^{2+}}$ ion was 18 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of $\mathrm{Ho(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made with each sample of holmium iodate. The enthalpy of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Ho(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Ho(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until pH of 3.25 was obtained. $\mathrm{Ho(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and the $\mathrm{Ho(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Holmium iodate; $\mathrm{Ho(IO_3)_3}$; [23340-47-8]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 218. Solubility of $\mathrm{Ho(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.162 ± 0.001
Methanol	5.03	2.89	0.791 ± 0.0002
	9.88	5.81	0.547 ± 0.0001
	19.76	12.16	0.272 ± 0.0003
	40.11	27.35	0.050 ± 0.0004
Ethanol	4.84	1.95	0.749 ± 0.001
	10.03	4.18	0.474 ± 0.001
	19.84	8.82	0.196 ± 0.0000
	40.01	20.69	0.036 ± 0.001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The holmium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Holmium iodate was prepared by dropwise addition of both the acidic solution of holmium nitrate and the aqueous solution of potassium iodate into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The holmium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Ho–22.67; IO_3–72.13; H_2O–5.0 mass %; calculated for $\mathrm{Ho(IO_3)_3 \cdot 2H_2O}$: Ho–22.73; IO_3–72.31; H_2O–5.0 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of $0.98 \pm 0.05\ \mu\mathrm{S\ cm^{-1}}$ was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 3 %.

Temperature: precision ± 0.05 K.

Components:

(1) Holmium iodate; $\mathrm{Ho(IO_3)_3}$; [23340-47-8]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 219. Solubility of $\mathrm{Ho(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.162 ± 0.001
5.03	1.21	0.965 ± 0.003
10.03	2.51	0.786 ± 0.001
20.09	5.48	0.505 ± 0.0003
40.03	13.34	0.193 ± 0.0000

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Holmium iodate was prepared by a dropwise addition of both the acidic solution of holmium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytical grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 0.3 %.

Temperature: precision ± 0.05 K.

Components:

(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan, and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility of erbium iodate in water at 298 K have been reported in 5 publications (1–5). One study (1) deals solely with the binary system, and the activity solubility product was also reported therein. The solubility in pure water with the solubilities in aqueous–organic solvent mixtures has been reported in (3, 4): the value in water was treated a single independent measurement.

Erbium iodate was prepared from solutions of erbium salts and alkali metal iodates. Bertha and Choppin (2) used the anhydrous iodate for solubility measurements, and Miyamoto et al. (3, 4) used the dihydrate. Firsching and Paul (1), and Vasil'ev and Serebrennikov (5) did not report the number of hydrating water molecules in the salt used. It seems that the equilibrium solute is the dihydrate because $\mathrm{Er(IO_3)_3 \cdot 2H_2O}$ was prepared by (3, 4) near 298 K.

Experimental data of erbium iodate in water at 298 K are summarized in Table 220.

TABLE 220. Experimental solubility data of $\mathrm{Er(IO_3)_3}$ in water

T / K	$10^3 c_1$ / mol dm^{-3}	Analytical method	Reference
298	0.488	Iodometry	(5)
298	1.31	Iodometry	(2)
298.15	1.379	Iodometry	(3, 4)
298.2	1.38	Iodometry (IO_3^-) Spectrophotometry (Er^{3+})	(1)

The solubility result in water at pH 3.25 reported by Bertha and Choppin (2) is in fair agreement with that of Firsching and Paul (1) and Miyamoto et al. (3, 4). The solubility value in water of pH 5.6–5.7 reported by Vasil'ev and Serebrennikov (5) is distinctly different from those obtained by other investigators (1–4), and the reported value of 0.488×10^{-3} mol dm^{-3} is rejected as being too low. The mean of the 3 reasonable values from (1–4) is 1.36×10^{-3} mol dm^{-3}, and the standard deviation is 0.02×10^{-3} mol dm^{-3}. The mean value is designated as the tentative solubility at 298 K.

The Ternary System

Miyamoto et al. (3, 4) have reported the solubilities of erbium iodate in various organic solvent–water mixtures at 298.15 K. Methanol, ethanol and dimethyl sulfoxide were used as the organic solvents, and their concentrations in the mixtures were changed between 0–40 mass %. In each case the solubility decreases with increasing the concentration of the organic co-solvent.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
5. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).

Components:
(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported erbium and iodate concentrations in the saturated solution at 25.0 °C:

[Er^{3+}] = $(1.40 \pm 0.04) \times 10^{-3}$ mol dm^{-3}
[IO_3^-] = 4.1×10^{-3}, 4.1×10^{-3}, 3.9×10^{-3}, and 4.1×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following solubility result:

$$\text{Solubility of } \mathrm{Er(IO_3)_3} = 1.38 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 3.9 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 8.4 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:
About 10 g of erbium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air–cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of IO_3^- determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by iodometric titration using a standard thiosulfate solution and starch as an indicator. Erbium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium erbium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:
Erbium iodate was prepared by dissolving about 10 g erbium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of erbium. The precipitated erbium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:
Solubility: precision of ± 2 % or less.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Er(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 $^\circ\mathrm{C}$ is given below:

0.438 g $\mathrm{dm^{-3}}$	(authors)
0.633×10^{-3} mol $\mathrm{dm^{-3}}$	(recalculated by the compilers)

The number of hydrating water molecules in the equilibrium solid was not reported. Molar ratio of erbium to iodate was found to be 1:3. A formation of solid $\mathrm{Er_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:
A weighed sample of erbium iodate was introduced with distilled water into a 25 $\mathrm{cm^3}$ pycnometer with a ground–glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). The water used for solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in 2 samples of the solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:
Erbium iodate was prepared from acidic solution of erbium chloride (99.8+ % pure) and potassium iodate solution. The erbium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:
Solubility: nothing specified.
Temperature: precision ± 0.01 K (authors of (1)).

References:

[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:
(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Er(IO_3)_3}$ in water at 25 °C is 1.31×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Er(IO_3)_3}$ in water at 25 °C is 19.3×10^{-12} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Er(IO_3)_3}$ at infinite dilution was determined to be 13.0 kJ mol^{-1}. The free energy and entropy of the solution for $\mathrm{Er(IO_3)_3}$ calculated by the authors were 61.2 kJ and -0.162 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{ErIO_3^{2+}}$ ion was 19 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
A sample of $\mathrm{Er(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometric titration. Three or 4 measurements of the iodate concentration were made with each sample of the erbium iodate solution. The heat of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Er(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Er(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of its iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Er(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Er(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:
(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:
Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 221. Solubility of $\mathrm{Er(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.379 ± 0.0001
Methanol	5.03	2.89	0.919 ± 0.001
	9.88	5.81	0.621 ± 0.001
	19.76	12.16	0.283 ± 0.001
	40.11	27.35	0.058 ± 0.001
Ethanol	4.84	1.95	0.866 ± 0.0001
	10.03	4.18	0.549 ± 0.001
	19.84	8.82	0.224 ± 0.001
	40.01	20.69	0.040 ± 0.0001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The erbium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept further in the thermostat for about 1 h. Aliquots were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Erbium iodate was prepared by dropwise addition of both acidic solution of erbium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The erbium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Er–22.92; IO_3–72.08; H_2O–5.1 mass %; calculated for $\mathrm{Er(IO_3)_3 \cdot 2H_2O}$: Er–22.98; IO_3–72.08; H_2O–4.9 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours, distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.01 and ± 0.2 %.
Temperature: precision ± 0.05 K.

Components:
(1) Erbium iodate; $\mathrm{Er(IO_3)_3}$; [14723-96-7]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:
H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:
Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

TABLE 222. Solubility of $\mathrm{Er(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.379 ± 0.0001
5.03	1.21	1.120 ± 0.001
10.03	2.51	0.904 ± 0.001
20.09	5.48	0.573 ± 0.0002
40.03	13.34	0.208 ± 0.002

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:
The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were further kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:
Erbium iodate was prepared by a dropwise addition of both the acidic solution of erbium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 μS cm^{-1} was used.

Estimated Error:
Solubility: standard deviation between ± 0.01 and ± 1%.
Temperature: precision ± 0.05 K.

O. Thulium Iodate

Components:

(1) Thulium iodate; $\mathrm{Tm(IO_3)_3}$; [14723-97-8]

(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility of thulium iodate in water at 298 K have been reported in 5 publications (1–5). Firsching and Paul (1) have performed solely the determination with the binary system, and the solubility product was also reported. The solubility in pure water with the solubilities in aqueous–organic solvent mixtures has been reported in (3, 4), and the solubility in water was treated a single independent measurement.

The solubility of the iodate in water of pH 3.25 has been reported by Bertha and Choppin (2). The study (5) deals with a ternary system, and the solubility in water (based on mass % units) was given as one point on the corresponding phase diagram.

Thulium iodate was prepared from solutions of thulium salts and alkali metal iodates or iodic acid. Shklovskaya et al. (5) have reported that the solid phase equilibrated with the saturated aqueous solution at 298 K was the tetrahydrate. Bertha and Choppin (2) used anhydrous salt for the solubility measurements, and Miyamoto et al. (3, 4) used the dihydrate. By analogy to other lanthanides it seems that the equilibrium solute is the dihydrate. Firsching and Paul (1) did not report the number of hydrating water molecules in the solid.

Experimental data of thulium iodate solubility in water are summarized in Table 223.

TABLE 223. Experimental solubility data of $\mathrm{Tm(IO_3)_3}$ in water

T / K	10^3c_1 / mol dm^{-3}	10^3m_1 / mol kg^{-1}	Analytical method	Reference
298	—	1.44	Iodometry (IO_3^-); Complexometry (Tm^{3+})	(5)
298.2	1.46	—	Iodometry (IO_3^-); Spectrophotometry (Tm^{3+})	(1)
298.15	1.467	—	Iodometry	(3, 4)
298	1.47	—	Iodometry	(2)

The reported values based on mol dm^{-3} units are in good agreement and the mean value of 1.47×10^{-3} mol dm^{-3} is designated as the recommended solubility at 298 K. The reported value of 1.44×10^{-3} mol kg^{-1} (5) may be designated as a tentative solubility at 298 K, but it seems that this value should be slightly higher because by a comparison with the solubilities expressed in mol kg^{-1} and mol dm^{-3} units for $\mathrm{La(IO_3)_3}$, the difference should be smaller than 1%.

Miyamoto et al. (3, 4) measured the solubilities of $\mathrm{Tm(IO_3)_3}$ in the mixtures of various organic solvents and water at 298.15 K. The authors used methanol (3), ethanol (3) and dimethyl sulfoxide (4) as organic solvents, and studied the solubilities in these mixtures with the purpose of testing any applicability of the simple electrostatic model. The solubility of the thulium iodate decreases with increasing the concentration of organic solvents.

Shklovskaya et al. (5) have studied the solubilities of the ternary $\mathrm{Tm(IO_3)_3}$–$\mathrm{LiIO_3}$–$\mathrm{H_2O}$ system by isothermal method. The system is of eutonic type with formation of a restricted solid solution based on thulium iodate tetrahydrate.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).
5. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, A. G. Tokareva, and V. A. Kuzina, Zh. Neorg. Khim. **35**, 270 (1990); Russ. J. Inorg. Chem. (Engl. Transl.) **35**, 154 (1990).

Components:
(1) Thulium iodate; $\mathrm{Tm(IO_3)_3}$; [14723-97-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:
One temperature: 298.2 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported thulium and iodate concentrations in the saturated solution at 25.0 °C:

[$\mathrm{Tm^{3+}}$] = $(1.45 \pm 0.06) \times 10^{-3}$ mol dm^{-3}
[$\mathrm{IO_3^-}$] = 4.4×10^{-3}, 4.4×10^{-3}, 4.3×10^{-3}, and 4.4×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following solubility result:

Solubility of $\mathrm{Tm(IO_3)_3}$ = 1.45×10^{-3} mol dm^{-3}.

The activity solubility product was reported by the authors:

$$K_s^\circ = 4.4 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 8.8 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the equilibrium solid was reported.

Auxiliary Information

Method/Apparatus/Procedure:
About 10 g of thulium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected by 4 concentrations of $\mathrm{IO_3^-}$ determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. Thulium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium thulium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:
Thulium iodate was prepared by dissolving about 10 g of thulium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of thulium. The precipitated thulium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:

Solubility: precision of ± 2 % or less.

Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).
[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:

(1) Thulium iodate; $\mathrm{Tm(IO_3)_3}$; [14723-97-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Tm(IO_3)_3}$ in water at 25 °C is 1.47×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Tm(IO_3)_3}$ in water at 25 °C is 2.83×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Tm(IO_3)_3}$ at infinite dilution was determined to be 10.9 kJ mol^{-1}. The free energy and entropy of solution of $\mathrm{Tm(IO_3)_3}$ calculated by the authors were 60.2 kJ and -0.165 J mol^{-1} K^{-1}, respectively. The formation constant of $TmIO_3^{2+}$ ion was 19 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $\mathrm{Tm(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made with each sample of thulium iodate. The enthalpy of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:

$\mathrm{Tm(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Tm(ClO_4)_3}$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. $\mathrm{Tm(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Tm(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Thulium iodate; $\mathrm{Tm(IO_3)_3}$; [14723-97-8]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 224. Solubility of $\mathrm{Tm(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.467 ± 0.001
Methanol	5.03	2.89	1.008 ± 0.0001
	9.88	5.81	0.682 ± 0.0001
	19.76	12.16	0.313 ± 0.002
	40.11	27.35	0.060 ± 0.004
Ethanol	4.84	1.95	0.974 ± 0.0002
	10.03	4.18	0.599 ± 0.001
	19.84	8.84	0.250 ± 0.002
	40.01	20.01	0.049 ± 0.001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified thulium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 $^\circ\mathrm{C}$ for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Thulium iodate was prepared by dropwise addition of both acidic solution of thulium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The thulium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product: Tm–23.08; IO_3–72.02; H_2O–5 0 mass %; calculated for $\mathrm{Tm(IO_3)_3 \cdot 2H_2O}$: Tm–23.15; IO_3–71.91; H_2O–4.9 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 $^\circ\mathrm{C}$ was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 $^\circ\mathrm{C}$ was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: precision between ± 0.02 and ± 6 %.

Temperature: precision ± 0.05 K.

Components:

(1) Thulium iodate; $\mathrm{Tm(IO_3)_3}$; [14723-97-8]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 225. Solubility of $\mathrm{Tm(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.467 ± 0.001
5.03	1.21	1.217 ± 0.0002
10.03	2.51	0.978 ± 0.0002
20.09	5.48	0.616 ± 0.0001
40.03	13.34	0.214 ± 0.0001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrate was kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Thulium iodate was prepared by a dropwise addition of both the acidic solution of thulium nitrate and aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated values for dihydrate salt was within ± 0.5 %. Analytical grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.02 % and ± 0.1 %.

Temperature: precision ± 0.05 K.

Components:

(1) Thulium iodate; $Tm(IO_3)_3$; [14723-97-8]
(2) Lithium iodate; $LiIO_3$; [13765-03-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, A. G. Tokareva, and V. A. Kuzina, Zh. Neorg. Khim. **35**, 270–2 (1990); Russ. J. Inorg. Chem. (Engl. Transl.) **35**, 154–5 (1990).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 226. Composition of saturated solutions in the ternary $Tm(IO_3)_3$–$LiIO_3$–H_2O system at 25 °C

$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	Solid phase[b]
—	—	0.10[c]	2.6×10^{-3}	A
3.85	0.395	0.0050	1.3×10^{-4}	C
7.07	0.748	0.0046	1.27×10^{-4}	C
12.60	1.408	0.0043	1.26×10^{-4}	C
17.56	2.07	0.0040	1.23×10^{-4}	C
22.87	2.85	0.0035	1.14×10^{-4}	C
25.81	3.33	0.0033	1.11×10^{-4}	C
28.99	3.89	0.0050	1.75×10^{-4}	C
35.51	5.17	0.0058	2.21×10^{-4}	C
39.22	6.01	0.0060	2.41×10^{-4}	C
42.73	6.88	0.0070	2.9×10^{-4}	C
43.80	7.17	0.016	6.9×10^{-4}	B+C
43.82[c]	7.17	—	—	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $Tm(IO_3)_3 \cdot 4H_2O$; B = $LiIO_3$; C = solid solution based on $Tm(IO_3)_3 \cdot 4H_2O$.

[c] For the binary systems the compilers computed the following solubility values:

Solubility of $Tm(IO_3)_3$ = 1.44×10^{-3} mol kg^{-1}
Solubility of $LiIO_3$ = 4.29 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The equilibrium in the system was reached in isothermal conditions after 20–25 d. Aliquots of the coexisting phases were analyzed for iodate anion by an iodometric titration and for thulium cation in the solid “residues” by complexometric titration using xylenol orange as an indicator, and in the liquid phase specimens by photometry using arsenazo III agent and checked spectrographically. The lithium content was found by difference and confirmed by flame emission spectroscopy. The solid phases were checked by X-ray diffraction.

Source and Purity of Materials:

“Special purity” grade lithium iodate was used. Thulium iodate was prepared from thulium nitrate and iodic acid.

Estimated Error:

Solubility: reproducibility 3–8 %.
Temperature: precision ± 0.1 K.

Components:

(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility of ytterbium iodate in water at 298 K have been reported in 6 publications (1–6). Firsching and Paul (1) deal solely with the binary system and the solubility product was also reported therein. The solubility in pure water with the solubilities in aqueous–organic solvent mixtures has been reported by Miyamoto et al. (3, 4) and the solubility in water was treated as single independent measurement. The solubilities of the ytterbium iodate in water at pH 3.25 and pH 5.6–5.7 have been reported by Bertha and Choppin (2) and Vasil'ev and Serebrennikov (6), respectively. The pH values of water solvent were not specified by the other investigators. The remaining one study (5) deals with a ternary system, and the solubility in water based on mass % units was given as one point on a phase diagram enclosed.

Ytterbium iodate was prepared from solutions of ytterbium salts and alkali metal iodates or iodic acid. Shklovskaya et al. (5) have reported that the solid phase equilibrated with the aqueous saturated solution at 298 K was the tetrahydrate. Bertha and Choppin (2) used the anhydrous salt for solubility measurements and Miyamoto et al. (3, 4) used the dihydrate. Firsching and Paul (1), and Vasil'ev and Serebrennikov (6) did not report the number of hydrating water molecules in the solid.

Experimental data of solubility of ytterbium iodate in water at 298 K are summarized in Table 227.

TABLE 227. Experimental solubility data of $\mathrm{Yb(IO_3)_3}$ in water

T / K	10^3c_1 / mol dm^{-3}	10^3m_1 / mol kg^{-1}	Analytical method	Reference
298	0.694	—	Iodometry(IO_3^-)	(6)
298.15	1.620	—	Iodometry	(3, 4)
298	1.63	—	Iodometry	(2)
298.2	1.64	—	Iodometry(IO_3^-) Spectrophotometry(Yb^{3+})	(1)
298	—	1.72	Photometry(Yb^{3+})	(5)

The value of Vasil'ev and Serebrennikov (6) is markedly smaller than those of the other investigators and is therefore rejected. The arithmetic mean of the 3 reasonable values is 1.63×10^{-3} mol dm^{-3}, and the standard deviation is 0.01×10^{-3} mol dm^{-3}. The mean value is designated as a recommended solubility. The solubility result expressed in mol kg^{-1} (5) seems to be slightly too high.

The Ternary System

Miyamoto et al. (3, 4) measured the solubilities of ytterbium iodate in the mixtures of various organic solvents and water at 298 K. Methanol, ethanol, and dimethyl sulfoxide were used as the organic solvents. The authors studied the solubilities in various mixed solvents with the purpose of testing the applicability of the simple electrostatic model. The results are designated as tentative solubilities.

Shklovskaya et al. (5) studied the solubilities for the ternary $\mathrm{Yb(IO_3)_3}$–$\mathrm{LiIO_3}$–$\mathrm{H_2O}$ system. The system is of the eutonic type and restricted solid solutions based on ytterbium iodate tetrahydrate are formed.

References:

1. F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414 (1966).
2. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).
3. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).
4. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **95**, 2973 (1986).
5. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, B. I. Kidyarov, V. A. Kuzina, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **33**, 219 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 122 (1988).
6. G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103 (1962).

Components:
(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. I. Vasil'ev and V. V. Serebrennikov, Tr. Tomsk. Gos. Univ., Ser. Khim. **154**, 103–4 (1962).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Yb(IO_3)_3}$ in water acidified to pH 5.6–5.7 at 25 $^\circ\mathrm{C}$ is given below:

0.484 g dm^{-3}	(authors)
0.694×10^{-3} mol dm^{-3}	(recalculated by the compilers).

The number of hydrating water molecules in the solid was not reported, but molar ratio of ytterbium to iodate was found to be 1:3. A formation of solid $\mathrm{Yb_2(IO_3)_6}$ is suggested by the authors.

Auxiliary Information

Method/Apparatus/Procedure:
A weighed sample of ytterbium iodate was introduced with water into a 25 cm^3 pycnometer with a ground-glass stopper which was sealed externally with paraffin wax. The pycnometer was placed in a thermostat and shaken mechanically at the selected temperature for 10–12 h until saturation (1). The distilled water used for solubility determinations was acidified to pH 5.6–5.7 before use. The iodate content was determined iodometrically in 2 samples of the saturated solution. The solutions were titrated with thiosulfate standard solution in presence of starch as an indicator after addition of potassium iodide and sulfuric acid.

Source and Purity of Materials:
Ytterbium iodate was prepared from acidic solution of ytterbium chloride (99.8+ % pure) and potassium iodate solution. The ytterbium iodate precipitate was separated from the solution by filtration and washed with water. Distilled water was used.

Estimated Error:
Solubility: nothing specified.
Temperature: precision ± 0.01 K (authors of (1)).

References:
[1] G. I. Vasil'ev and V. V. Serebrennikov, Zh. Neorg. Khim. **6**, 2716 (1961); Russ. J. Inorg. Chem. (Engl. Transl.) **6**, 1372 (1961).

Components:

(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

F. H. Firsching and T. R. Paul, J. Inorg. Nucl. Chem. **28**, 2414–6 (1966).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The authors reported ytterbium and iodate concentrations in the saturated solution at 25.0 °C:

[Yb^{3+}] = $(1.65 \pm 0.07) \times 10^{-3}$ mol dm^{-3}

[IO_3^-] = 4.9×10^{-3}, 4.9×10^{-3}, 4.8×10^{-3}, and 4.9×10^{-3} mol dm^{-3}
(from different procedures).

The compilers computed the following value of the solubility:

$$\text{Solubility of } \mathrm{Yb(IO_3)_3} = 1.64 \times 10^{-3} \text{ mol dm}^{-3}.$$

The activity solubility product was reported by the authors:

$$K_s^\circ = 6.1 \times 10^{-11} \text{ mol}^4 \text{ dm}^{-12}.$$

The activity coefficients were calculated using the ionic strength of the equilibrium solution ($I = 9.8 \times 10^{-3}$) and the ion size data of Kielland (1).

No number of hydrating water molecules in the solid phase was reported.

Auxiliary Information

Method/Apparatus/Procedure:

About 10 g of ytterbium iodate and 15 cm^3 of water were placed in an Erlenmeyer flask, which was fitted with an air-cooled condenser, and placed on an oscillating hot plate at 80 °C for 8 h. The flask was allowed to cool to the selected temperature with agitation for 24 h and then placed in a constant temperature cabinet for several days. Three slightly modified procedures were also used; their results are reflected in 4 concentrations of IO_3^- determined. The saturated solution was centrifuged to remove any suspended particles. The iodate content was determined by an iodometric titration using a standard thiosulfate solution and starch as an indicator. Ytterbium was analyzed by the method of Stewart and Kato (2) using a recording spectrophotometer. The spectra of the equilibrium ytterbium iodate solution was compared to spectra of the known solution of pure corresponding perchlorate.

Source and Purity of Materials:

Ytterbium iodate was prepared by dissolving about 10 g ytterbium oxide (99.8–99.9 % purity) in about 200 cm^3 of water and an excess of HNO_3. A solution containing a slight stoichiometric excess of recrystallized KIO_3 (reagent grade) was slowly poured, with stirring, into the nitric acid solution of ytterbium. The precipitated ytterbium iodate was separated and then agitated with 15 cm^3 of water at 80 °C for several hours to remove any soluble species.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: precision ± 0.2 K.

References:

[1] J. Kielland, J. Am. Chem. Soc. **59**, 1675 (1937).

[2] D. C. Stewart and D. Kato, Anal. Chem. **30**, 164 (1958).

Components:
(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto

Experimental Data

The solubility of $\mathrm{Yb(IO_3)_3}$ in water at 25 °C is 1.63×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $\mathrm{Yb(IO_3)_3}$ in water at 25 °C is 4.01×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $\mathrm{Yb(IO_3)_3}$ at infinite dilution was determined to be 9.6 kJ mol^{-1}. The free energy and entropy of solution for $\mathrm{Yb(IO_3)_3}$ calculated by the authors were 59.4 kJ and -0.167 J mol^{-1} K^{-1}, respectively. The formation constant of $\mathrm{YbIO_3^{2+}}$ ion was 19 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:
Sample of $\mathrm{Yb(IO_3)_3}$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the iodate concentration were made for each sample of ytterbium iodate solution. The heat of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:
$\mathrm{Yb(IO_3)_3}$ was prepared from a vigorously stirred dilute solution of $\mathrm{Yb(ClO_4)_3}$ by addition of an equivalent amount of $\mathrm{NaIO_3}$ solution. The precipitate was washed several times with slightly acidified water (with $\mathrm{HClO_4}$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of $\mathrm{HIO_3}$ with NaOH until a pH of 3.25 was obtained. $\mathrm{Yb(ClO_4)_3}$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $\mathrm{HClO_4}$. The solutions were evaporated under a heat lamp to remove excess acid and then the $\mathrm{Yb(ClO_4)_3}$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:
Solubility: precision ± 2 % or less.
Temperature: nothing specified.

Components:

(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 228. Solubility of $\mathrm{Yb(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	1.620 ± 0.003
Methanol	5.03	2.89	1.081 ± 0.001
	9.88	5.81	0.734 ± 0.001
	19.76	12.16	0.338 ± 0.002
	40.11	27.35	0.065 ± 0.002
Ethanol	4.84	1.95	1.060 ± 0.0003
	10.03	4.18	0.641 ± 0.003
	19.84	8.82	0.276 ± 0.0003
	40.01	20.69	0.051 ± 0.0004

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified ytterbium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Ytterbium iodate was prepared by dropwise addition of both acidic solution of ytterbium nitrate and aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product was filtered, dried under reduced pressure at room temperature, and stored in the dark. The ytterbium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Yb–23.53; IO_3–71.78; H_2O–4.9 mass %; calculated for $\mathrm{Yb(IO_3)_3 \cdot 2H_2O}$: Yb–23.58; IO_3–71.51; H_2O–4.9 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and then distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.03 and ± 3 %.
Temperature: precision ± 0.05 K.

Components:

(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]
(2) Sulfinyl bis-methane (dimethyl sulfoxide); $\mathrm{C_2H_6OS}$; [67-68-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 229. Solubility of $\mathrm{Yb(IO_3)_3}$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	1.620 ± 0.003
5.03	1.21	1.343 ± 0.001
10.03	2.51	1.085 ± 0.001
20.09	5.48	0.662 ± 0.001
40.03	13.34	0.227 ± 0.001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrate was kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Ytterbium iodate was prepared by a dropwise addition of both the acidic solution of ytterbium nitrate and aqueous solution of potassium iodate into the $\mathrm{KNO_3}$ solution. The agreement between the analytically found and the theoretically calculated values for dihydrate salt was within ± 0.5 %. Analytically pure dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 % and ± 0.5 %.
Temperature: precision ± 0.05 K.

Components:

(1) Ytterbium iodate; $\mathrm{Yb(IO_3)_3}$; [14723-98-9]

(2) Lithium iodate; $LiIO_3$; [13765-03-2]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, B. I. Kidyarov, V. A. Kuzina, A. G. Tokareva, and L. A. Kolobkova, Zh. Neorg. Khim. **33**, 219–21 (1988); Russ. J. Inorg. Chem. (Engl. Transl.) **33**, 122–3 (1988).

Variables:

Composition of the salts

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 230. Composition of saturated solutions in the ternary $\mathrm{Yb(IO_3)_3}$–$LiIO_3$–H_2O system at 25 °C

$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	Solid phase[b]
—	—	0.120[c]	3.1×10^{-3}	A
3.41	0.349	0.024	6.4×10^{-4}	B
5.25	0.546	0.015	4.1×10^{-4}	B
9.9	1.08	0.0040	1.1×10^{-4}	B
14.7	1.68	0.0020	6.0×10^{-5}	B
21.50	2.64	0.0012	3.8×10^{-5}	B
27.08	3.55	0.0010	3.4×10^{-5}	B
29.43	3.97	0.0009	3.2×10^{-5}	B
34.77	5.02	0.0020	7.5×10^{-5}	B
38.70	5.89	0.0050	2.0×10^{-4}	B
40.91	6.42	0.010	4.1×10^{-4}	B
43.34	7.05	0.039	1.7×10^{-3}	B+C
43.82[c]	7.17	—	—	C

[a] Calculated by the compilers.

[b] The solid phases are: A = $\mathrm{Yb(IO_3)_3 \cdot 4H_2O}$; B = solid solution based on $\mathrm{Yb(IO_3)_3 \cdot 4H_2O}$; C = $LiIO_3$.

[c] For the binary systems the compilers computed the following values:

Solubility of $\mathrm{Yb(IO_3)_3}$ = 1.72×10^{-3} mol kg^{-1}
Solubility of $LiIO_3$ = 4.29 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied by the isothermal method. Equilibrium was reached in 20–30 d. Samples of the coexisting phases were analyzed for iodate ion by an iodometric titration. Ytterbium was determined in the solid "residues" by a complexometric titration using xylenol orange as an indicator and in the liquid phases by a photometric method using arsenazo III agent after the iodate ion had been reduced with hydroxylamine; its content was checked by a spectrographic method. The lithium in the sample was calculated by difference and checked by a flame emission spectroscopy. The solid phases were confirmed by X-ray diffraction.

Source and Purity of Materials:

"Special purity" grade $LiIO_3$ was used. Ytterbium iodate tetrahydrate was prepared from ytterbium nitrate and iodic acid.

Estimated Error:

Solubility: the uncertainty did not exceed 3–8 %.

Temperature: precision ± 0.1 K.

Q. Lutetium Iodate

Components:

(1) Lutetium iodate; $\mathrm{Lu(IO_3)_3}$; [15513-87-8]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. October 2001.

Critical Evaluation:

The Binary System

Data for the solubility in the binary $\mathrm{Lu(IO_3)_3}$–H_2O system at 298 K have been reported in 4 publications (1–4). One value based on mol kg^{-1} units was reported by Shklovskaya et al. (4). They stated that the solid phase equilibrated with the aqueous saturated solution of lutetium iodate at 298 K was the tetrahydrate. The reported solubility value of 2.00×10^{-3} mol kg^{-1} is designated as a tentative solubility at 298 K.

Data for the solubility based on mol dm^{-3} units have been reported by 2 groups: 1.78×10^{-3} by Bertha and Choppin (1), and 2.036×10^{-3} by Miyamoto et al. (2, 3). The result of Miyamoto et al. (2, 3) is more precise and is in better agreement with that of Shklovskaya et al. (4) rather than that of (1). The authors of (1) reported that their solubility determination was performed in water of pH 3.25, Miyamoto et al. did not report the pH value. The difference between the two values based on mol dm^{-3} units could be a result of the difference of the pH value as well as purity of the reagents used: Miyamoto et al. (2, 3) used materials of higher purity. The equilibrium solid phase deduced from paper of (2, 3) should be rather the dihydrate but the matter needs further investigations.

The arithmetic mean of the two reported values (1–3) is 1.9×10^{-3} mol dm^{-3}, and the mean value is designated as a doubtful solubility at 298 K.

The Ternary System

Miyamoto et al. (2, 3) measured the solubility of lutetium iodate in mixtures of various organic solvents and water at 298 K. Methanol (2), ethanol (2) and dimethyl sulfoxide (3) were used, and the concentrations of organic solvents were changed between 0 and 40 mass %. The solubilities in the mixed solvents were studied with the purpose of testing the applicability of the simple electrostatic model. The reported solubility values are designated as tentative values.

Shklovskaya et al. (4) have studied the solubilities of the ternary $\mathrm{Lu(IO_3)_3}$–$\mathrm{LiIO_3}$–H_2O system by isothermal method. The system is of eutonic type, and a restricted solid solution based on lutetium iodate tetrahydrate is formed.

References:

1. S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613 (1969).

2. H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485 (1985).

3. H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973 (1986).

4. R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, and N. L. Svetlichnaya, Zh. Neorg. Khim. **34**, 1904 (1989); Russ. J. Inorg. Chem. (Engl. Transl.) **34**, 1083 (1989).

Components:

(1) Lutetium iodate; $Lu(IO_3)_3$; [15513-87-8]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

S. L. Bertha and G. R. Choppin, Inorg. Chem. **8**, 613–7 (1969).

Variables:

One temperature: 298 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

The solubility of $Lu(IO_3)_3$ in water at 25 °C is 1.78×10^{-3} mol dm^{-3}. The solubility product, K_s°, of $Lu(IO_3)_3$ in water at 25 °C is 5.59×10^{-11} mol^4 dm^{-12}. The K_s° for a standard state of infinite dilution was calculated by employing mean activity coefficient from the Debye–Hückel equation.

The enthalpy of the solution of $Lu(IO_3)_3$ at infinite dilution was determined to be 8.4 kJ mol^{-1}. The free energy and entropy of solution for $Lu(IO_3)_3$ calculated by the authors were 58.6 kJ and -0.168 J mol^{-1} K^{-1}, respectively. The formation constant of $LuIO_3^{2+}$ ion was 19 mol^{-1} dm^3 at ionic strength of 0.10 mol dm^{-3}.

Auxiliary Information

Method/Apparatus/Procedure:

Sample of $Lu(IO_3)_3$ was heated in water of pH 3.25 at 80 °C for 4 d with magnetic stirring and refluxing. After cooling to room temperature, the solutions were kept in a water bath at the selected temperature for several days before analysis for iodate ion concentration by iodometry. Three or 4 measurements of the concentration were made for each sample of lutetium iodate solution. The enthalpy of the salt solution in water was measured in a calorimeter.

Source and Purity of Materials:

$Lu(IO_3)_3$ was prepared from a vigorously stirred dilute solution of $Lu(ClO_4)_3$ by addition of an equivalent amount of $NaIO_3$ solution. The precipitate was washed several times with slightly acidified water (with $HClO_4$ to pH 3.25). Thermal balance curves for the product indicated that there were no waters of crystallization. The purity, better than 99 %, was determined by conversion of the iodate to the oxide and comparing weights. Sodium iodate was prepared by neutralizing a solution of HIO_3 with NaOH until a pH of 3.25 was obtained. $Lu(ClO_4)_3$ solution was prepared by dissolving the oxide (Lindsay Chemical Division, 99.9 % pure) in a slight excess of 2 mol dm^{-3} $HClO_4$. The solutions were evaporated under a heat lamp to remove excess acid and the $Lu(ClO_4)_3$ solution was adjusted to a pH between 3.0 and 3.3. Distilled water was used.

Estimated Error:

Solubility: precision ± 2 % or less.

Temperature: nothing specified.

Components:

(1) Lutetium iodate; $\mathrm{Lu(IO_3)_3}$; [15513-87-8]
(2) Methanol; CH_4O; [67-56-1]
or (2) Ethanol; C_2H_6O; [64-17-5]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Shimura, and K. Sasaki, J. Solution Chem. **14**, 485–97 (1985).

Variables:

Concentration of methanol or ethanol: 0–40 mass %
One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto

Experimental Data

TABLE 231. Solubility of $\mathrm{Lu(IO_3)_3}$ in methanol–water and ethanol–water mixtures at 25.00 °C

Alcohol	$100w_2$	$100x_2$ [a]	10^3c_1 / mol dm^{-3}
None	—	—	2.036 ± 0.005
Methanol	5.03	2.89	1.347 ± 0.001
	9.88	5.81	0.804 ± 0.002
	19.76	12.16	0.395 ± 0.001
	40.11	27.35	0.080 ± 0.002
Ethanol	4.84	1.95	1.279 ± 0.001
	10.03	4.18	0.791 ± 0.001
	19.84	8.82	0.320 ± 0.001
	40.01	20.69	0.055 ± 0.0001

[a] Calculated by the compiler.

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The purified lutetium iodate and the solvent mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles continuously rotated for 48 h. After settling of the saturated solution in the thermostat for about 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrates were kept in the thermostat about 1 h. Aliquots of the saturated solution were diluted with water to adjust to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Lutetium iodate was prepared by dropwise addition of both the acidic solution of lutetium nitrate and the aqueous solution of potassium iodate into the aqueous KNO_3 solution while heating. The mixture was continuously heated and stirred for about 5 h, and then allowed to settle for 1 d at room temperature. The precipitated iodate was separated and agitated with cold water for several hours to remove soluble species. The product wase filtered, dried under reduced pressure at room temperature, and stored in the dark. The lutetium, iodate and water contents were determined by complexometry, iodometry and thermogravimetry, respectively. Analysis of the product showed: Lu–23.71; IO_3–71.32; H_2O–4.9 mass %; calculated for $\mathrm{Lu(IO_3)_3 \cdot 2H_2O}$: Lu–23.78; IO_3–71.21; H_2O–4.9 mass %. Chemically pure methanol was refluxed over freshly calcined CaO for several hours, distilled, treated with $AgNO_3$, and then redistilled. The product was treated with Mg ribbon for 1 d and distilled twice. A fraction boiling at 64.6 ± 0.1 °C was used. Chemically pure ethanol was refluxed over $AgNO_3$ and KOH for several hours and distilled. The product was treated with Ca metal and then distilled twice. A fraction boiling at 78.2 ± 0.1 °C was used. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.1 and ± 0.4 %.

Temperature: precision ± 0.05 K.

Components:

(1) Lutetium iodate; $Lu(IO_3)_3$; [15513-87-8]

(2) Sulfinyl bis-methane (dimethyl sulfoxide); C_2H_6OS; [67-68-5]

(3) Water; H_2O; [7732-18-5]

Original Measurements:

H. Miyamoto, H. Iijima, and M. Sugawara, Bull. Chem. Soc. Jpn. **59**, 2973–8 (1986).

Variables:

Concentration of dimethyl sulfoxide: 0–40 mass %

One temperature: 298.15 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 232. Solubility of $Lu(IO_3)_3$ in dimethyl sulfoxide–water mixtures at 25.00 °C

$100w_2$	$100x_2$	10^3c_1 / mol dm^{-3}
0	0	2.036 ± 0.005
5.03	1.21	1.629 ± 0.001
10.03	2.51	1.304 ± 0.004
20.09	5.48	0.794 ± 0.0003
40.03	13.34	0.258 ± 0.0001

The equilibrium solid phase was not reported.

Auxiliary Information

Method/Apparatus/Procedure:

The supersaturation method was used. The iodate and dimethyl sulfoxide–water mixtures were placed into glass-stoppered bottles, which were slowly rotated in a thermostat at 27 °C for about 12 h. The temperature was lowered to the selected level and the bottles were continuously rotated for 48 h. After settling of the saturated solution in the thermostat for 1 h, the solutions were filtered through a vacuum jacketed sintered-glass filter. The filtrate was kept in the thermostat for about 1 h. Aliquots of the saturated solution were diluted with water to a suitable concentration for titration. The iodate content was determined iodometrically.

Source and Purity of Materials:

Lutetium iodate was prepared by a dropwise addition of both the acidic solution of lutetium nitrate and the aqueous solution of potassium iodate into the KNO_3 solution. The agreement between the analytically found and the theoretically calculated composition values for dihydrate salt was within ± 0.5 %. Analytically grade dimethyl sulfoxide (from Wako Chemical Co) was distilled under reduced pressure 3 times. Redistilled water with a specific conductivity of 0.98 ± 0.05 μS cm^{-1} was used.

Estimated Error:

Solubility: standard deviation between ± 0.03 % and ± 0.4 %.

Temperature: precision ± 0.05 K.

Components:

(1) Lutetium iodate; $Lu(IO_3)_3$; [15513-87-8]
(2) Lithium iodate; $LiIO_3$; [13765-03-2]
(3) Water; H_2O; [7732-18-5]

Original Measurements:

R. M. Shklovskaya, S. M. Arkhipov, L. I. Isaenko, L. A. Kolobkova, and N. L. Svetlichnaya, Zh. Neorg. Khim. **34**, 1904–5 (1989); Russ. J. Inorg. Chem. (Engl. Transl.) **34**, 1083 (1989).

Variables:

Composition of the salts
One temperature: 298 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 233. Composition of saturated solutions in the ternary $Lu(IO_3)_3$–$LiIO_3$–H_2O system at 25 °C

$100w_2$	$100x_2$[a]	$100w_1$	$100x_1$[a]	Solid phase[b]
—	—	0.14[c]	3.6×10^{-3}	A
3.83	0.393	0.0124	3.3×10^{-4}	C
6.89	0.728	0.0056	1.53×10^{-4}	C
12.60	1.408	0.0039	1.13×10^{-4}	C
17.10	2.00	0.0037	1.12×10^{-4}	C
22.35	2.77	0.0036	1.16×10^{-4}	C
24.47	3.11	0.0035	1.15×10^{-4}	C
31.34	4.33	0.0025	9.0×10^{-5}	C
34.80	5.02	0.0019	7.1×10^{-5}	C
38.62	5.87	0.0056	2.3×10^{-4}	C
43.60	7.11	0.013	5.5×10^{-4}	B+C
43.82[c]	7.17	—	—	B

[a] Calculated by the compilers.

[b] The solid phases are: A = $Lu(IO_3)_3 \cdot 4H_2O$, B = $LiIO_3$, C = solid solution based on $Lu(IO_3)_3 \cdot 4H_2O$

[c] For the binary systems the compilers computed the following solubility values:

Solubility of $Lu(IO_3)_3$ = 2.00×10^{-3} mol kg^{-1}
Solubility of $LiIO_3$ = 4.29 mol kg^{-1}.

Auxiliary Information

Method/Apparatus/Procedure:

The system was studied by the isothermal method. Equilibrium time was 20–25 d. The concentrations of iodate anion and lutetium cation in aliquots of the coexisting phases were determined; iodate ion was determined by iodometric titration, lutetium in the solid phases by complexometric titration using xylenol orange as an indicator. Lutetium in the liquid phase was determined photometrically using arsenazo III agent after reducing the iodate ion with hydroxylamine, the result was verified spectrographically. The lithium content in the samples was found by difference and verified by flame emission spectroscopy. The solid phases were identified by X-ray diffraction.

Source and Purity of Materials:

"Highly pure" grade lithium iodate was used. Lutetium iodate was prepared from lutetium nitrate and iodic acid.

Estimated Error:

Solubility: reproducibility of 3–8 %.
Temperature: precision ± 0.1 K.

X. ACTINIDE IODATES

A. Thorium Iodate

Components:
(1) Thorium iodate; $\mathrm{Th(IO_3)_4}$; [14693-78-8]
(2) Water; H_2O; [7732-18-5]
or (2) Ammonia (liquid); NH_3; [7664-41-7]

Evaluators:
Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. December 2001.

Critical Evaluation:

The Binary System

There is only one reported solubility measurement (1) of $\mathrm{Th(IO_3)_4}$ in water at 298 K. The value of 2.17×10^{-4} mol dm^{-3} is designated as a tentative solubility.

There is only one reported solubility determination (2) of $\mathrm{Th(IO_3)_4}$ in liquid ammonia at 298 K. The value of 9×10^{-4} mol kg^{-1} is designated as a tentative solubility.

It looks that liquid NH_3 is slightly better solvent for $\mathrm{Th(IO_3)_4}$ than H_2O.

References:

1. G. Spacu and P. Spacu, Z. Anal Chem. **128**, 226 (1948).
2. G. W. Watt, W. A. Jenkins, Jr., and J. M. McCuiston, J. Am. Chem. Soc. **72**, 2260 (1950).

Components:
(1) Thorium iodate; $\mathrm{Th(IO_3)_4}$; [14693-78-8]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
G. Spacu and P. Spacu, Z. Anal. Chem. **128**, 226–9 (1948).

Variables:
One temperature: 298 K

Prepared By:
Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 234. Solubility of $\mathrm{Th(IO_3)_4}$ in water at 25 °C

γ_1 / mg dm^{-3}	$10^4 c_1$ / mol dm^{-3}	$10^{16} K_s$ / mol^5 dm^{-15}
201.2	2.15	1.176
204.9	2.19	1.307[a]
	(av.) 2.17[b]	(av.) 1.23[b]

[a] 1.307 reported by the authors should be read 1.290.
[b] Calculatex by the compilers.

Solubility of thorium(IV) iodate in 0.01 mol dm^{-3} KIO_3 solution at 25 °C was found to be 7.83×10^{-6} mol dm^{-3}; the latter value calculated from the solubility product (K_s) is 6.78×10^{-6} mol dm^{-3} when the activity coefficients according to Debye–Hückel theory were taken into account.

Auxiliary Information

Method/Apparatus/Procedure:
Thorium iodate (0.3–0.4 g) was shaken with water (50 cm^3) at room temperature for 2 h. 10 cm^3 of the saturated solution was added to 1 g KI and 5 cm^3 2 mol dm^{-3} H_2SO_4 solution. The liberated iodine was titrated with 0.1 mol dm^{-3} sodium thiosulfate solution tracing the end point potentiometrically.

Source and Purity of Materials:
Thorium iodate was prepared by mixing thorium nitrate solution with potassium iodate (pure for analysis from Merck) solution.

Estimated Error:
Solubility: precision better than ± 1 %.
Temperature: nothing specified.

Components:

(1) Thorium iodate; $\mathrm{Th(IO_3)_4}$; [14693-78-8]

(2) Ammonia (liquid); NH_3; [7664-41-7]

Original Measurements:

G. W. Watt, W. A. Jenkins, Jr., and J. M. McCuiston, J. Am. Chem. Soc. **72**, 2260–2.(1950).

Variables:

One temperature: 298.2 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

The solubility of $\mathrm{Th(IO_3)_4}$ in liquid ammonia at 25.0 $^\circ\mathrm{C}$ is

0.08 g / 100g NH_3 (authors)
9×10^{-4} mol kg^{-1} (compilers)

$\mathrm{Th(IO_3)_4}$ is the equilibrium solid phase at 25 and -33.5 $^\circ\mathrm{C}$.

Auxiliary Information

Method/Apparatus/Procedure:

Four grams of thorium iodate were treated with 70 cm^3 of liquid ammonia for 3 h, washed, and dried in vacuo. There was no visual evidence of reaction and the starting material was recovered unchanged except for a small retention of ammonia. Analytical results for the solid were as follows: found: Th–24.0; N–0.5 mass % and calculated for $\mathrm{Th(IO_3)_4}$: Th–24.9 mass %. The solubility of $\mathrm{Th(IO_3)_4}$ in ammonia was determined by the method described in (1). The experiments were performed in a glass apparatus placed in a dry box. The solute was placed in a tube and a suitable quantity of the solvent was condensed on the solid sample at ~ -75 $^\circ\mathrm{C}$. The sealed tube was warmed up and placed in a thermostat at the selected temperature for 48 h. The tube was removed, inverted and centrifuged for 3–5 min. The saturated solution filtered was cooled to ~ -75 $^\circ\mathrm{C}$ and the tube was open allowing the solvent to escape and finally evacuated. The deposit of $\mathrm{Th(IO_3)_4}$ was weighed.

Source and Purity of Materials:

All chemicals employed were reagent grade. Thorium(IV) iodate was prepared by the reaction between pure thorium(IV) nitrate and potassium iodate. Analysis of the purified and dried product gave Th–24.6 mass %, calculated for $\mathrm{Th(IO_3)_4}$ formula: Th–24.9 mass %. Commercial liquid ammonia was dried and dispensed as in (1).

Estimated Error:

Solubility: precision ± 12 % (authors of (1)).

Temperature: precision ± 0.1 K (authors of (1)).

References:

[1] G. W. Watt, W. A. Jenkins, and C. V. Robertson, Anal. Chem. **22**, 330 (1950).

B. Uranyl Iodate

Components:

(1) Uranium, bis(iodato-*O*)dioxo (uranyl iodate); $UO_2(IO_3)_2$; [13702-42-6]
(2) Water; H_2O; [7732-18-5]

Evaluators:

Hiroshi Miyamoto, Niigata University, Niigata, Japan and Cezary Guminski, University of Warsaw, Warsaw, Poland. December 2001.

Critical Evaluation:

The Binary System

Only one solubility study for the binary $UO_2(IO_3)_2$–H_2O system has been reported by Artmann (1). Three different methods of preparation of the iodate crystals resulted in different solubility values at the same temperature. The solubility of $UO_2(IO_3)_2 \cdot H_2O$ in stable form in water at 291 K is 1.69×10^{-3} mol dm^{-3}, and it is designated as a tentative value.

A solubility of $UO_2(IO_3)_2 \cdot 2H_2O$ is almost twice higher: 3.3×10^{-3} mol dm^{-3}.

The Ternary System

Solubility data for the $UO_2(IO_3)_2$–KIO_3–H_2O system adjusted at ionic strength of 0.2 at 298 and 333 K have been reported by Klygin et al. (2). The product used in these studies (2) was $UO_2(IO_3)_2 \cdot \frac{4}{3}H_2O$, thus the formula of the equilibrium solid phase needs clarification. The system was studied by the isothermal method. The authors stated that the solubility of $UO_2(IO_3)_2$ in aqueous potassium iodate decreases through the salting-out effect as its concentration is increased and reaches a minimum at $[IO_3^-] = (1.00 \pm 0.18) \times 10^{-3}$ mol dm^{-3}. A further increase in potassium iodate concentration up to 2.09×10^{-3} mol dm^{-3} raises the solubility by $[UO_2(IO_3)_3^-]$ complex formation. The mixed $KUO_2(IO_3)_3 \cdot 3H_2O$ salt, found by Artmann (1) as the equilibrium solid phase, was not identified in this study.

References:

1. P. Artmann, Z. Anorg. Chem. **79**, 327 (1913).

2. A. E. Klygin, I. D. Smirnova, and N. A. Nikol'skaya, Zh. Neorg. Khim. **4**, 1674 (1959); Russ. J. Inorg. Chem. (Engl. Transl.) **4**, 754 (1959).

Components:

(1) Uranium, bis(iodato-*O*)dioxo (uranyl iodate); $UO_2(IO_3)_2$; [13702-42-6]

(2) Water; H_2O; [7732-18-5]

Original Measurements:

P. Artmann, Z. Anorg. Chem. **79**, 327–42 (1913).

Variables:

One temperature: 291 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 235. Solubility of uranyl iodate hydrates in water at 18 °C

Uranyl iodate hydrates[a]	Mass-volume ratio[b]	$10^3 c_1$ / mol dm^{-3} [c]
$UO_2(IO_3)_2 \cdot H_2O$ (Type I, in prisms)	0.1049	1.69
$UO_2(IO_3)_2 \cdot H_2O$ (Type II, in pyramids)	0.1214	1.96
$UO_2(IO_3)_2 \cdot 2H_2O$	0.2044	3.30
$KUO_2(IO_3)_3 \cdot 3H_2O$	0.108	1.74

[a] Uranyl iodate n-hydrates were used to determine the solubility in water.

[b] Mass-volume ratio is the mass (gram units) of $UO_2(IO_3)_2$ per 100 cm^3 of H_2O.

[c] Calculated by the compilers.

Type I of $UO_2(IO_3)_2 \cdot H_2O$ is the stable form at room temperature.

Auxiliary Information

Method/Apparatus/Procedure:

Uranyl iodates and water were shaken in closed bottles at room temperature. The mixtures were filtered after 2 h and the saturated solutions titrated by thiosulfate after addition of KI.

Source and Purity of Materials:

Uranyl iodate monohydrate was obtained from a hot or an acidic solution of uranyl nitrate and iodates of sodium or ammonium. It occurs in two polymorphs. Type I iodate was obtained from acidic solution at room temperature in form of rhombic prisms. Type II iodate was obtained at boiling the solution without an addition of an acid. The latter is transformed into rhombic prisms in presence of acid at ordinary temperature. Uranyl iodate dihydrate was precipitated from solutions of uranyl nitrate and the iodates (except KIO_3). When KIO_3 and $UO_2(IO_3)_2$ solutions are mixed in ratio of 5:1 then $KUO_2(IO_3)_3 \cdot 3H_2O$ is formed.

Estimated Error:

Nothing specified.

Components:

(1) Uranium, bis(iodato-*O*)dioxo (uranyl iodate); $UO_2(IO_3)_2$; [13702-42-6]
(2) Potassium iodate; KIO_3; [7758-05-6]
(3) Hydrochloric acid; HCl; [7647-01-0]
(4) Ammonium chloride; NH_4Cl; [12125-02-9]
(5) Water; H_2O; [7732-18-5]

Original Measurements:

A. E. Klygin, I. D. Smirnova, and N. A. Nikol'skaya, Zh. Neorg. Khim. **4**, 1674–6 (1959); Russ. J. Inorg. Chem. (Engl. Transl.) **4**, 754–6 (1959).

Variables:

Composition of solutions
Temperature: 298 and 333 K

Prepared By:

Hiroshi Miyamoto and Cezary Guminski

Experimental Data

TABLE 236. Solubility of $UO_2(IO_3)_2$ in the $UO_2(IO_3)_2$–KIO_3–H_2O system at ionic strength of 0.2 at 25 or 60 °C

t / °C	$10^3[H^+]$ / mol dm^{-3}	pH [a]	$10^2[IO_3^-]$ / mol dm^{-3}	$10^4 c_1$ / mol dm^{-3} (obsd.)	$10^4 s_1$ / mol dm^{-3} (calcd.)[b]
25	8.32	2.08	1.00	10.4	10.4
(Expt. 1)	7.94	2.10	1.84	3.60	3.62
	7.41	2.13	3.69	1.53	1.42
	6.76	2.17	5.73	1.13	1.09
	6.03	2.22	8.22	0.945	1.06
	5.62	2.25	11.4	0.987	1.13
	5.13	2.29	13.8	1.13	1.21
	4.79	2.32	16.1	1.28	1.30
	4.17	2.38	18.6	1.41	1.41
	3.72	2.43[c]	20.9	1.66	1.51
25	0.0692	4.16	1.17	8.30	7.78
(Expt. 2)	0.0347	4.45	2.09	3.32	2.88
	0.0295	4.40	4.05	1.51	1.31
	0.0151	4.82	6.00	1.14	1.08
	0.0170	4.77	7.99	1.05	1.05
	0.0102	4.99	11.5	1.03	1.13
	0.00708	5.15	14.1	1.13	1.22
	0.00708	5.15	16.1	1.33	1.27
	0.0129	4.89	18.2	1.45	1.39
	0.0178	4.75	20.9	1.69	1.51
60	9.33	2.03	1.36	13.27	13.35
	8.91	2.05	2.15	6.38	6.18
	7.94	2.10	4.16	2.84	2.77

7.08	2.15	6.08	2.29	2.20
6.46	2.18	8.16	1.87	2.06
6.03	2.22	10.2	1.83	2.06
5.75	2.24	11.6	1.93	2.20
5.37	2.27	14.1	2.33	2.20
4.79	2.32	16.5	2.44	2.31
4.37	2.36	18.4	2.48	2.41
3.72	2.43	20.8	2.50	2.55

[a] Calculated by the compilers.

[b] Calculated by the authors from the following fitting equations:

$$\text{25 °C:}\quad s_1 = \frac{9.85\times10^{-6}}{[\mathrm{IO_3^-}]^2} + 5.28\times10^{-5} + 4.58\times10^{-4}[\mathrm{IO_3^-}],$$

$$\text{60 °C:}\quad s_1 = \frac{2.23\times10^{-7}}{[\mathrm{IO_3^-}]^2} + 1.23\times10^{-4} + 6.08\times10^{-4}[\mathrm{IO_3^-}].$$

[c] Reported by the authors.

The authors fitted the solubility data to a smoothing equation assuming that the species in solution were UO_2^{2+}, $UO_2(IO_3)_2$ and $UO_2(IO_3)_3^-$. The solubility of $UO_2(IO_3)_2$ calculated from the smoothing equation at 25 and 60 °C are given in Table 236. K_1 and $1/K_2$ values were also calculated from the obtained smoothing equations.

$$UO_2(IO_3)_2 \rightleftharpoons UO_2^{2+} + 2IO_3^-; \qquad K_1 = \frac{[UO_2^{2+}][IO_3^-]^2}{[UO_2(IO_3)_2]}$$

$$= 1.86 \times 10^{-3}\ \text{mol}^2\ \text{dm}^{-6}\ (\text{at } 25\ ^\circ\text{C})$$

$$= 1.81 \times 10^{-3}\ \text{mol}^2\ \text{dm}^{-6}\ (\text{at } 60\ ^\circ\text{C})$$

$$UO_2(IO_3)_2 + IO_3^- \rightleftharpoons UO_2(IO_3)_3^-; \qquad 1/K_2 = \frac{[UO_2(IO_3)_2][IO_3^-]}{[UO_2(IO_3)_3^-]}$$

$$= 0.116\ \text{mol dm}^{-3}\ (\text{at } 25\ ^\circ\text{C})$$

$$= 0.202\ \text{mol dm}^{-3}\ (\text{at } 60\ ^\circ\text{C})$$

The detailed information of the calculation procedure for the smoothing equation has been described in the original paper.

Auxiliary Information

Method/Apparatus/Procedure:

The solubility of $UO_2(IO_3)_2$ in water containing KIO_3 and HCl was studied by the isothermal method. The ionic strength was kept at 0.2 with NH_4Cl. In the experiment I at 25 °C and in that at 60 °C, pH was adjust to 2.03–2.43 by addition 10 cm^3 of 0.1099 mol dm^{-3} HCl. In the experiment II at 25 °C the dissolved salts decreased acidity to pH of 3.80–4.75. The mixtures in graduated flasks were shaken automatically in a thermostat for 2 d. The saturated solutions were filtered through a G4 filter. The uranium content in the filtrates was determined photocolorimetrically with arsenazo reagent, and the iodate iodometrically. The pH was measured with a pH meter equipped with a glass electrode. The solubility was calculated by a formula relating the solubility with iodate concentration.

Source and Purity of Materials:

Potassium iodate was recrystallized twice. 42.8 g (0.2 mol) of the potassium iodate was added to 200 cm^3 of hot water. The aqueous KIO_3 solution was added to 100 cm^3 of 1 mol dm^{-3} uranyl nitrate solution. The precipitate was filtered off after cooling to room temperature, washed 6 times with water, and dried in air to constant weight. The product was analyzed for iodate by iodometry and for uranium by ignition and weighing as U_3O_8. The air-dried material contained 41.63 mass % UO_2^{2+} and 54.53 mass % IO_3^-, which corresponds to formula: $UO_2(IO_3)_2 \cdot \frac{4}{3}H_2O$. Distilled water was used.

Estimated Error:

Solubility: nothing specified.
Temperature: precision within ± 0.1 K.

System Index

Page numbers written in boldface refer to evaluation text, whereas lightfaced roman type refer to compilations.

Registry Number Index

Page numbers written in boldface refer to evaluation text, whereas lightfaced roman type refer to compilations.

Author Index

Page numbers written in boldface refer to evaluation text, italicized ones refer to auxiliary information of data sheets, whereas lightfaced roman type refer to compilations.