Developing and Maintaining a Laboratory Quality Management System

Version 3.0

Student Manual

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An eLearning Course by the American Industrial Hygiene Association

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Foreword

Thank you for purchasing the Developing and Maintaining a Laboratory Quality Management System self-study course.

Course format

The program is divided into 12 chapters dealing with different aspects of the quality assurance/quality control (QA/QC) process. Each chapter contains modules designed to teach a specific concept. The course includes a Learning Agenda, this Student Manual, Reading Materials, and a final exam/evaluation. This Student Manual contains information designed to introduce the student to the topics essential to a well-designed laboratory quality system that is compliant with ISO/IEC 17025 and the American Industrial Hygiene Association (AIHA) Laboratory Accreditation Programs (AIHA-LAP, LLC) requirements. Those laboratories seeking accreditation should be certain to review all current requirements of the accreditation body prior to applying for accreditation since requirements may change.

This Student Manual includes learning objectives, and competency exercises designed to reinforce the material presented in the course and is designed to lead the student through the learning process. The Reading Materials include ISO/IEC 17025:2005, selected AIHA-LAP, LLC policy modules, attachments, and appendices. The attachments included and referenced in the chapters of the Student Manual are mandatory reading and the student may be tested on the contents of these attachments. The appendices are suggested reading and for reference purposes only. The student will not be tested on the contents of the appendices.

Final Exam & Evaluation

The final exam will test your knowledge of the materials you have studied. The final exam should be taken by yourself, working alone, but you may consult tables, graphs, charts, calculator, manuals, course materials and other references as the need arises.

- Exam
- Evaluation

Once completed, you will receive notification of your updated Education Transcript with CM, COC and CEU credit for this course.

Scoring

Final Exam scoring is as follows:

- 90-100% Excellent
- 80-89% Good
- 70-79% Fair
- <70% Exam must be retaken

Retaking the Final

If you fail to achieve 70% on your first try, you may retake the Final Exam after re-reviewing and re-studying course materials. If you fail to score 70% on the second test, you will not be awarded CM, CEU or COC credit.

Course Credit

The course will award 40 CM credit/contact hours, 4.0 CEU and 4.0 COCs.

Completion Time

You have 6 months from the date of purchase to complete the course and exam. If more time is needed please contact the AIHA eLearning Program Assistant at **dlassistant@aiha.org**.

Contents

Chapter 1

Laboratory (Quality) Management System

Learning Objectives:

After completing this chapter, the student should be able to:

- Define the purpose and importance of a quality management system
- Identify the current document defining general requirements for testing and calibration laboratories and the basis of this document
- Identify key elements of ISO/IEC 17025:2005
- Understand key terms in ISO/IEC 17025:2005 and AIHA-LAP, LLC policy modules

Introduction

Laboratory analysts use the science of quality measurements. Every aspect of the analyst's job is designed for the ultimate production of data. Data result from some form of measurement. As laboratory instrumentation becomes more and more sophisticated, measurement techniques have become more and more sensitive and precise. Analytical techniques that a few years ago resulted in detection limits of analytes in the milligram range now more frequently report in the microgram or nanogram range. The data produced by these measurements frequently are used in major decision-making processes affecting human health and high-cost remediation and/or process engineering projects. These uses create a great need to ensure and document the quality of the data. This study program explores the elements necessary to ensure that quality data are produced and appropriate procedures and records are maintained to support these data.

The term "quality assurance" (QA) describes a system of activities which is used to ensure the quality of data and to provide evidence of data quality to the producer or end user of the data. Quality assurance consists of two related, but separate, activities. "Quality control" (QC) describes the activities and procedures used to produce consistent data of known accuracy and precision. "Quality assessment" describes the activities and procedures used to evaluate the quality of the data that are produced. Therefore, it is essential for a laboratory to establish a quality system that ensures the quality of the data produced by the analytical processes. Development of a quality system can help the laboratory balance data production activities with activities that reduce or eliminate errors in the produced data.

To be effective, quality systems must be more than token statements that the laboratory will perform all procedures in a manner that will produce the highest quality data. The quality system must include a written program detailing laboratory policies and procedures and incorporate a

number of very important elements. The American Industrial Hygiene Association (AIHA) has been accrediting laboratories since 1974. Since the mid 1990's, AIHA's laboratory accreditation programs have incorporated the recommended practices in ISO/IEC Guide 25 "General requirements for the competence of testing and calibration laboratories." In 1999, this guide was updated to incorporate the requirements of ISO 9000:2000 series quality system certifications and was published as a consensus Internal Standard ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories." This standard has been adopted as the primary basis for accreditation by most U.S.-based and international accrediting bodies. This standard was adopted by AIHA in 2001 as an integral component of its laboratory accreditation programs. ISO/IEC 17025 was revised again in 2005 to align it with the principles of ISO 9001 that was revised in 2004. The 2005 edition is still in effect, but revisions are in the draft stage. It is expected that revisions to ISO/IEC 17025 will be finalized in 2017 and will be that the revised standard will be implemented by accreditation bodies over the course of two years following finalization. Laboratories seeking accreditation by the American Industrial Hygiene Association-Laboratory Accreditation Programs, LLC (AIHA-LAP, LLC) are currently required to comply with all aspects of the current editions of ISO/IEC 17025 and applicable current AIHA-LAP, LLC Laboratory Accreditation Program policy modules. Because quality is such an integral component of the overall laboratory management system, ISO/IEC 17025 refers to the laboratory quality system as the laboratory management system.

ISO/IEC 17025:2005 is organized in the following manner:

- Forward
- Introduction
- 1. Scope
- 2. Normative References
- 3. Terms and Definitions
- 4. Management Requirements
- 5. Technical Requirements

Information included through Section 3 provides background regarding the standard, scope and applicability, alignment with other international standards, and reference to other documents and standards that form an integral part of the standard. It is important to understand that the notes provided within the standard are not mandatory but are included as guidance and clarification. Sections 4 and 5 include the management and technical requirements and are organized as follows:

- 4. Management Requirements
- 4.1 Organization
- 4.2 Management System
- 4.3 Document Control
- 4.4 Review of Requests, Tenders and Contracts

- 4.5 Subcontracting of Tests and Calibrations
- 4.6 Purchasing Services and Supplies
- 4.7 Service to the Customer
- 4.8 Complaints
- 4.9 Control of Nonconforming Testing and/or Calibration Work
- 4.10 Improvement
- 4.11 Corrective Action
- 4.12 Preventive Action
- 4.13 Control of Records
- 4.14 Internal Audits
- 4.15 Management Reviews

- 5 Technical Requirements
- 5.1 General
- 5.2 Personnel
- 5.3 Accommodation and Environmental Conditions
- 5.4 Test Methods and Method Validation
- 5.5 Equipment
- 5.6 Measurement Traceability
- 5.7 Sampling
- 5.8 Handling of Test and Calibration Items
- 5.9 Assuring the Quality of Test and Calibration Results
- 5.10 Reporting the Results

Additional 2016 AIHA- LAP, LLC requirements are detailed in the policy modules as follows:

- Module 1– Accreditation Overview
- Module 2A– General Management System Requirements
- Module 2B– IHLAP Additional Requirements
- Module 2C– ELLAP Additional Requirements
- Module 2D– EMLAP Additional Requirement
- Module 2E– Unique Scopes Additional Requirements
- Module 2F– FoodLAP Additional Requirements
- Module 3– Accreditation, Maintenance and Reaccreditation Processes
- Module 4– Suspension, Denial, or Withdrawal of Accreditation
- Module 5– Appeals Process
- Module 6– Proficiency Testing (PT) and Round Robin Programs

- Module 7– Reference to Accreditation and Advertising
- Module 8– Miscellaneous
- Module 9– Terms and Acronyms
- Appendix A-Reserved
- Appendix B-Reserved
- Appendix C-Reserved
- Appendix D-Reserved
- Appendix E-Reserved
- Appendix F-Reserved
- Appendix G Estimation of Uncertainty of Measurement
- Appendix H Traceability of Measurement

Students will be directed to read applicable sections of ISO/IEC 17025:2005 and AIHA-LAP,LLC policies in the Introduction of subsequent chapters of the Student Manual.

Please note that ISO/IEC 17025 is for both testing and calibration laboratories. AIHA-LAP, LLC laboratories are testing laboratories. Calibration laboratories are those that typically calibrate equipment or reference standards. When referring to ISO/IEC 17025, most instances of "calibration" can be ignored unless the testing laboratory is performing its own internal calibration of equipment, reference materials or reference standards.

When utilizing a standard such as ISO/IEC 17025, it is very important to understand the terminology used. A number of terms may have multiple meanings and interpretations. The student should refer to the AIHA-LAP, LLC Module 9 for definition of terms. AIHA-LAP, LLC's definitions of policy and procedure and interpretation of other terms contained within the standard are of critical importance in understanding the standard and its requirements. Module 9 includes the following:

"Policy—An organization's written statement of commitment to implement a management program element."

"Procedure—A written set of instructions that describes how to implement a policy requirement, or how to carry out a specific task."

A policy can be considered "what" the organization intends to do, while a procedure can be considered the "who, how, when, where and in what manner" a specific task is carried out. What may be most familiar are test methods or analytical procedures. While these are often detailed and technical in nature, they share commonality with other procedures (including quality management system procedures) in that they address the "who, how, when, where and in what manner," and what records are generated when an analytical test or sample preparation is conducted. Bear this in mind when creating required policies and procedures.

Other terms that you will encounter and their meanings as interpreted by AIHA-LAP, LLC include:

- **Arrangement** – a general description of how an outcome will be achieved (must be included in a written or electronic document).
- **Define** – explain or specify details (must be included in a written or electronic document).
- **Document (noun)** – a written or electronic arrangement, policy, procedure, form, work instruction, reference text, standard or software that describes how the laboratory operates. Subject to document control procedures. Documents can also take the form of instructional audio or video productions.
- **Document (verb)** – same as **Record** (verb).
- **Ensure** – this is a performance requirement. This requirement does not have to be included in a laboratory's written documentation. A nonconformity written against such "ensure" clauses requires objective evidence of failure to achieve a required outcome.
- **Programme (noun)** – a schedule and procedure.
- **Record (verb)** – to set down in writing or electronically: furnish written or electronic evidence of (copies of forms or templates that contain data become records).
- **Record (noun)** – written or electronic history of what was done (objective evidence).
- **Shall** – mandatory (must).
- **Should** – not mandatory but strongly recommended.
- **Specify** – define (must be included in a written or electronic document).

There is often confusion regarding the difference between documents and records and how they must be controlled. The easiest way to think of these is that documents are instructional. They include policies, procedures, external source documents, forms, software applications, etc. that provide direction and instruction as to how to complete a task. Control of documents is further described in Chapter 3 under ISO/IEC 17025 Section 4.3.

Records are not instructional, but rather provide a history and evidence of what has been done. Raw instrument data and printouts, completed bench sheets, completed checklists, completed forms, QC data, completed (populated) spreadsheet applications, final reports, management and quality reports are examples of records. The control of records is further discussed in Chapter 5 under ISO/IEC 17025 Section 4.13.

The chapters of this reading manual are organized to follow the structure of ISO/IEC 17025 and AIHA-LAP, LLC Policy Modules.

Chapter 1 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 153.

1. **What is the purpose of a quality management system?**

2. **Explain the differences between quality assurance, quality control and quality assessment.**

3. **What document are the AIHA-LAP, LLC and many other accrediting organizations using to define the basic requirements of a quality system for accredited laboratories?**

4. **List the subsections included within the management requirements of ISO/IEC 17025:2005.**

5. **List the subsections included within the technical requirements of ISO/IEC 17025:2005.**

6. **Explain the difference between policy, procedure and arrangement.**

Chapter 2

Organization (4.1) and Management System (4.2)

Learning Objectives:

After completing this chapter, the student should be able to:

- Understand requirements related to the laboratory operation and laboratory management responsibilities and authorities
- Understand the purpose of an organization chart
- Define the basic and required positions in a laboratory organization and understand where the duties and responsibilities of each position are to be defined
- Identify the required elements of the quality management system and the purpose of each
- Understand the required elements of the quality manual
- Identify the required elements of the laboratory's quality policy statement

Introduction

Sections 4.1 through 4.15 of ISO/IEC 17025:2005 (referenced within this document as "the standard") specify management system requirements. AIHA-LAP, LLC Modules include additional accreditation requirements. The student should read sections 4.1 and 4.2 of ISO/IEC 17025 and Module 1 and Module 2A, sections 2A.1 to 2A.4.2 of the AIHA-LAP, LLC policies in conjunction with reviewing the sections presented in this chapter.

4.1 Organization

This section of the standard describes the applicability of the standard, expectations of the organization and other organizational elements. Note that the standard is focused largely on the needs of the customer. The following are highlighted areas of section 4.1:

- **4.1.4:** *If the laboratory is part of an organization performing activities other than testing* (e.g. quality control laboratory, IH lab for a manufacturing facility, lab for an engineering or consulting firm) *the responsibilities of key personnel in the organization that have an involvement or influence on the testing activities of the laboratory shall be defined in order to identify potential conflicts of interest.*

  This requirement can be satisfied through an available job description or the responsibilities of key personnel may be defined in the quality manual.

- **4.1.5.a:** *The laboratory shall have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests, and to initiate actions to prevent or minimize such departures (see also 5.2).*

  The authority to carry out these duties is often overlooked. These authorities can be stated in the quality manual or included in job descriptions.

- **4.1.5.b:** *The laboratory must have arrangements to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work.*

  AIHA-LAP, LLC requires that these arrangements are documented and often take the form of conflict of interest agreements, isolation of testing personnel from customer contact, and review and management of laboratory workload to reduce potential pressure on analysts. A process should be in place to report and deal with any actual or perceived conflicts of interest. This is often a human resources element.

- **4.1.5.c:** *The laboratory shall have policies and procedures to ensure the protection of its customers' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results.*

  The laboratory's policy must be included in the quality manual. A procedure(s) is required and can include such elements as employee confidentiality agreements, identification of samples with only laboratory-generated sample numbers, password protection of electronic records and reports, and confidentiality statements on all forms of external communication (faxes, email and final reports).

- **4.1.5.d:** *The laboratory shall have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity.*

  The laboratory's policy must be included in the quality manual. A procedure(s) is required and often takes the form of "ethics" requirements or procedures and may be part of the employee handbook. This is often a human resources element. A process should be in place to report and deal with any actual or perceived integrity issues.

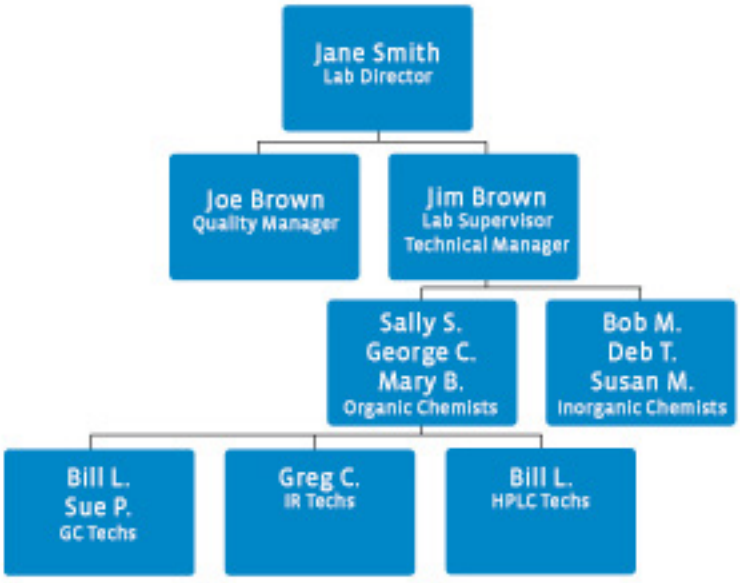
- **4.1.5.e:** *The laboratory shall define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services.*

  This is typically defined in an organizational chart. Relationships between quality management, technical operations and support services are most easily defined through a brief narrative. AIHA-LAP, LLC requires this section to be included in the quality manual.

- **4.1.5.f:** *The laboratory shall specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the tests and/or calibrations.*

  These responsibilities, authorities and interrelationships can be stated in the quality manual or defined in job descriptions or position descriptions. Quality systems are more effective when each individual in the organization understands his or her particular responsibilities and how to report problems when they occur. Laboratory organizational structures can be complex for large companies or extremely simple for small laboratories. However, there are some similarities between all laboratory organizational structures. A sample organizational chart is shown in Figure 2.1.

Figure 2.1 – Sample Organizational Chart



- **4.1.5.g:** *The laboratory shall provide adequate supervision of testing and calibration staff, including trainees, by persons familiar with methods and procedures, purpose of each test, and with the assessment of the test results.*

  This does not require documentation other than evidence that staff is adequately supervised by qualified staff members. Some of the responsibilities which may be assumed or delegated by laboratory management include ensuring that all laboratory personnel are properly trained in laboratory procedures including those procedures associated with sample receipt, storage, analyses, data interpretation, report generation and QA/QC.

- **4.1.5.h:** *The laboratory shall have technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operations.*

  This responsibility can be stated in the quality manual or defined in job descriptions or position descriptions. Whether the title is laboratory director, supervisor or technical manager, one individual generally has overall responsibility for the operation of the laboratory. This responsibility includes the establishment, implementation and ongoing maintenance of a laboratory quality system. Ultimately, this individual is responsible for the quality of the data produced by the laboratory. Larger organizations may also have one or more laboratory supervisors who are responsible for the daily operations of the laboratory or a section of the laboratory. In smaller organizations, the laboratory manager will also be responsible for supervising the daily operations of the laboratory. Specific responsibilities should be established for the laboratory manager and laboratory supervisor(s) based on the laboratory's organizational structure. AIHA-LAP, LLC has established minimum educational and experience requirements for the technical manager and permits multiple technical managers. These requirements are detailed in Section 2A.5.2 of AIHA-LAP, LLC policies and program specific modules.

- **4.1.5.i:** *The laboratory shall appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times; the quality manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources.*

  This specific responsibility and authority can be stated in the quality manual or defined in job descriptions or position descriptions. It is important that the quality manager has direct access to the top management of the laboratory. The quality manager (QM) is responsible for the quality system at the laboratory. In a larger organization, the QM position may be a full-time position with all the QM's time devoted to quality activities. At smaller laboratories, the QM may be assigned other activities such as laboratory management, analyses or sample receipt and

data entry. The QM must be qualified by experience and education to implement the quality system. AIHA-LAP, LLC has established minimum educational and experience requirements for the quality manager. These requirements are detailed in Section 2A.5.2 of AIHA-LAP, LLC policies.

- **4.1.5.j:** *The laboratory shall appoint deputies for key managerial personnel.*

  The appointment of deputies (in the absence of key managerial personnel) can be defined in the quality manual, job descriptions or can be done on an ad hoc basis. If assigned on an ad hoc basis, the mechanism should be defined within the quality system.

- **4.1.6:** *Top management shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.*

  Meetings and memos are typical ways of communicating the effectiveness of the management system. Communication of the results of the annual internal quality audit and the management review as appropriate, with laboratory staff assists in meeting the communication requirement, although more frequent communication than annual is recommended.

4.2 Management System

The written quality system documentation serves as the base for the laboratory's (quality) management program. It must be comprehensive in defining and specifying required elements of the quality program that include arrangements, policies, procedures and programs. (See AIHA-LAP, LLC Policy Module 9.) The management (quality) system is typically a collection of documents. ISO/IEC 17025 requires that the laboratory quality policy statement, all policies related to quality, and several addition elements are included in the laboratory quality manual. AIHA-LAP, LLC requires that the quality manual is updated whenever necessary and is reviewed and approved by management at least annually. The required and suggested elements of the quality manual are summarized in the following sections.

- **Title Page** – It is recommended that the title, revision number and/or date and applicable laboratory location(s) are included on the title page. The approval of top management, the technical management and quality management are also recommended. Inclusion of this section in the quality manual is a suggestion.
- **Table of Contents** – When using controlled page numbering (i.e., page 1 of 33) for the entire document, simple reference to the appropriate page number is all that is required. If sections are revised on varying schedules, the revision of the current section is required in the table of contents and total page numbers for that section is recommended. Inclusion in the quality manual is a suggestion.
- **Quality Manual Maintenance and Update Procedures** – This section can address details on periodic reviews and updating of the written quality manual and any other required documentation. Inclusion of this section in the quality manual is a suggestion.

- **Customers' Confidential Information and Proprietary Rights** – This section must address the laboratory's policy regarding protection of customers' confidential information and proprietary rights. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.1.5.c.)
- **Impartiality and Operational Integrity** – This section must address the laboratory's policy regarding maintaining impartiality and operational Integrity. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.1.5.d.)
- **Organization and Responsibility** – The laboratory's quality system must include a description of the organizational structure (or a detailed organizational chart) describing the reporting structure of the laboratory and inter-relationships of staff and other support operations. Inclusion in the quality manual is a suggestion. The chart should include clear reporting lines for the quality manager. The technical management (technical manager) and the quality manager (or equivalent) must be specified and his or her duties and responsibilities and authorities detailed. These roles and responsibilities must be included in the quality manual, as opposed to referencing another quality system document. The quality manual must also define the responsibilities of quality and/or technical management for ensuring compliance with ISO/IEC 17025. Quality responsibilities and authorities should be summarized for all other positions on the organizational chart. The chart and duties/responsibilities should be updated as required. The appointment of deputies for key technical personnel (quality manager or technical manager) can be discussed in this section. (See Sections 4.1.5.e,f,g,h,i,j and 4.2.6.)
- **Quality Assurance Objectives and Policies** – A quality policy statement is required and must address the five elements in ISO/IEC 17025 Section 4.2.2 a-e. This statement must be issued under the authority of top management of the laboratory. Signature endorsement of the quality policy statement or signature endorsement of the entire quality manual by top laboratory management satisfies this requirement. This section must state the objectives of the quality system and detail the policies that will be used to obtain those objectives. Measurable objectives are preferable. Note that the quality objectives must be established and reviewed (See Section 4.2.2) as part of the management review. (See Section 4.15.1.) This is not stated in the management review section of the standard and is often overlooked by laboratories.
- **Review of Requests, Tenders, and Contracts** – This section must address the laboratory's policy regarding the review of requests (for analytical services), tenders (proposals), and contracts. (See Section 4.4.) (NOTE: All work done by a laboratory is considered a contract unless performed exclusively for internal laboratory use. This includes oral contracts and routine sample submittals in which case the sample submittal form constitutes the contract.) The laboratory's associated procedure(s) must be included or referenced.
- **Purchasing of Services and Supplies** – This section must address the laboratory's policy for the selection and purchasing of services and supplies it uses that affect the quality of tests. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.6.)

- **Complaints** – This section must address the laboratory's policy for the resolution of complaints received from customers or other parties. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.8.)
- **Control of Nonconforming Testing Work** – This section must address the laboratory's policy that shall be implemented when any aspect of its testing work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.9.)
- **Corrective Action** – This section must address the laboratory's policy for implementing corrective action when nonconforming work or departures from the policies and procedures in the management system or technical operations have been identified. The laboratory's associated procedure(s) must be included or referenced. (See Section 4.11.)
- **Personnel Qualifications and Training** – This section must include the laboratory's policy for providing training and identifying training needs. It must include or reference laboratory procedures for providing training and identifying training needs. Periodic performance reviews, internal audits and the monitoring of routine quality control and proficiency testing activities are ways to identify training needs. These procedures are also a good place to specify minimum internal annual continuing education requirements for each position, as applicable. (See Section 5.2.)
- **Reference to other Management System Documentation** – Section 4.2.5 of the standard specifies that "The quality manual shall include or make reference to the supporting procedures including technical procedures. It shall outline the structure of the documentation used in the management system." This can be accomplished in a single section of the quality manual, although it is also preferable to include references to specific supporting procedures within each applicable section of the quality manual.

Section 4.2.7 of the standard states, "Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented." In order to maintain compliance with this requirement, the laboratory must make sure the updating and/or reference to other related quality system documentation is made when changes are made to the quality manual, and that changes in personnel or responsibilities and inter-relationships are updated when any of these changes are made. No policy or procedure is required by the standard for this requirement.

Chapter 2 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 155.

1. **According to ISO/IEC 17025:2005, what are the primary responsibilities of the laboratory?**

2. **What is the purpose of a laboratory organizational chart?**

3. **Identify the principal positions in a laboratory organization and indicate where the duties and responsibilities for each position are defined.**

4. **Which of the following elements is not normally part of the written laboratory quality management system?**

    a. Quality policy statement
    b. Personnel training procedures
    c. Employee pay scales
    d. Audit procedures
    e. Confidentiality policy
    f. Procurement procedures

5. **What sections of ISO/IEC 17025 and AIHA-LAP, LLC policies designate the required content of the laboratory quality manual?**

6. **What are the five elements that must be included in the laboratory's quality policy statement and where can they be found?**

7. **Under whose authority is the quality policy statement issued?**

8. **True or false: All quality system policies and procedures must be included in the quality manual.**

   ☐ True
   ☐ False

Chapter 3

Document Control (4.3) through Complaints (4.8)

Learning Objectives:

After completing this chapter, the student should be able to:

- Understand the purpose and requirements of a document control program
- Identify the types of documents that must be controlled
- Understand the use of requests, tenders and contracts
- Understand the requirements for subcontracting analytical work
- Identify the requirements for purchasing quality-affecting items and services
- Define arrangements the laboratory must have for servicing its customers
- Understand how to deal with quality-related complaints from all parties

Introduction

Sections 4.1 through 4.15 of ISO/IEC 17025:2005 (referenced within this document as "the standard") specify quality management system requirements. AIHA-LAP, LLC Modules include additional accreditation requirements. The student should read Sections 4.3 through 4.8 of ISO/IEC 17025 and Module 2A, Sections 2A.4.3 through 2A.4.8 of the AIHA-LAP, LLC policies in conjunction with reviewing the sections presented in this chapter.

4.3 Document Control

This section of the standard describes general requirements for the control of both internally generated and external source documents. As described in Chapter 1, documents are considered instructional and include arrangements, policies, procedures that indicate what is to be done, or how it is to be conducted. Section 4.3.1 of the standard states, "The laboratory shall establish and maintain procedures to control all documents that form part of its management system (internally generated or from external sources), such as: regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals." Note 1 in this section of the standard further describes various types of documents. Note 1 states, "In this context "document" could be policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written." Note 2 in this section further states, "The control of data related to testing and calibration is covered in

5.4.7. The control of records is covered in 4.13." This note is intended to help clarify that data and records are subject to different requirements than documents.

As one can see, there are many types of documents that must be controlled by the laboratory. However, the listing in the standard is not complete. Forms (such as analytical request forms, purchasing forms, bench sheets, temperature and balance logs, etc.) are also documents and must be controlled as such. This also holds true for electronic forms and templates that may be used for recording and calculating data, quality control samples, and for generating reports. Examples of some of the types of documents that are common to most laboratories are listed below:

- Regulations (e.g., OSHA Lab Standard)
- Standards (e.g., ISO/IEC 17025, AIHA-LAP, LLC Policy modules)
- Test and/or calibration methods (e.g., NIOSH, OSHA methods, Lab SOPs)
- Drawings (probably not applicable)
- Software (e.g., instrument operating, chromatographic, LIMS, spreadsheet applications, electronic forms, report templates)
- Instructions (e.g., manufacturers' equipment operating instructions)
- Manuals (e.g., quality manual, equipment maintenance manual/software manuals)
- Policies
- Procedures
- Forms (hard copy and electronic)
- Postings (Method Summaries)

Document control procedures can vary greatly based upon the size of an organization or laboratory. Several suppliers have developed software applications specifically to control documents. The most significant requirements of the standard are included below with additional comments:

- **4.3.2.1** – All documents issued to personnel in the laboratory as part of the management system must be reviewed and approved for use by authorized personnel prior to use.

Review is typically conducted by the technical and/or quality management. An approval signature line with date is recommended.

- **4.3.2.1** – A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the management system shall be established and be readily available to preclude the use of invalid and/or obsolete documents.

The master list must include both internally generated and external source documents. This is typically maintained as an actual list in tabular format (using either word processing or spreadsheet

applications); however, the master list may take the form of a directory structure in laboratories where all documents are maintained electronically. The current revision status can be the date or revision identification of internally generated documents and is typically the publication date/and or revision number for external source documents. The master list must be readily available to affected staff so they can determine the current approved revision of the controlled document and the distribution (location) of the document.

- **4.3.2.2** – The procedure(s) adopted shall ensure that:

  a. authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;
  b. documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;
  c. invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;
  d. obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

These requirements are fairly straight forward. It is important to note that the laboratory's document control procedures don't need to address or include these elements, but rather only ensure that these requirements are met. It is however advisable to address each of these requirements within the document control procedures to help "ensure" that these requirements are satisfied.

- **4.3.2.3** – Management system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, the total number of pages or a mark to signify the end of the document, and the issuing authority(ies). Most laboratories use control page number (e.g. page 1 of 27) to satisfy page numbering requirements, although a sequentially paginated document with "end of document" on the last page also satisfies this requirement. Note that these requirements only apply to documents generated by the laboratory and do not apply to external source documents.

Sections 4.3.3.1 and 4.3.3.3 are straight forward and do not require further discussion.

- **4.3.3.2** – Where practicable, the altered or new text shall be identified in the document or the appropriate attachments. The intent of this requirement is that the user of the document can easily identify what changes have been made. This can be accomplished using commercially available software (such as Word track changes feature), or by bolding, highlighting, or underlining of new text, or any other method of identification. Some type of method of identification needs to be used to identify the changes from the prior to the current revision of the document. A revision history that is included as part of the document can also detail the changes that have been made.

- **4.3.3.4** – Procedures shall be established to describe how changes in documents maintained in computerized systems are made and controlled. This is applicable to documents (such as test methods, policies, procedures) maintained electronically on networks, electronic forms and templates, LIMS, etc. Procedures describing how changes are made and controlled could include such elements as authorized individuals, the use of password or network authorizations, and applying read-only attributes to require the renaming of subsequent revisions of documents.

4.4 Review of Requests, Tenders and Contracts

The requirements in this section of the standard are clear, however, the full extent of these requirements is often misunderstood by laboratories. AIHA-LAP, LLC considers any analytical work that is conducted by the laboratory to be a contract. A contract can be written or verbal. Under the simplest circumstances, samples accepted by the laboratory for analysis constitute a contract to conduct work. Requests are considered request for quotations and proposals, tenders are laboratory proposals or quotations to conduct the requested work and the contract can be a formal document or, in the case indicated above, simply an accepted analytical request form or chain of custody form.

As with document control requirements, the policies and procedure for review of requests, tenders and contracts leading to a contract for testing shall ensure that:

a. the requirements, including the methods to be used, are adequately defined, documented and understood (see 5.4.2);
b. the laboratory has the capability and resources to meet the requirements;
c. the appropriate test and/or calibration method is selected and is capable of meeting the customers' requirements (see 5.4.2).

Although procedures are not required to address or include these elements, it is advisable to address each of these requirements within the laboratory procedures to help "ensure" that these requirements are satisfied.

Note that records of review must be maintained. Defining the acceptance of routine samples by sample receiving staff as documentation of the review of routine projects or review by the analyst may be acceptable provided that requests for either large numbers of samples or rapid turnaround are considered when evaluating the laboratory capabilities and resources to meet those requirements. In those instances, a record of management, supervisor or analyst review is required.

The laboratory must also maintain records of pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract. Laboratories typically meet this requirement by documenting discussions in a standardized "customer communication log," on the analytical request form, with e-mail communication, or within the LIMS. The date, name of customer, name of laboratory representative and outcome of the discussion should

be documented. Some laboratories request that all communications related to requests for work or results are documented in writing either through the use of facsimile or e-mail—this is a good practice.

4.5 Subcontracting of Tests

This section of the standard can be summarized as requiring that the customer is advised of the subcontract arrangement in writing and approves the subcontracting of the customer's samples and that samples are subcontracted to a competent subcontractor. The lab is responsible for the work unless the subcontractor is specified by the customer or a regulatory authority. The laboratory must maintain a register (list) of subcontractors and a record of compliance with ISO/IEC 17025. A laboratory's accreditation certificate satisfies the record of compliance. Furthermore, AIHA-LAP, LLC accredited laboratories must subcontract samples to a laboratory accredited by AIHA-LAP, LLC or other ILAC MRA Signatory and advise the customer of the subcontract arrangement in writing, including the subcontractors' accreditation credentials (scope and accrediting body).

It should be noted that these requirements for subcontracting only apply to the laboratory's accredited fields of testing (FoTs). If the laboratory does not subcontract samples within its accredited FoTs, then these requirements are not applicable although it is advisable to use accredited laboratories for FoTs outside the laboratory's accredited FoTs.

4.6 Purchasing Services and Supplies

Section 4.6.1 states, "The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the tests and/or calibrations. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the tests and calibrations."

Procedures for the selection and purchasing of services and supplies can vary greatly between organizations. What is most important is that qualified suppliers are selected and utilized for both services and supplies. Selection criteria are up to the laboratory, but the following list is provided for consideration:

- QS registration (ISO 9000/9001)
- Accreditations (ISO/IEC 17025, ISO/IEC 17043, ISO Guide 34)
- Original Equipment Manufacturer (OEM)
- Past Experience
- Industry standard
- Sole Source
- Meets Requirements

Laboratories often overlook service suppliers that include calibration laboratories, equipment service and maintenance firms, ventilation testing firms, proficiency testing providers, etc.

Specific procedures are also required for the purchase, reception and storage of reagents and laboratory consumable materials. AIHA-LAP, LLC has some additional requirements related to the receipt of standards and reagents and traceability in AIHA-LAP, LLC Sections 2A.5.6.1 though 2A.5.6.6.

Section 4.6.2 requires that purchased supplies and reagents and consumable materials are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the tests and/or calibrations concerned and that records of actions taken to check compliance are maintained. This can involve testing materials for suitability prior to use; however, records of checks of products received (packing lists) and associated certificates of analysis (as applicable) vs. requested products on purchasing documents satisfy this requirement.

Purchasing documents must contain sufficient detail to clearly identify the requested item and required grade. Supplies are often specified by catalog number and this is acceptable. The standard requires review and approval of the purchasing documents for technical content prior to release. Many laboratories order and purchase items and services through the Internet. If that is the case, a procedure must be in place to review the purchase request for technical content prior to release. Having technically qualified individuals placing the order can satisfy this requirement.

The laboratory is required to evaluate suppliers of critical consumables, supplies and services and to maintain records of these evaluations and list those approved. The records of evaluation may include detailed questionnaires and product quality checks, but the extent of evaluation is up to the laboratory.

4.7 Service to the Customer

The standard does not require any policy or procedure for this section. The section does however require the laboratory to be willing to cooperate with customers and to seek feedback, both positive and negative, from its customers. Customer feedback can be solicited through periodic customer surveys, links for feedback on laboratory websites, or questionnaires included with customer reports. This feedback is a required element to be taken into account during management reviews. Laboratories should not underestimate the value of periodic personal communication (either face-to-face or via telephone) to elicit frank feedback regarding services.

4.8 Complaints

The laboratory shall have a policy and procedure for the resolution of complaints received from customers and other parties. The “other parties” are often overlooked by laboratories. Other parties can include employees, end users of data, subcontractors, suppliers, accreditation bodies, regulatory agencies and laboratory neighbors. Many laboratories focus their complaint policies and procedures on data or reporting errors made by the laboratory. Although this type of complaint is very important, do

not overlook failure to achieve turnaround time commitments, understandability of reports and clear communications as important quality issues that may warrant a complaint investigation.

The laboratory may have separate and unique procedures for handling complaints, or complaints can be handled in a manner similar to and feeding into the laboratory's nonconforming work and corrective action procedures.

Chapter 3 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 156.

1. Which of the following are documents that must be controlled (select all that apply)?

- ☐ a. Laboratory Quality Manual
- b. ISO/IEC 17025
- c. Instrument Maintenance Log Book
- d. Blank Analytical Worksheet
- e. Microscope Instrument Manual
- f. Analyst Training Documentation
- g. Excel spreadsheet template for preparing QC charts
- h. Reference book used to identify fungal organisms
- i. Worksheet containing Fiber Count Data
- j. Analytical procedures

2. What is the purpose of the master list of controlled documents?

3. How do laboratories most commonly document their contracts with customers?

4. What records related to review of requests, tenders and contracts must be maintained?

5. True or False: Laboratories do not need to notify customers when they subcontract analytical work to another laboratory.

- ☐ True
- ☐ False

6. List at least four (4) commonly used criteria for evaluating suppliers of quality-related materials and services.

1.
2.
3.
4.

7. Which of the following do not need to be on a laboratory's approved supplier list?

a. Manufacturer of the gas chromatograph
b. Provider of analytical reagents
c. Company providing service for analytical balances
d. Provider of microscope slides, cover slips, etc.
e. Provider of printer paper

8. True or False: Laboratories are required to seek feedback from customers.

True
False

9. List at least three (3) parties, other than customers, whose complaints must be addressed by a laboratory's complaint policies and procedures.

1.
2.
3.

Chapter 4

Nonconforming Testing Work (4.9) through Preventive Action (4.12)

Learning Objectives:

- After completing this chapter, the student should be able to:
- Define and recognize nonconforming testing work
- Identify responsibilities, authorities and requirements related to nonconformities and corrective action
- Understand when and how the corrective action process must be initiated
- Summarize the basic components of the corrective action process
- Understand the difference between corrective and preventive actions
- Recognize the processes that contribute to continuous quality management improvement

Introduction

Sections 4.1 through 4.15 of ISO/IEC 17025:2005 (referenced within this document as "the standard") specify quality management system requirements. AIHA-LAP, LLC Modules include additional accreditation requirements. The student should read Sections 4.9 through 4.12 of ISO/IEC 17025 and Module 2A, Sections 2A.4.9 through 2A.4.12, of AIHA-LAP, LLC policies in conjunction with reviewing the sections presented in this chapter.

4.9 Control of Nonconforming Testing Work

This section of the standard describes requirements for the control of nonconforming testing work. Nonconforming testing work is defined within the context of ISO/IEC 17025 Section 4.9.1 as "…when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer." This requirement is often misinterpreted as only applying to the result of the work (i.e., errors in the final reported results). Note that the standard is much broader and includes "…when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer." So this requirement also extends to nonconformities with respect to the laboratory's procedures and the agreed requirements of the customer. These nonconformities may or may not adversely affect the quality of the reported data.

The policy must be included in the quality manual and the policy and laboratory procedures must "ensure" the following. Although the procedures must only ensure it is recommended that the laboratory procedures address each of the following issues:

a. the responsibilities and authorities for the management of nonconforming work are designated and actions (including the halting of work and the withholding of test reports and calibration certificates, as necessary) are defined and taken when nonconforming work is identified;
b. an evaluation of the significance of the nonconforming work is made;
c. correction is taken immediately, together with any decision about the acceptability of the nonconforming work;
d. where necessary, the customer is notified and work is recalled;
e. the responsibility for authorizing the resumption of work is defined.

The ISO/IEC 17025 Notes in this section of the standard include examples of nonconforming work and various means through which nonconforming work can be identified. Two of the most common processes for identifying nonconforming work in AIHA-LAP, LLC laboratories are the data review that AIHA-LAP, LLC requires prior to the reporting of customer test results and the annual internal quality system audit.

Responsibilities and authorities for the management of nonconforming work are typically designated within the nonconforming work procedure, but often some aspects of dealing with nonconformities may be included in analytical method SOPs. Issues related to calibration and continuing calibration verification failures and batch quality control issues are examples.

The evaluation of significance is often the responsibility of the technical manager or quality manager, but supervisors and analysts may also have these responsibilities for work under their control. ISO/IEC 17025:2005 was revised from the 1999 version to require that "correction" as opposed to "corrective action" is taken immediately together with any decision about the acceptability of the nonconforming work. Correction is something that is done immediately and could include instrument calibration or maintenance or a recalibration that has not, or will not, affect results that have been reported.

Section 4.9.2 requires that formal corrective action procedures (as defined in Section 4.11 of the standard) are promptly followed in cases "Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures."

AIHA-LAP, LLC Policy 2A.4.9.1 additionally requires that "The laboratory shall document and keep records of all nonconforming events." This applies to all nonconforming events, not only those requiring formal corrective action. The detail of these records varies with the nonconformity and significance of the nonconformity. A nonconforming instrument calibration conducted prior to sample analyses is

a nonconformity that may only require instrument maintenance, re-analysis and/or re-preparation of calibration solutions to obtain a satisfactory instrument calibration. Depending upon the laboratory's procedures, documentation of such activities within instrument maintenance records, or on the instrument output, or within a laboratory notebook can suffice as a record of the nonconformity and the associated correction. On the other hand, if this same unacceptable calibration was not identified by the analyst and data were deemed acceptable for reporting, the laboratory has not followed its own policies and procedures and corrective action (as described in Section 4.11) must be followed.

AIHA-LAP, LLC Policy 2A.4.9.2 additionally requires that "Any outlier from a PT, Round Robin, or Demonstration of Competency shall be addressed as a nonconforming event" requiring the laboratory to document and maintain records of the investigation of any outlier.

4.10 Improvement

This section of the standard is one of the few sections that do not require any additional specific policies or procedures. Rather, it is a performance-based requirement that references other sections of the standard. It states, "The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review."

The laboratory's implementation of these referenced activities and the laboratory's documented evidence of identifying and implementing suggestions for improvement form sufficient evidence of conformity with this section of the standard. It is important that laboratories not only maintain, but take efforts to improve the effectiveness of the management system.

4.11 Corrective Action

Corrective action is a very important element of a properly functioning management system. Things do and will go wrong. It is important that laboratories evaluate the cause of the nonconformities and implement effective corrective actions in an effort to prevent the recurrence of the problem. The laboratory policy regarding corrective action must be included in the quality manual.

ISO/IEC 17025 Section 4.11.1 states, "The laboratory shall establish a policy and a procedure and shall designate appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the management system or technical operations have been identified." The ISO/IEC 17025 Notes for this section state: "A problem with the management system or with the technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, feedback from customers and from staff observations." If you think these requirements appear very similar to the Section 4.9 requirements, you are correct. They are similar but not the same. The difference is that corrective action is required when the nonconforming work evaluation indicates that

the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures.

Section 4.11.2 of the standard requires that the procedure for corrective action start with an investigation to determine the root cause(s) of the problem. The Note states: "Cause analysis is the key and sometimes the most difficult part in the corrective action procedure. Often the root cause(s) is not obvious and thus a careful analysis of all potential causes of the problem is required. Potential causes could include customer requirements, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration."

True root cause(s) analysis can be a science in and of itself. The intent of the standard is not to require a complete and exhaustive root cause investigation for each nonconformity, but to delve deeper than a common superficial cause of "analyst error." Analysts will make occasional errors, but frequent errors of the same type are often indicative of underlying root cause(s) that could include inadequate training or instruction, excessive workload, excessive responsibilities, distractions and interruptions, etc. Attachment 4-1 contains an elementary root cause analysis procedure and indicates some elements that can be considered as part of the root cause(s) investigation. Another often effective technique is the "Five Whys?" When using this technique, the investigator asks "why" something occurred rather than "what" occurred. When the first "why" question is answered, the investigator asks why that occurred. This iteration continues until there are no further answers to the "why" question. Often five iterations will get you to the root cause(s) or closer to the root cause(s). If several potential root causes are identified, each can be pursued with this technique.

This section of the standard also requires that the following actions are taken by the laboratory:

- The laboratory shall identify potential corrective actions.
- It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.
- Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.
- The laboratory shall document and implement any required changes resulting from corrective action investigations.
- The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

Section 4.11.5 of the standard requires: "Where the identification of nonconformities or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this International Standard, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with 4.14 as soon as possible."

The extent of this audit is up to the laboratory and is typically part of the investigation regarding the cause(s) of the nonconformity or departure. Follow-up on the effectiveness of corrective actions taken is also a requirement and may involve a follow-up audit of the activity or some other form of specifically monitoring the effectiveness. In some instances, follow-up may not be required or feasible due to the corrective action taken.

Records of corrective actions must be maintained. There is no specific format for a corrective action plan and corrective action records, however, corrective action records should include the following:

- A statement of the nonconformity identified. Records should include a statement of how and when the nonconformity was detected.
- Investigation regarding the root cause(s) of the problem.
- The corrective action plan should include proposed corrective actions that will be used to correct the nonconformity.
- Laboratory personnel should be assigned responsibility for implementing each of the remedial action steps.
- The corrective action plan should include a timetable for the implementation of the corrective actions. The deadlines should be reasonable (not too long or too short).
- The corrective action plan should include a follow-up method to ensure that the corrective actions have been completed and that the nonconformity has been corrected. The plan may specify a deadline to perform a follow-up audit to make these determinations. If so, laboratory personnel should be assigned the responsibility for the follow-up audit.
- The corrective action plan should be reviewed and approved by a responsible party such as the technical manager or the quality manager.

An example corrective action record template is included as Attachment 4-2. The form highlights required elements and helps to guide the laboratory through the corrective action process. A sequentially numbered record is suggested for ease of tracking and monitoring. Many laboratories utilize computerized systems for documentation and tracking of corrective actions. Whatever method is selected, corrective action records should be organized and readily available for review. AIHA-LAP, LLC requires that corrective actions be summarized in quarterly quality assurance reports to management. Corrective actions must also be taken into account as part of the annual management review.

Lastly, AIHA-LAP, LLC section 2A.4.11.1 states “Any PT round that leads to the NP status of a laboratory shall be addressed by the corrective action process.” Since non-proficient performance in proficiency testing is significant, a full corrective action investigation is expected.

4.12 Preventive action

Preventive action is the process for identifying needed improvements, and identifying and correcting potential nonconformities before they occur. If a nonconformity has occurred, it is then subject to

corrective action rather than preventive action. Accordingly, preventive action is a pro-active process that occurs prior to a problem, where corrective action is reactive and occurs after a problem has been identified.

Many laboratories do a very good job of identifying and conducting preventive actions, but a poor job of documenting preventive action plans. The standard requires that, when identified, the laboratory develops, implements and monitors action plans to reduce the likelihood of the occurrence of the potential nonconformity and to take advantage of the opportunities for improvement. Section 4.12.2 states: "Procedures for preventive actions shall include the initiation of such actions and the application of controls to ensure that they are effective."

The Notes in this section of the standard provide good guidance. In addition, many labs conduct periodic quality control or laboratory meetings where preventive actions may be discussed. It is suggested that laboratories review their ongoing efforts in this regard and, where applicable, define such activities as part of their preventive action procedure. Internal audits and outside assessments are also opportunities for improvement and identification of preventive actions. For example, if a nonconformity is observed in one laboratory area and the root cause is determined to be an SOP with insufficient detail, revision of SOPs in other laboratory sections (where a nonconformity has not yet been identified) is a preventive action to deter a similar potential nonconformity. Also consider "close calls", "near misses", review of trends in quality control data, and other suggestions for improvement as opportunities to identify and implement preventive actions.

Records of preventive action must be maintained by the laboratory. The similarity of the processes associated with corrective and preventive action permits the use of a combined corrective and preventive action record to document the preventive action plan. Such an example is included in Attachment 4-2.

AIHA-LAP, LLC requires that preventive actions are addressed in quarterly quality assurance reports to management. Preventive actions must also be taken into account as part of the annual management review. As with corrective actions, organized and readily available preventive action records are needed to satisfy these reports and reviews.

Chapter 4 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 157.

1. True or False: All nonconformities affect the quality of data reported by the laboratory.

- ☐ True
- ☐ False

2. Which of the following would not be considered a nonconformity?

a. Unacceptable result on a PT sample.
b. Incorrect results on a final report.
c. One QC result exceeding the warning limits.
d. An analysis that did not fully comply with the laboratory's analytical procedure.
e. A purchased chemical solution that does not match the manufacturer's certificate of analysis.

3. What two (2) laboratory processes are commonly used to identify nonconformities?

1.
2.

4. Identify four (4) elements that must be ensured by the laboratory's policies and procedures for nonconforming work.

1.
2.
3.
4.

5. True or False: All nonconformities and corrective actions taken must be documented.

True
False

6. What is the first step of the corrective action process?

7. Name five (5) additional activities that are part of the corrective action process.

1.
2.
3.
4.
5.

8. Explain the major difference between corrective and preventive actions.

9. True or False: Preventive actions do not need to be documented.

True
False

10. What parts of its management system can a laboratory use to continually improve its effectiveness (name at least three (3))?

1.
2.
3.

Chapter 5

Control of Records (4.13) through Management Review (4.15)

Learning Objectives:

After completing this chapter, the student should be able to:

- Identify all types of quality records that must be controlled
- Understand the requirements for record retention
- Summarize several methods for protecting and storing quality and technical records
- Identify the basic items that an internal audit must address
- Summarize the documentation requirements for an internal audit
- Understand the purpose of the management review and how it differs from the internal audit
- Identify elements that must be considered during the management review

Introduction

Sections 4.1 through 4.15 of ISO/IEC 17025:2005 (referenced within this document as "the standard") specify management system requirements. AIHA-LAP, LLC Modules include additional accreditation requirements. The student should read Sections 4.13 through 4.15 of ISO/IEC 17025 and Sections 2A.4.13 through 2A.4.15 of AIHA-LAP, LLC Module 2A in conjunction with reviewing the sections presented in this chapter.

4.13 Control of Records

As stated in Chapter 1, records provide historical evidence of what has been conducted or completed by the laboratory. Do not confuse records with documents that are instructional. The control of documents is addressed in Section 4.3 of the standard and the course materials.

Section 4.13.1.1 of the standard has one of the most interesting requirements as it includes eight (8) verbs related to control of records. This section states: "The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions." Both quality and technical records are addressed by this section of the standard. Quality records include, but are not limited to, internal audit checklists and reports, management reviews, corrective and preventive actions, complaints, archived procedures, personnel education and experience. Technical records include, but are not limited to, training records, bench sheets, instrument logs, raw data, QC summaries,

data review records, validation records, MDL studies, PT results, reporting limit verifications and customer test reports. Regardless of whether records are categorized as quality or technical records, they must be controlled.

This section of the standard appears to demand a significant level of detail to satisfy these procedural requirements. The litmus test for the control of records procedure is for someone not intimately involved with the laboratory operations to be able to identify various laboratory records and understand how they are collected, indexed, accessed, filed, stored, maintained and disposed of by the laboratory. With the laboratory procedure in hand, an individual with minimal knowledge of the laboratory could locate current records. Since this can be a rather formidable task, records management matrices, such as that included as an Excel spreadsheet file (Attachment 5-1), can assist in simplifying this procedure. The example records management matrix takes the form of a spreadsheet, but a database or other methods of organizing this information can be used. It can also take the form of a narrative procedure, but many find the records management matrix approach both simpler and more descriptive. The example provided includes completed sections for several records and a listing of records and associated reference to ISO/IEC 17025 and/or AIHA LAP, LLC policy requirements. This is not an all-inclusive inventory of required records, but provides a good starting point to develop your laboratory's record management matrix.

Section 4.13.1.2 requires that all records be legible and are stored and retained in such a way that they are readily retrievable and are stored in a suitable environment to prevent damage or deterioration and to prevent loss. When dealing with hard copy (paper) records, this requires an area that is secure and not subject to damage from water and vermin (rats, mice). Individual pages of records (such as bench sheets or preparation records), even when filed in a standard three-ring binder are easily removed and misplaced, increasing the risk for lost data. Bound laboratory notebooks are commercially available and can help reduce the risk of losing information and data. Standard bound laboratory notebooks contain numbered, lined pages. Documentation of analytical procedures in this type of notebook should be chronological. Each page should contain information and data from only one analytical procedure. When documentation of an analytical procedure is complete, the rest of that page should be lined out and the analyst's signature placed at the bottom of the page. Pages should never be removed from the bound notebook. Laboratories also have the option of creating and binding their own customized notebooks.

The standard requires that retention times of records be established. Laboratory analytical and quality records may be required to be produced at some future date for a variety of reasons. The contents of one or more records may be used in a legal proceeding such as a worker's compensation suit, or the contents of many records may be used for an epidemiological study. Additionally, some OSHA and EPA regulations require record retention for a specified period of time. Record retention should be based on a review of accreditation requirements and federal, state and local regulations as well as any specific record retention requirements of customers. AIHA-LAP, LLC requires that all records be maintained for a minimum of three (3) years. The Environmental Lead Laboratory Accreditation Program (ELLAP) requires

a 5 year retention of records related to this program. In addition, AIHA-LAP, LLC requires that records needed to support current laboratory activities must be kept as long as necessary beyond 3 years. These records may include, but are not limited to:

- Training/authorization records
- Method validation records
- Equipment maintenance records
- Equipment/reference standard calibration records
- Reference material certificates of analysis

Laboratories that only use hand-written records in notebooks, logbooks, etc. will need to physically store the information and data. This implies the use of multiple storage cabinets, a storage facility or the use of a commercial archival service. The laboratory must develop and implement a system to track and account for all archived records. Such a system may include assigning unique identification numbers to each notebook or logbook, maintaining a list of all archived records, maintaining a list of storage locations, and implementing a system for retrieval and return of archived records. For larger laboratories, physical storage of laboratory records may require an inordinate amount of storage space making alternate storage systems more cost effective.

A number of laboratories are now taking advantage of advances in electronic storage technologies to simplify record keeping. One advantage to storing laboratory information and data using computer systems is the ease of retrieval. Some software packages include a system to "browse" through the stored data and search for key words to facilitate retrieval of a specific document. The desired document can then be printed, saved to a disk, or even sent by e-mail to a recipient. Many labs are attempting to minimize paper records and are scanning paper documents and then storing images electronically. This method is acceptable to AIHA-LAP, LLC as long as scanned images are legible, retrievable and suitably backed up.

Record control procedures must also address the protection and back-up of records generated electronically. These requirements are typically satisfied through routine back-up of instrument data files and LIMS, and password protected access to files, directories or laboratory network functions. AIHA-LAP, LLC considers computer records as satisfactory without hard copy files, provided copies can be produced as needed and data edits are documented within the computer files. If the laboratory chooses this option, the laboratory must beware of computer software and hardware changes that may render older data irretrievable. The laboratory may need to maintain copies of the software (and possibly hardware) used for initial sample data acquisition and reduction. Although not specifically required by the standard or AIHA-LAP, LLC, off-site storage of electronic back up records is suggested to protect records in the event of a catastrophic loss at the laboratory facility.

Care should be taken to ensure that electronic records cannot be changed after storage. This can be accomplished through the use of "read-only" files. Some laboratories choose to maintain the original hard copy records at an off-site location specifically because computer files may be corrupted. In this way, laboratory space is not lost to storage, rapid retrieval of data can occur through the computer files, and the original records can still be retrieved to prove that the data has not been corrupted if required.

Section 4.13.2 specifically addresses technical records. Sections 4.13.2.1 and 4.13.2.2 establish requirements for detailed records that can provide an audit trail permitting the test to be repeated under very similar conditions and to facilitate identification of factors affecting the uncertainty of the test result. This is a significant requirement for laboratories. Laboratory records should include, as applicable, the following information at a minimum:

- Date the sample preparation and/or analysis was performed
- Identification of the analyst preparing and/or performing the analysis
- Analytical method used
- Reagents used (lot numbers) and expiration date of reagents
- Eluents used and expiration dates
- Instrument conditions (injection temperature, detector temperature, etc.)
- Carrier gas flow rates, sample uptake rates
- Quantity of eluent or digestion reagents used
- Final volumes of digestate or desorbate
- Calibration standards source, traceability and preparation
- Calculations for desorption efficiencies
- Calculations and acceptance criteria for calibration curves including initial and continuing calibration verification
- Sources of and calculations for quality control samples
- Raw data
- Calculations for analytical data
- Comments concerning any changes to standard method
- Comments concerning any problems encountered during the analysis
- Records of data review

Nonconformities are often cited for this clause since laboratories may fail to adequately record information including:

- identification of preparation analyst;
- sample preparation logs (to identify preparation batches and associated batch QC);
- identification of specific lots of reagents used for sample preparation and analysis, specific standards used for QC sample preparation and instrument calibration, and calibration verification; and
- identification of mechanical pipettes and dispensers used for critical volumes.

ISO/IEC 17025 Section 4.13.2.3 states: "When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialled by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data."

AIHA-LAP, LLC Sections 2A.4.13.2 and 2A.4.13.3 additionally require that the date of the change is indicated, prohibit the use of correction fluid on original records, and require that all entries to hardcopy records are made with indelible ink.

The responsibility for maintaining laboratory records should be assigned. In large organizations this activity may be the duty of a records custodian or a records management department. In smaller organizations it may be the responsibility of the quality or technical manager or other designated individual. In all cases a periodic review of record files should be conducted, either as part of the annual quality system audit or as a separate activity.

4.14 Internal Audits

This section of the standard requires that laboratories periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality management system and this International Standard. AIHA-LAP, LLC requires that these audits be conducted annually. Although it is advantageous to conduct the entire audit at one time, audit activities can be broken down into quarterly or monthly segments, provided all elements are audited within each 12 month interval.

The requirements "to verify that its operations continue to comply with the requirements of the management system and this International Standard" mean that the laboratory must verify compliance with the laboratory's management system (all policies, procedures and arrangements) as well as ISO/IEC 17025. Additionally, AIHA-LAP, LLC Section 2A.4.14.2 requires that the internal audits also evaluate the laboratory's compliance with AIHA LAP, LLC requirements.

The internal audit is vitally important, since this is when a detailed audit of compliance with the laboratory's own policies and procedures is conducted. External assessments may sometimes assist in this regard, but no one understands the intricacies of the laboratory management system better than the laboratory itself. It is therefore imperative that the procedure and audit findings clearly address and document the laboratory's compliance with its own policies and procedures. It is also imperative that the laboratory clearly documents its compliance with ISO/IEC 17025 and AIHA-LAP, LLC policies. Some laboratories have developed their own checklists, but under close review, these checklists often fail to address all ISO/IEC 17025 and/or AIHA-LAP. LLC requirements. It is suggested that laboratories utilize the current AIHA-LAP, LLC site assessment checklist as a starting point for the audit checklist as this includes the complete requirements of ISO/IEC 17025 and AIHA-LAP, LLC requirements. The checklist is available from AIHA-LAP, LLC with proof of purchase of ISO/IEC 17025. The Excel version of the 2016 AIHA-LAP, LLC Site Assessment checklist is included as Appendix 5-1 for the student's convenience. If the laboratory uses this checklist to document its applicable policies and procedures that address ISO/IEC 17025 and AIHA-LAP, LLC requirements and then audits against the lab's specific policies and procedures, these internal audit requirements can be satisfied.

Note that the quality manager is not responsible for conducting internal audits, but is responsible to plan and organize audits. The quality manager is permitted to conduct the internal audit and many have this responsibility. It is also important to note that "such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited." In small laboratories, staffing and resources may not permit independent auditors, although suitably qualified independent third party auditors (consultants) may be utilized by the laboratory. In any circumstance, the auditor should have knowledge of ISO/IEC 17025 and auditing techniques. Successful completion of this distance learning course is considered as adequate training by the AIHA LAP, LLC although further training in auditing techniques is recommended.

The standard also states, "The internal audit programme shall address all elements of the management system, including the testing and/or calibration activities." This means that the laboratory must also audit its test methods. In very large laboratories, annual audits of every test method are impractical. What is considered acceptable is to audit each field of testing (technology) at least annually and to cover all test methods over a period of several years. The internal audit procedure should address the following elements. This list is not all inclusive, as the ISO/IEC 17025 and AIHA-LAP, LLC checklist details many additional elements that must also be evaluated.

- Review the laboratory quality management system documentation to ensure that all required elements of ISO/IEC 17025 and AIHA-LAP, LLC policies have been adequately addressed with laboratory policies and procedures. Document the specific laboratory quality management system policies and procedures that satisfy these requirements within the applicable sections of the internal audit checklist.
- Verify compliance with the laboratory's management system policies and procedures. Interview staff and review representative records. Document the staff interviewed and records reviewed.

- Review sample receipt, log-in and storage procedures and interview and/or observe staff to determine if the procedures are followed and are accomplishing the stated purpose of the program.
- Visually inspect the sample receipt, storage and sample preparation and analysis areas. Identify any potential contamination sources.
- Verify that required safety features and contamination control equipment such as laboratory hoods and BSCs have been tested and are working properly. Verify that required contamination control sampling (e.g., Pb wipes in metals, analytical areas or microbiological air or surface monitoring in micro labs) has been completed and recorded. Ensure that corrective actions were taken when required.
- Visually inspect the reagent/solvent storage areas. Verify that all chemical containers are labeled properly and each container has an expiration date. Verify that no chemicals are stored beyond their expiration date.
- Randomly select and review one or two projects for each accredited field of testing. Additional projects should be reviewed for fields of testing with a large number of test methods. Choose projects that have been processed and completed since the last internal audit. Trace the samples from receipt through release of the final report.
- Check the paperwork to ensure that the samples were logged in properly, the chain-of-custody was documented, the samples were stored in the appropriate area, and the storage area was appropriately maintained, as applicable. Check for any special customer requirements and verify that these special requirements were adhered to.
- Review the sample preparation and analytical procedures for the selected projects and interview and/or observe staff to determine if the procedures were and are being followed.
- Verify authorizations and demonstrations of competence for staff conducting sample preparation and analysis have been documented.
- Review equipment calibration procedures and records of analytical equipment calibrations for adequacy. Review the laboratory equipment logs to determine if scheduled and unplanned maintenance are conducted and recorded.
- Review analytical procedures and records to determine if the preparation and analysis of required batch quality control (QC) samples were done correctly and documented, calculations were accurate, data were reviewed by another qualified individual, and a final report was issued with all required information.
- Verify that support equipment (balances, pipettes, thermometers) used as part of the analysis were appropriately calibrated, that QC sample results were within acceptance limits, and that any required corrective actions were documented. Check for signatures upon receipt at the laboratory, and for signatures or initials on each page of the laboratory notebook (as applicable), on data review records, and on the final report.

- Verify that QC sample results are documented and compared with QC charts or databases. Ensure that QC charts and databases are updated as appropriate and reviewed for trends in data.

Also review records for proficiency testing samples, internal proficiency testing samples, and round robin samples analyzed to support outside laboratory accreditations. Ensure that records are complete and reflect the same treatment as customer samples submitted to the laboratory. Review the reports prepared by the proficiency testing provider. Identify any outliers and determine if corrective action regarding investigation of any outlying results was taken and documented.

The internal audit must be well documented. The use of the recommended site assessment checklist is an excellent means to ensure that required elements are audited; however, if the AIHA-LAP, LLC site assessment checklist is simply completed with check marks, this is not considered a sufficient record of the activity audited. Audit findings must document both conformity and nonconformity with requirements. The audit findings must include documentation of the specific laboratory policies and procedures audited, the personnel interviewed and/or observed, and the records reviewed to support conformity or nonconformity with requirements. A brief narrative report is also recommended to summarize the overall findings.

Too often, laboratories do not implement timely corrective actions to internal audit findings. AIHA-LAP, LLC expects laboratories to implement its corrective action procedures for all but the most insignificant internal audit and external assessment findings. If the audit results in concerns regarding the validity of data, the laboratory must notify customers in writing if investigations show that the laboratory results may have been affected.

The standard also requires follow-up activities to verify and record the implementation of corrective actions taken. This is no different than the requirement to follow-up on the effectiveness of corrective actions taken under ISO/IEC 17025 Section 4.11.4. Entering internal audit nonconformities into the laboratory corrective action process should ensure compliance with this requirement and that follow-up audits are completed when warranted.

Additional guidance on internal audits is provided in the Asia Pacific Laboratory Accreditation Cooperative—*APLAC Internal Audits for Laboratories and Inspection Bodies*. This document is available at www.aplac.org/documents/tc/tc_002.pdf.

4.15 Management Reviews

The management review is a distinctly separate and different activity from the internal audit. Many laboratories have the misconception that the management review is another audit conducted by management. This is not the case. The internal audit is an evaluation of activities versus defined requirements, those being ISO/IEC 17025, AIHA-LAP, LLC policies, and the laboratory's quality

management system. The management review is a top level review, from a business perspective, to determine the continued suitability and effectiveness of the management system and to introduce necessary changes or improvements. The management review can be likened to an annual physical examination and consultation. Your physician utilizes audit results (lab tests and a physical examination) to evaluate your conformity with "normal" levels and expected conditions. This annual physical and consultation (management review) takes a number of issues into account (e.g. test results, age, exercise, life style) and results in an evaluation of your current lifestyle (management system) and recommendations for improvement in the form of diet, exercise, or life style changes that may improve your health and longevity. Just like the physical examination, the laboratory's management review is intended to take into account a number of elements with the intent of determining the continued suitability of the laboratory policies and procedures (quality management system) and to introduce any necessary changes and improvements.

The laboratory must have a schedule and procedure for the management review. This is a prescriptive section of ISO/IEC 17025 and laboratories can take liberty in plagiarizing this section of the standard (4.15.1) regarding the elements that must be taken into account as part of the review. Also, as indicated in Chapter 2, ISO/IEC 17025 Section 4.2.2 requires the laboratory to review the overall quality objectives during the management review. This requirement is often overlooked.

AIHA-LAP, LLC requires that the management review is conducted at least annually. This does not necessarily mandate a single quality management review once per year. Many laboratories discuss most or all of the elements that must be taken into account during more frequent laboratory, quality, or management meetings. This is a suitable approach as long as all of the required elements of the management review are considered at least once per year. What is also critical is that the top level of management responsible for quality policy and resources of the laboratory participates in the management review process and concurs and supports the findings. A narrative report of the management review findings that addresses at a minimum, each of the required elements of section 4.15.1 and 4.2.2, is appropriate. The laboratory management must also set timeliness for completion of any action items that result from the management review.

Additional guidance on management review is provided in the Asia Pacific Laboratory Accreditation Cooperative — *APLAC Management Review for Laboratories and Inspection Bodies*. This document is available at **http://www.aplac.org/documents/tc/tc_003.pdf**.

Note that Section 2A.4.15.2 of the AIHA-LAP, LLC policies requires that "At least quarterly, the Quality Manager shall provide reports to laboratory management regarding quality assurance matters. These reports shall include information on internal audits, proficiency program performance, nonconformities and corrective/preventive actions taken." This is an additional and specific AIHA-LAP, LLC requirement. The purpose of this report is to keep management apprised of the status of these issues on at least a quarterly basis. This is more critical in laboratories where some of the laboratory management is not intimately involved in day-to-day activities of the laboratory. The reports can

be very brief and may vary with the size of the organization and may simply be meeting notes from periodic meetings where required elements are discussed. These reports often serve as the basis for some of the required elements that must be taken into account in the management review.

Chapter 5 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 159.

1. Which of the following are records that must be controlled (select all that apply)?

a. Blank spreadsheet used to calculate fiber count results
b. Internal audit reports
c. GC Maintenance log books
d. Analyst training file
e. SOP for formaldehyde analysis
f. Electronic instrument data file from metals analysis by ICP-AES

2. Which of the following accurately describes procedures that should be used for recording results of analytical procedures in laboratory notebooks (select all that apply)?

a. Entries should be in chronological order.
b. Entries should be made in pencil to facilitate changes.
c. The analyst should sign or initial and date each page.
☐ d. Documentation of new analytical procedures should always start on the same page as the previous analytical procedure.
e. Errors can be corrected using "white-out."
f. Errors should be corrected using a single strike-out line with initials and date.

3. List the eight (8) activities that must be described in procedures for controlling records and indicate a good method for addressing these activities.

1.
2.
3.
4.
5.
6.
7.
8.

4. How should a laboratory decide the correct length of time for storing records?

5. **List at least two (2) ways records may be stored.**

    1.
    2.

6. **The laboratory's internal audit must evaluate compliance with what sources of requirements?**

7. **What audit records are required?**

8. **True or False: A laboratory is expected to implement its corrective action procedures for all but the most insignificant internal audit and external assessment findings.**

    True
    False

9. **Under what circumstances would a follow-up audit be required after an internal or external audit?**

10. **Explain how the management review differs from the internal audit.**

11. **Which element of a laboratory's quality management system is often overlooked as part of the management review process?**

Chapter 6

General (5.1), Personnel (5.2), and Accommodation and Environment (5.3)

Learning Objectives:

- After completing this chapter, the student should be able to:
- Identify factors affecting the correctness and reliability of laboratory test results
- Understand management responsibilities related to personnel
- Define personnel training and authorization requirements
- Understand facility considerations that can affect analytical data quality

Introduction

Sections 5.1 through 5.10 of ISO/IEC 17025:2005 (referenced within this document as "the standard") specify technical requirements. The following sections are included:

- 5.1 General
- 5.2 Personnel
- 5.3 Accommodation and Environmental Conditions
- 5.4 Test Methods and Method Validation
- 5.5 Equipment
- 5.6 Measurement Traceability
- 5.7 Sampling
- 5.8 Handling of Test and Calibration Items
- 5.9 Assuring the Quality of Test and Calibration Results
- 5.10 Reporting the Results

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.1 through 5.3 of ISO/IEC 17025 and Sections 2A.5.1 through 2A.5.3 of AIHA-LAP, LLC Module 2A in conjunction with reviewing the sections presented in this chapter.

5.1 General

Like Section 4.10 of the standard, Section 5.1 is a performance requirement and does not require any specific additional laboratory policies or procedures. It is rather a statement of factors that must be taken into account when developing procedures, training staff, and selecting and appropriately calibrating equipment. This section is self-explanatory.

5.2 Personnel

Section 5.2.1 of the standard requires that laboratory management ensures the competence of all staff that operate specific equipment, perform tests, evaluate results and sign test reports; provides appropriate supervision; and uses appropriately qualified personnel. Note that opinions and interpretation are not appropriate or applicable to AIHA-LAP, LLC accredited testing labs as these labs only provide test results. Opinions and interpretation are applicable to labs such as materials failure testing labs where an opinion regarding performance and/or reason for failure is provided by the laboratory.

Note that laboratory staff must be qualified by education, experience, training, and/or demonstration of skills in laboratory procedures. A laboratory may also designate different levels of analysts such as senior chemist, chemist, technician and so forth. Whatever title individuals hold, they are responsible for complying with the laboratory's established policies and procedures including relevant quality system procedures.

Individuals responsible for sample receipt and data entry must be familiar with the requirements of the quality system and the laboratory's established procedures. They are responsible for ensuring that these procedures are followed, that sample integrity is maintained, and that data are entered as accurately as possible.

The laboratory management is required to formulate the goals with respect to the education, training and skills of the laboratory personnel. The simplest formulation of goals for personnel can be job descriptions that address the laboratory requirements with respect to the education, training and skills of the laboratory personnel. AIHA-LAP, LLC Policy Section 2A.5.2 includes a number of educational and experience requirements for the technical manager, quality manager, and analysts. The requirements in AIHA-LAP, LLC Policy Module 2A apply to all accreditation programs with the exception of the ELLAP program which includes its own requirements. Any additional program specific elements are addressed in the corresponding AIHA-LAP, LLC policy modules. These should be carefully reviewed to ensure that the laboratory has staff to satisfy AIHA-LAP, LLC requirements. It is also very important to note that AIHA-LAP, LLC requires that all results are reviewed by a qualified individual for data review prior to reporting results to customers.

This section of the standard also requires the laboratory to have a policy and procedures for identifying training needs and providing training of personnel. It is interesting to note that this is the only policy

requirement in Section 5 of the standard. Accordingly, this policy must be included in the quality manual. Procedures to identify training needs and provide training of personnel must include both current and anticipated tasks of the laboratory. The laboratory should consider the evaluation of routine QC data, proficiency testing results, trends in quality control data, and the findings of internal audits and external assessment, and management review as methods of identifying training needs. If the laboratory conducts periodic performance reviews, this is an additional method of identifying training needs.

The effectiveness of the training actions must also be evaluated by the laboratory. This can include written testing and/or documented interviews following training, but effectiveness is typically evaluated through demonstrations of proficiency by the employee following training and by monitoring ongoing quality control and proficiency testing results.

The laboratory must maintain current job descriptions for managerial, technical and key support personnel involved in testing conducted by the laboratory. Although some laboratories generate a specifically tailored job description for each individual employee, this is not required by the standard. A series of generic job descriptions can be sufficient. It is recommended within the standard that these job descriptions address required qualifications, experience, education, training and managerial duties.

Section 5.2.5 of the standard requires management to authorize specific personnel to perform particular types of sampling and tests, to issue test reports, to give opinions and interpretations, and to operate particular types of equipment. As indicated earlier in this chapter, opinions and interpretations are not applicable and the operation of particular types of equipment in AIHA-LAP, LLC accredited laboratories is typically part of the authorization to perform a test method utilizing that equipment. An authorization that is sometimes overlooked is that of data review. If data review is conducted by individuals that do not issue (sign and approve) the test report, this authorization must also be documented.

This section of the standard mandates a number of records including relevant authorization(s), competence, educational and professional qualifications, training, skills, and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed. AIHA-LAP, LLC Policy 2A.5.2.3 also requires that training records include a description of the content and duration of the training program. The content and duration of the training program can be documented with a combination of the training procedure and training records. Laboratories often develop training checklists to document these required elements and authorizations.

AIHA-LAP, LLC Policy 2A.5.2.4 additionally requires that all analysts complete a Demonstration of Competency (DOC) every six months for each accredited Field of Testing in which they participate. This can be demonstrated through the accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples. Do not overlook the use of routine

in-house quality control samples to demonstrate proficiency. The in-house quality control samples can include laboratory spiked media samples, in-house reference slides (for asbestos and spore traps) and laboratory control samples analyzed with ongoing samples.

5.3 Accommodation and Environmental Conditions

Considerations for facilities and the laboratory environment are well described in this section of the standard. The laboratory must be adequately designed, equipped and controlled to provide a safe and comfortable work environment, to prevent contamination and interferences from incompatible activities, and to control access to the laboratory.

As long as the work environment is maintained within reasonable constraints of temperature and humidity, most instrumental test methods are not significantly affected since they include batch-related quality control samples that detect problems and include calibration and calibration verification controls that either detect or compensate for changes in conditions. Where environmental conditions may have an effect, requirements must be documented and conditions must be controlled, monitored and recorded. Laboratory surfaces must also be kept clean to avoid contamination of samples and laboratory chemicals. Surface wipes of laboratory surfaces may be taken periodically to demonstrate that housekeeping procedures are adequate for controlling surface contamination in the laboratory. AIHA-LAP, LLC considers the following situations where environmental conditions and/or laboratory facilities may affect results and these requirements apply:

- Temperature of RI fluids during PLM analysis
- Contamination control for environmental microbiology laboratories (air samples and/or surface samples)
- Contamination control for environmental lead laboratories (surface wipe sampling)
- Temperature and humidity for gravimetric analyses

Testing must be stopped whenever environmental conditions may adversely affect the test results. An entirely different level of concern may arise when testing is conducted outside of permanent laboratory facilities. Mobile or field portable laboratories may require further controls and monitoring to assure that test results are not compromised by environmental conditions.

Separation of incompatible activities, and measures to prevent cross-contamination and ensure good housekeeping are also addressed in this section of the standard. Poor housekeeping procedures in a laboratory can introduce errors into the analytical process. Housekeeping is an element that is typically addressed in the laboratory Chemical Hygiene Plan (CHP) or biosafety plan. Housekeeping is especially critical in relationship to cleaning laboratory glassware. Many laboratory instruments can measure analyte concentrations in microgram (µg) or even lesser concentrations. Even minute concentrations of contaminants on glassware can result in errors in sample analysis; therefore, glassware must be extremely clean. Laboratory procedures for cleaning glassware must be developed and updated as

necessary. Many laboratories have reduced glassware cleaning requirements by utilizing disposable labware. When required, cleaning procedures will vary depending on the type glassware and the suspected contaminants. Glassware used for organic analyses may require an initial rinsing with an appropriate solvent. Glassware that may contain trace amounts of metals should be rinsed with a nitric acid solution followed by several deionized or distilled water rinses prior to reuse. Glassware used for microbiological analyses may need to be sterilized following cleaning. Other glassware should be washed with a good quality, warm detergent solution followed by at least two rinses—one with tap water and the final rinse with deionized or distilled water. The detergent should not contain any contaminants that may interfere with analysis. The glassware should be heat or air dried, not dried with a towel. Salt deposits should not be allowed to dry on glassware. If salt deposits are present, the glassware should be rinsed again with deionized or distilled water.

The extent of access control and measures taken to "ensure" that results are not adversely affected is a performance-based standard, so it is up to the laboratory to determine the extent of control and measures taken. The following are several potential situations that should be considered by laboratories:

- Separation of bulk PLM and airborne PCM/TEM analyses
- Separation of solvent extraction activities (e.g., environmental or bulk samples) from volatile organic analyses—consider separate air handling (conditioning) systems for these areas where possible
- Separate acid digestion areas from instrumental areas to reduce corrosion potential—consider separate air handling (conditioning) systems for these areas where possible

AIHA-LAP, LLC Section 2A.5.3.1 additionally require that ventilation hood face velocities are appropriate and are measured and recorded at least semiannually (annually, if alarmed).

It should be noted that AIHA-LAP, LLC Policy 2A.6 includes expectations for laboratories to follow applicable federal, state and local regulations regarding safety and health, for example, OSHA Standard 29 CFR 1910.1450, "Occupational Exposures to Hazardous Chemicals in Laboratories," and a biosafety plan for EMLAP laboratories. The laboratory must provide a written statement that the laboratory complies with all applicable standards. The AIHA-LAP, LLC assessor does not perform a safety inspection of the laboratory; however, the assessor will verify that a written chemical hygiene plan (and biosafety plan for EMLAP laboratories) exists for the laboratory operation.

Chapter 6 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 161.

1. List seven (7) factors that can affect correctness and reliability of test results and indicate the one that labs usually have the least control over.

1.
2.
3.
4.
5.
6.
7.

2. What are four (4) primary responsibilities of laboratory management related to personnel?

1.
2.
3.
4.

3. Which of the following qualifications are important to consider when hiring a laboratory analyst?

a. Education
b. Experience
c. Training
d. All of the above

4. Which of the following is a job function that requires authorization?

a. Perform analyses
b. Review data
c. Sign reports
d. Operate analytical instruments
e. All of the above
f. Only A and D

5. **Which of the following are true statements related to requirements for the technical manager in an AIHA-LAP, LLC accredited laboratory (select all that apply)?**

    a. Must possess a bachelor's degree in an appropriate physical or biological science.
    b. Must hold certification by the American Board of Industrial Hygiene (ABIH).
    c. May be a consultant or an employee of the company.
    d. Must be present on site at least 20 hours per week or 50 percent of the lab's operating hours.

6. **List the information that must be included in personnel training records.**

7. **How must competency be demonstrated for analysts to satisfy AIHA, LAP, LLC requirements, and how frequently must these demonstrations be conducted?**

8. **List four (4) actions a laboratory must take to ensure environmental conditions do not affect the quality of work being done.**

    1.
    2.
    3.
    4.

9. **List four (4) analyses that require environmental monitoring under AIHA-LAP, LLC accreditation requirements along with the type of monitoring required for each.**

    1.
    2.
    3.
    4.

Chapter 7
Elementary Statistics

Learning Objectives:

After completing this chapter, the student should be able to:

- Understand basic statistical procedures
- Understand the differences in data types and the variability present in each
- Define and understand the concepts of variability, accuracy, trueness, precision and uncertainty
- Explain the difference between populations, samples, and subsamples and the impact the difference has statistically on the interpretation of the data
- Identify different population distributions and how to check for suitability of data for analysis
- Define mean, variance and standard deviation
- Calculate the mean, variance and standard deviation for a data set
- Calculate the relative standard deviation and coefficient of variation for a data set
- Determine a confidence interval for a parameter, using a sample data set
- Explain the meaning of a confidence interval
- Perform linear regression analysis to determine the equation of the best-fitting line representing a set of data
- Calculate the correlation coefficient and coefficient of determination for a set of data

Introduction

Before delving into test methods, method development, estimation of uncertainty of measurement and statistical quality control requirements in subsequent chapters, it is important to review some statistical considerations.

The American Heritage College Dictionary defines statistics as “the mathematics of the collection, organization, and interpretation of numerical data.” Most laboratories recognize data as the lifeblood of their profession. The very nature of a laboratory is to generate data. Laboratory employees need to realize that the reliability of data is as important as the quantity of data produced. Many people view data produced through the experimental process as information. However, in the strictest sense, data do not become information until some analysis has been performed. These analyses are what make up the field of statistics.

Data

Many times scientists erroneously assume that the term "data" refers only to numerical values produced through some scientific measurement process. In reality, data can be used to describe any acquired facts. The types of data depend upon how the data were obtained and recorded. Two broad descriptors which are sometimes used to refer to different types of data are "qualitative" and "quantitative."

Qualitative Data

Words are most often used to convey information that is qualitative. Many processes can produce qualitative data, but opinion polls are typical large sources. Qualitative data from opinion polls may also include numerical descriptors, such as percentages of a sample group who expressed a certain opinion. Examples of qualitative test data would be a report that a majority of individuals polled reported that a certain chemical smelled "sweet," or the type of asbestos identified in a sample, or the genus and/or species of organisms identified in a microbiological analysis. While these data may be important for a scientific study or decision making, qualitative data do not lend easily to statistical analysis.

Quantitative Data

Quantitative data are normally described numerically. Numerical data can usually be analyzed using statistical techniques to produce reliable information. However, not all numerical data are subject to standard statistical analyses. When the numerical data are subjective in nature, statistical analyses may produce unreliable results.

Two other terms are commonly used to describe data: "soft" data and "hard" data. Typically, soft data refers to qualitative data, which cannot be easily analyzed statistically; hard data refers to quantitative data, which has been objectively obtained.

Quantitative data can be further categorized based on the process or processes used to obtain it. For the purposes of this study, only the broad category "experimental data" will be discussed, since it is the primary concern of this course and laboratory testing.

Experimental Data

Most of the data produced by analysts are experimental data. The data result from a direct measurement procedure. Several types of experimental data can be obtained depending on the measurement procedure. Several types of experimental data are discussed below.

Discrete Data

Discrete data come from a finite range of possible numbers with only specific values allowed. A good example of discrete data comes from rolling a die. Only the values one through six are available, and

each roll produces one of the six numbers. Numerous rolls of the die will produce a set of discrete data. Discrete data are not commonly encountered in chemical laboratory analyses, but they are common in fiber counting and microbiology.

Counting Data

Counting data are the results of direct counting by an analyst. Typically the procedure includes a set of rules to define what should and should not be counted. This is a particular type of discrete data. An example of this procedure would be counting viable microbial colonies growing on a specific media. The analyst examines the culture media after a specified incubation period and counts the number of colonies using a stereomicroscope. Another example is counting the number of asbestos fibers or fungal structures in a particular view field in an air sample.

Continuous Data

The measurement procedures used in analytical laboratories usually produce continuous data. Continuous data are not limited to a finite range of values. The data are a result of measurement procedures and any restrictions on values are caused by limitations in the equipment and analysts used in the measurement procedure.

All types of quantitative data can be analyzed statistically, but the statistical methods, any statistical decision criteria, and any interpretations must be appropriate for the nature of the data. Determining factors include the type of data, the amount of data, and the assumed distribution for the variable(s) of interest.

Populations, Sample Groups and Distributions

A basic concept of statistics requires an understanding of the difference between a population and a sample. A population includes all objects (or people, or a set of measurements) of interest. It could be, for example, the air in a factory or all the workers in a certain profession (possibly in the U.S. or in the world). A sample is a portion of the population that is used to represent the population. Table 7.1 contains examples of the difference between a population and samples.

Statistical methods are used to estimate values for a population using data taken from a sample. This is commonly done by determining best estimates of key characteristics in the sample data and then expanding the statistics to estimate the same characteristic in the population. Proper selection of a representative sample group is essential to ensure that the data are not biased.

Sampling often includes the need for “sub-sampling” from a chosen sample from a population. For example, an air filter provides a sample of materials in the air of a factory, and then the microscopist reads a portion (or portions) of the air filter. Sub-sampling usually follows a defined procedure to ensure

the portion taken is representative (or several subsamples are used). Statistical methods give estimates for the population from which the sample (or subsample) is drawn, based on variability of the samples observed. It is often the case that subsamples show less variability than do samples from the larger population. That is, different microscopic fields from a single filter will show lower variability than averages for air filters taken at different times and locations in a factory.

Statistical methods are needed to identify the magnitude of different types of variability so that a method's capabilities can be described accurately and they can be improved.

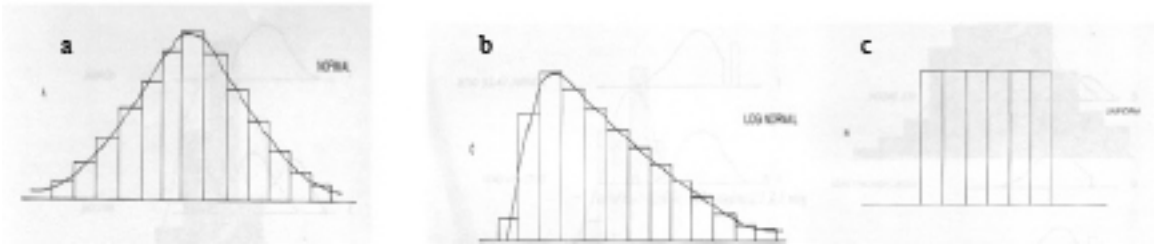
Table 7.1 - Populations and Samples

Population	Sample
All U.S. Voters	Poll of 1,000 registered voters
All cigarette smokers in the U.S.	Epidemiological group of 500 smokers
Water in city drinking system	100 mL sample used for analyses
All atmospheric air inside a city	1 cubic meter grab sample

The fiber counting technique using the National Institute for Occupational Safety and Health (NIOSH) Method 7400 is an example of using sample results to represent the entire population. Typically, a wedge of a mixed cellulose ester filter is cleared and mounted on a slide. The process of counting every field on the slide mount would be impractical and time-consuming; therefore, 20–100 fields are counted depending on the number of fibers present. The fiber counts for these fields (subsample) are used to estimate the total number of fibers on the filter as a whole (sample). This sample (the entire filter sample) is representative of the population of fibers to which the individual is exposed. Validation of the NIOSH method included statistical analyses and establishment of standards for the analytical process. The analytical process still results in a high degree of variability for the method, as is evidenced by a review of the variability in reported fiber counts for the proficiency analytical testing (PAT) program administered by the AIHA-PAT, LLC.

Population Distributions

Individual data points in a data set usually tend to cluster around some central value. The central value may or may not be the true value, but it is generally used to describe the data set as a whole (and, often the population average). The appropriate statistical analysis of the data depends on the manner in which the data points are distributed about the central value; that is, on the nature of the variability. There are several common types of distributions, and data must be treated differently statistically based on the type of distribution. Three common types of distributions encountered in analytical data sets are normal, log normal and uniform distributions. Figure 7.1 shows examples of each of these types of distributions.



a. Normal Distribution b. Log-Normal Distribution c. Uniform Distribution

Figure 7.1 – Distribution Types

The normal distribution is so called because it is "normally" expected to occur in some situations; two common examples are repeated measurements of the same characteristic in the same sample, or the distribution of a series of averages of the same quantity from a population. This distribution is also commonly called a "bell" curve, because it has that shape in histograms. Data should be examined to determine if it fits a normal distribution prior to statistical analyses. In normal distributions, approximately 68 percent (roughly 2/3) of the population is expected to fall within one standard deviation of the mean value; 95 percent of the population is expected to fall within two standard deviations of the mean value (19 out of 20); and 99.7 percent (virtually all) of the population is expected to fall within three standard deviations of the mean value.

A log-normal distribution occurs when large differences from the central value occur more frequently than small departures. It can also occur in populations where the common major source of variability is exponential, such as bacteria growth. The log-normal distribution becomes a normal distribution when the logarithms of the data are used.

A uniform distribution is one in which all the different values occur with the same frequency. Examples where a uniform distribution would be expected are rolling a six-sided die and recording the values, or recording the last digit on results of every weighing (figures that might be rounded).

Poisson Distribution

The Poisson distribution is a special distribution that may be encountered in some analytical laboratories, particularly those that perform fiber analysis using phase contrast microscopy (PCM). The Poisson distribution is a discrete distribution used to describe random phenomena in which the probability of occurrence is small but constant.

The analytical process in NIOSH Method 7400 involves counting fibers that meet specific counting criteria (counting rules). A number (20–100) of graticule areas are examined and fibers meeting the counting criteria are counted. The fiber density on the filter is calculated using the counts from the graticule areas and a final calculation is computed using the fiber density and air volume sampled.

The distribution of fibers in a graticule area follows a Poisson distribution, especially when the average number of fibers is small (1–10). The application of the Poisson component of distribution and derivation of the associated statistical treatment of data is described in detail in "The Quality of Fiber Count Data." A reprint of this article is included as Appendix 7-1. It is most important to note that the Poisson distribution becomes a normal distribution when the square roots of the data are used (as the mean gets large). This square root data transformation is used to evaluate blind recount data as mandated in NIOSH Method 7400. Some laboratories have applied this data transformation and the statistical test described in NIOSH Method 7400 to the analysis of airborne fungal spore and structure counting (spore trap) methods.

Mean, Variance and Standard Deviation

Three basic statistical parameters that are calculated for data sets are the mean, variance and standard deviation. If data from the total population are known, then the exact mean, variance and standard deviation can be calculated exactly; using the same formulas as for a sample, but using slightly different terms and "n" in the denominator for the standard deviation, rather than "n-1." Typically, however, the data are taken from a sample or subsample of the population, so estimates of the mean, variance and standard deviation are estimated from the data.

Table 7.2 – Mean, Variance and Standard Deviation

Data Set	Population	Sample
Mean	μ	$Xbar = \Sigma x_i / n$
Variance	σ^2 or V	$s^2 = [\Sigma (x_i - Xbar)^2] / (n-1)$
Standard Deviation	σ	$s = [(\Sigma (x_i - Xbar)^2) / (n-1)]^{1/2}$

The mean represents the arithmetic average value of all data points in the data set. The standard deviation represents the dispersion of the individual data point values around the mean. The variance is the square of the standard deviation.

The coefficient of variation or relative standard deviation is used to describe variability. The coefficient of variation (CV) is expressed as a proportion of the mean value and is computed using the formula CV = s/Xbar. An estimate of the coefficient of variation is sometimes symbolized as S_r (as in NIOSH Method 7400). Relative standard deviation is commonly expressed as a percentage by multiplying the CV by 100: RSD = s/Xbar * 100%.

Considerations for Suitability of Data for Analysis

It is always necessary to examine the data set for indications of problems, or to assure the suitability of data for analysis. With small datasets ($n<20$) it is difficult to rely on any firm test; instead it's common to look for deviations from expectations. For example, if a process has shown stability in the past, and 4 replicated measurements are 12.1, 12.2, 12.0 and 13.5, it is appropriate to check for reasons for a much

different value. Similarly, larger datasets (20–100) can be checked with tools such as histograms or with simple listings of data in size order, or time order.

Data sets need to be checked for various problems:

- Extreme results or outliers (also sometimes called blunders) that could be caused by calculation mistakes, use of incorrect units, or mislabeled samples. These results can heavily influence the mean and standard deviation, and must be removed before calculations, or accounted for with robust statistics (see below).
- Presence of more than one "central" point. This indicates a mixed population, and must be resolved before any analysis can continue.
- Unexpected distribution (difficult to check with small numbers of results).

If the data do not follow expected distributions or if there are influential results that cannot be discarded as outliers or blunders then you should consider using alternative statistics that are robust against departures from expected distributions. There are many options and they cannot be explained here. However, simple techniques are based on placing the data in numerical order by size. The middle value is the median—calculated as the middle value if the number of points is odd or the average of the two middle values if the number of data points is even. The median is a robust estimate of the central point and is the same as the mean in a normal distribution.

A robust estimate of the standard deviation can be obtained from the Interquartile Range (IQR). This statistic is calculated by finding the first and third quartiles (25th and 75th percentiles), which can be found easily by taking the "median" of each half of the dataset that is split by the median—that is, the middle ordered value if the half-set has an odd number of points and the average of the two middle points if the half-set has an even number. The difference between the third quartile and the first quartile is the IQR—that is, the range of values spanned by the middle 50 percent of results. A robust estimate of the standard deviation is 0.7413 times the IQR.

Outliers

It is important to consider gross outliers when evaluating quality control limits or evaluating round robin study data as they may significantly affect the statistical analysis of results. Results that fall outside of 1.5 (IQR) or 2 (IQR) on either side of the median value are considered outliers and should be removed before further statistical estimates are made. Other outlier tests that may be applied to normally distributed data to identify outliers include Grubb's test and the Dixon test. Information regarding these techniques is available at www.itl.nist.gov/div898/handbook/eda/section3/eda35h.htm and www.graphpad.com/quickcalcs/GrubbsHowTo.cfm.

Confidence Intervals

In many situations it is necessary to calculate confidence limits for results. This can be necessary, for example, to estimate the uncertainty of measurement for a result (see Chapter 9) or to determine control limits for statistical quality control charts. Control limits calculated from QC data, for example, might use a "2SD" limit to allow 95 percent confidence that when a procedure is "in control" that the results will be inside the limits. 2SD limits are typically used for warning limits. The use of "3SD" limits to allow 99.7 percent confidence is typically used for the upper and lower control limits. A Confidence interval is a range of values within which an experimental result will lie, with a stated confidence. This interval is usually calculated using the sample mean and standard deviation.

One of the most basic statistical calculations used in data analyses is to compute a confidence interval for results from a normal distribution. Calculating a confidence interval differs depending on whether the population or true standard deviation (σ) is known or an estimate of the standard deviation (s) is used. Since σ is rarely known for analytical data, the formula for calculating a confidence interval when s is used is shown as Equation 1 below. This interval tells us the range of results to expect for any randomly chosen member of the same population from which the original data were taken and using the same selection and measurement techniques.

(1) Equation 1: Confidence Interval for a single value (x):

CI(x) = xbar+/- (t * s)

Where:

xbar = estimate of the sample population mean

s = estimate of standard deviation

t = A value from a Student's t test table (available in most statistical reference books). The value depends on the probability level desired and the number of degrees of freedom upon which s is based.

The student's t table has columns across the top indicating the probability level desired. It is common practice to use a 95 percent probability level for analytical data, although some researchers prefer to use a 99 percent probability level. The far left-hand column of the table lists the degrees of freedom (df). If n and s are based on the same number of samples, df will be n-1. The degrees of freedom represent the number of independent estimates of the statistic of interest (mean, standard deviation, etc.) can be obtained from a given data set.

If there is a need to estimate a confidence interval for the mean of multiple results from the same sample, then the formula in Equation 2 is used.

(2) Equation 2: Confidence Interval for a mean of sample results (xbar):

CI(xbar) = xbar +/- [(t * s) / (n ½)]

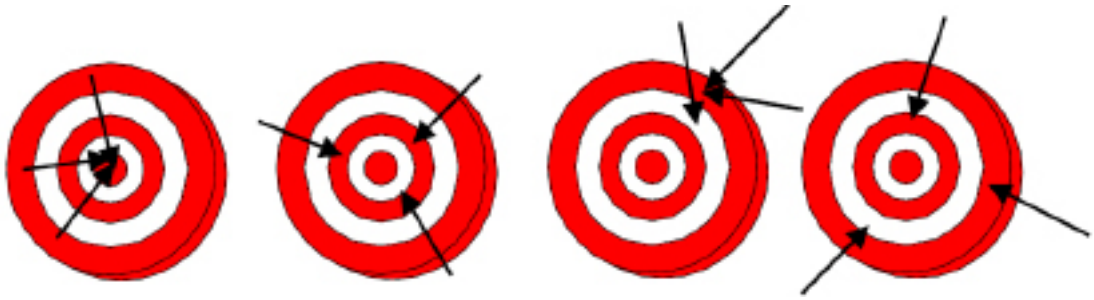
This is the same as Equation 1, except the width of the interval is divided by the square root of n, where: n = number of measurements used to calculate the mean sample result.

This gives a range of values within which a study mean is expected to lie, if the experiment were to be repeated. It is often interpreted as the range of values that contain the true population average, but that is not strictly true. Note that a confidence interval for a mean is much smaller than the confidence interval for a single result.

Accuracy: Trueness and Precision

The accuracy of a measurement result is determined by the trueness and precision of the measurement procedure. Trueness is a concept and can be defined as the ability to measure close to the true value of an analyte, and it is usually estimated with a measure of bias such as the difference between an average and a reference value. Precision can be defined as the ability to replicate a measured value, whether the value is biased or not, and it is usually estimated by a measure of imprecision, such as the standard deviation. Statistical procedures can be used to help determine if trueness and precision are acceptable for data produced by an analytical procedure. Standards for trueness and precision must be established prior to analyses and these standards can then be used to maintain statistical control of the analytical process.

By statistically analyzing data to estimate uncertainty you can determine whether the accuracy of a result (estimated through its bias and precision) is acceptable for your use. The graphics in Figure 7.2 show that you can have measurements that are inaccurate because of bias or imprecision (or both)—being accurate means being unbiased and having small imprecision.



Accurate Unbiased & Precise	Inaccurate Unbiased & Imprecise	Inaccurate Biased & Precise	Inaccurate Biased & Imprecise

Figure 7.2 – Accuracy: Bias and Precision

Variability

Even continuous data produced by precise analytical measurements inevitably contain some variability due to the nature of the analytical process. Replicate measurements of customer samples or laboratory control samples are typically performed that result in a series of values. Even when the measurements are made using the same sample material, the same instrument and the same analytical conditions, the values produced will be slightly different. There are several sources of variability in analytical processes, which result in the observed differences in data.

Some variability is to be expected to occur naturally, and is known as "common cause" variability (or uncontrollable variability); other sources of variability are due to special causes or causes introduced into the system. These sources of variability jointly contribute to measurement error, or the uncertainty of a measurement. Routine quality control procedures are used to identify special cause variability, while providing a long term description and monitor for common cause variability.

Uncertainty is inherent in every measurement procedure, and often many causes are uncontrollable, such as the numeral after the last digit on the balance display. However many of the sources of error are at least partially controllable. The major categories or sources of uncertainty are time, location, equipment, environmental conditions and operators.

The uncertainty of a measurement result is a critical component of the result because it is a statement of the reliability of the result—it gives a range of values in which the "true" value of the analyte lies. This allows a user to determine whether the result (or product) is adequate for its intended use. Many laboratory scientists assert that a measurement result is not complete without a statement of the uncertainty. Estimation of uncertainty of measurement is discussed in more detail in Chapter 9.

Uncertainty is sometimes implied by reported results when, in fact, the implied uncertainty cannot be supported. This situation can occur when analysts do not understand the concept of significant figures (sometimes called digits) when reporting analytical data. It is very easy to get caught up in reporting data to several decimal places simply because the calculator or computer displayed that number of decimal places. Accurate data reporting must comply with the rules of mathematics regarding significant figures. AIHA-LAP, LLC policies require that laboratories report the appropriate number of significant figures. This requirement is addressed further in Chapter 12.

Significant Figures

Numbers can either be exact or approximate. Seldom, if ever, are exact numbers reported in analytical computation. Measurements and computations typically result in approximate numbers. Normally the last digit in a number is the most inaccurate (or where the approximation occurs). Thus, the measurement recorded as 25 implies that the measurement occurred at the ones (5) but not at the tenths place. In the same way, the number 25.0 implies the measurement occurred at the tenths place but not the hundredths. Thus, 25 implies that the actual result of the measurement lies between 24.5

and 25.5, while 25.0 implies that the true value lies between 24.95 and 25.05. We can say that 25 has 2 significant figures or that it has 2-digit accuracy, and that 25.0 has 3 significant figures or that it has 3-digit accuracy. In summary, the number of significant figures is a measure of the accuracy of a number.

When using scientific notation, the number of significant figures is given by the total number of figures including those after the decimal point. Exponential notation should be used for numbers of large magnitude containing a small number of significant digits. The number of significant figures is indicated by the total number of digits included. Thus a population of 40,000 would be written as 4.0×10^4 if it is measured to 2 significant figures and 4.00×10^4 if measured to 3 significant figures.

Whenever computations are performed using approximate numbers, the final results are also approximate. Since modern computational devices normally produce a final result with many more significant figures than may be appropriate, care must be taken in reporting that result. If there is a measurement with 2-digit accuracy and another with 3-digit accuracy, how much digit accuracy should the final result have? For example, if we measure out a 10 milliliter (mL) aliquot of a sample (2-digit accuracy) and find 0.125 milligrams (mg) of benzene in the aliquot (3-digit accuracy), then compute a concentration of 0.125mg/10 mL or 0.0125 mg/mL, how should the final result be reported? Because the 10 mL aliquot has only 2-digit accuracy we know the values can range from 9.5 to 10.5 mL. Therefore, a range of concentrations from 0.0119 to 0.0132 mg/mL can be reported. Accordingly, a reported result of 0.0125 mg/mL would imply more precision than is actually present. The result should be reported as 0.012 mg/mL implying that the result is only accurate to 2 digits.

Rounding

Reporting data to the proper number of significant digits usually requires rounding of numbers to a specific decimal place. Rounding should be the last operation in a computation so as not to introduce rounding errors early in the process. In rounding, when the digit or digits immediately following the figure to be retained is/are greater than 5, 50, 500 and so on, the retained figure is increased by one. When the digit or digits immediately following the figure to be retained is/are less than 5, 50, 500 and so on, the retained figure is left unchanged. When the digit or digits immediately following the figure to be retained is/are exactly 5, 50, 500, and so on, the retained figure should be increased by one if the figure is odd and left unchanged if the figure is even. Thus, the following examples are rounded to the nearest tenth:

0.17 would be rounded to 0.2,
0.136 would be rounded to 0.1,
0.150 would be rounded to 0.2, and
0.25 would be rounded to 0.2.

The following rules are given to refresh the reader's memory on significant figures:

Addition and Subtraction

When several numbers are added, the sum should be rounded to the number of decimal places no greater than in the addend which has the smallest number of decimal places. For example:

```
  4.10  (2 decimal places)
0.0098  (4 decimal places)
+2.512  (3 decimal places)
------
6.6218
```

The result should be rounded to 6.62 (2 decimal places)

Multiplication

Round off the result to the same number of significant figures contained in the multiplicand or the multiplier, whichever has the smallest number of significant figures. For example:

```
    11  (2 significant figures)
  26.4  (3 significant figures)
X 4.234 (4 significant figures)
-------
1229.5536
```

The result should be rounded to 1,200 or 1.2 X 10^3 (2 significant figures)

Division

Round off the result to the same number of significant figures contained in the dividend or the divider, whichever has the smallest number of significant figures. For example:

```
   235  (3 significant figures)
+ 1.2457 (5 significant figures)
--------
188.64895
```

The result should be rounded to 189 (3 significant figures)

Powers and Roots

Round to the number of significant figures in the original data. For example:

$(26.1)^{1/2}$ (3 significant figures) = 5.108815

The result should be rounded to 5.11 (3 significant figures)

Linear Regression for Standard Curves

Analytical procedures typically include producing "standard curves" prior to analyses. Preparing working standards from a calibration stock solution using serial dilutions produces data, which can then be used to prepare standard curves. After a standard curve has been produced, analysts sometimes study a select number of calibration samples and compare the results to the standard curve as a quality control measure.

The analyst must be able to calculate the standard curve from the original QC data and determine whether the calibration samples show a good correlation to the standard curve. This module will present one statistical method to perform these tasks.

Scatter Diagrams

Plotting the data points from analyses of serial dilutions on regular graph paper can give the analyst a visual impression of the existing relationship between the variables (i.e., area under the curve and concentration). Figure 7.3 shows an example scatter diagram from the analytical data presented in Table 7.3.

Table 7.3 – Analytical Data Points

X Value	0.5	1.0	1.5	2.0	2.5	3.0	3.5
Y Value	12	21	41	49	55	87	100

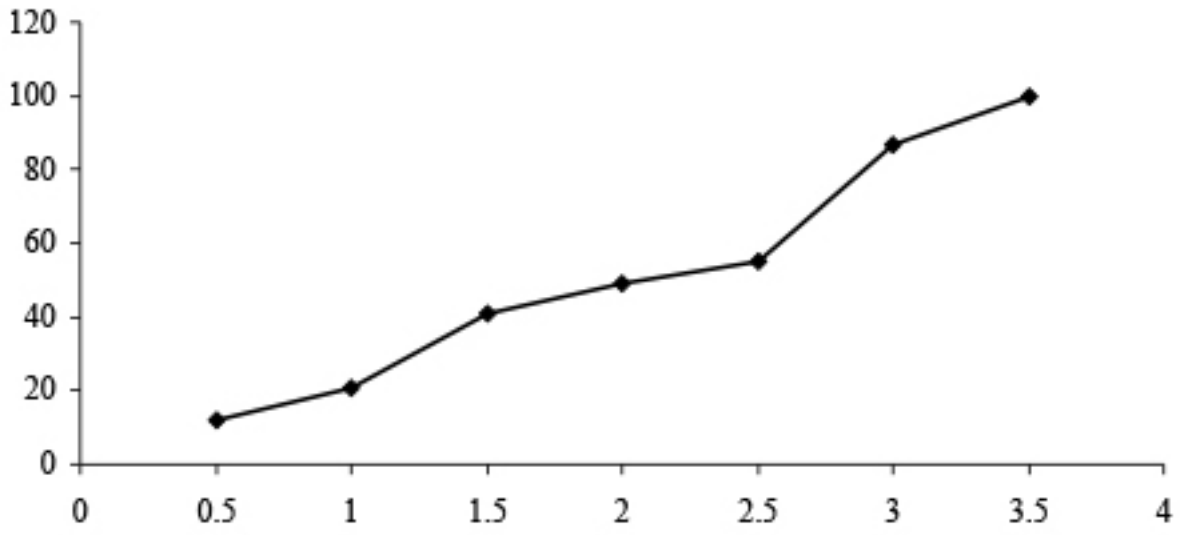


Figure 7.3 – Scatter Diagram

Best Fit of Line

There are several straight (linear) lines that could be drawn through the points in our scatter diagram of Figure 7.3 to connect two or more points; however no straight line will fit all points in the dataset. The objective is to find the single line with the "best" fit. In statistical analysis this is the "least squares" criterion— whereby the line minimizes the total squared vertical distance from points to the line. The equation of any line can be written as the following equation:

$$y = mx + b$$

where m is the slope of the line and b is the y intercept

Values for x and y have been measured, but the slope and intercept are still unknown. It is necessary to find the line that minimizes the differences between the measured data points and the predicted values on the regression line.

This line is commonly referred to as the linear regression line or least squares line of best fit. Most hand held calculators with statistical functions and spreadsheet applications include this function. Figure 7.4 graphically depicts the line of best fit using this function with a resulting equation of:

$$y=29.3x-6.4.$$

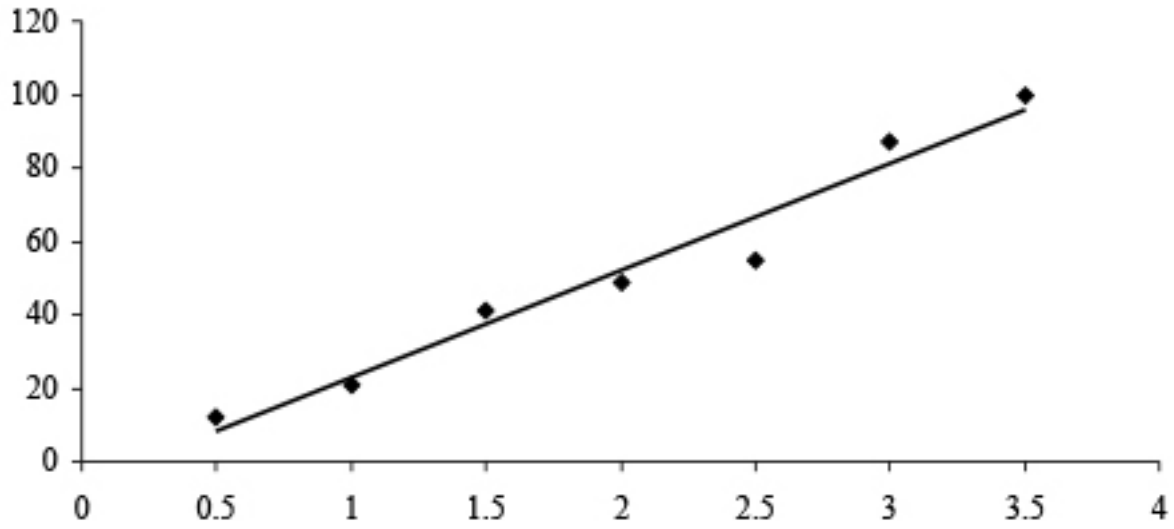


Figure 7.4 – Measured Data and Best-fit Line

Laboratories should have criteria for the fit of the line (see correlation) and for the maximum allowed difference between a point and the line, to determine whether a standard line is suitable for use. If there is a poor fit or if one of the points is off the line by a significant amount, the standard curve should not be used, and the process should be repeated or the reasons for the poor fit must be corrected.

Correlation

To estimate the strength of the linear relationship between the x and y we use the correlation coefficient, commonly noted as "r." The square of r is used to estimate the proportion of variability in y that is "explained" by variability in x. That is, the extent to which you can predict y if you know x (which is the use of standard curves). This statistic is often called the "coefficient of determination" and labeled as r^2. The coefficient of determination can be computed using most handheld calculators with statistical functions or spreadsheet applications. In our example,

$r^2 = 0.964$ and
$r = 0.981$

The calculated correlation coefficient (r) can be used to test whether the regression line provides a statistically significant (i.e., non-zero) model for variability in the y variable.

The generally accepted practice in analytical laboratories is to use an r value of 0.995 or greater as a criterion for a strong enough relationship to base predictions (that is, be useful rather than just statistically significant). This allows determination of whether the data are a good fit to the curve at a much greater level of significance (depending on the number of data points). For the above problem the data would not meet the criterion for an r value of 0.995.

Chapter 7 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 163.

Consider the following data set:

A laboratory analyst counts fungal colonies growing on malt extract agar (MEA) after the appropriate incubation period. The following data were recorded.

Taxa	Plate 1 counts	Plate 2 counts	Plate 3 counts
Cladosporium sp.	23 colonies	12 colonies	37 colonies
Aspergillus wentii	5 colonies	0 colonies	2 colonies
Penicillium sp.	11 colonies	2 colonies	13 colonies
Mycelia sterilia	18 colonies	6 colonies	14 colonies
Trichoderma sp.	4 colonies	1 colonies	0 colonies

1. The information in the above table represents what type of data?

- ☐ a. Qualitative Data
- ☐ b. Quantitative Data
- ☐ c. Neither of the Above

2. Which type of experimental data does the table present? (select all that apply)

- ☐ a. Discrete Data
- b. Counting Data
- ☐ c. Continuous Data
- d. None of the Above

3. If a second analyst counted the plates immediately after the first, how would you expect their separate data to compare?

a. The data should be the same.
b. The data should differ slightly.
c. The data should differ greatly.

4. If the second analyst's data differed from the data presented above, what would you expect to produce the variation?

5. In the following table, enter the data type (i.e., discrete, continuous, counting) next to the collection technique.

Experimental Process	Data Type
Recording Successive Rolls of a Die	
Concentration from Gas Chromatography Run	
Fiber Concentrations Using NIOSH 7400	
Lead Concentrations from Atomic Absorption Analysis	
Polling Lab Analysts to Determine Their Favorite Analyte	

6. Define accuracy, precision and bias.

Consider the following data for Question 7:

Represented below are several replicate analyses of the same sample, using the same analytical conditions (GC column, temperature, run time, etc.).

Analyte	True Value (µg)	Reported Values (µg)
Acetone	1.005	1.004,1.005, 1.006, 1.005
Benzene	1.10	1.45, 1.46, 1.47, 1.47
Chloroform	1.03	1.20, 1.74, 2.20, 3.54
Ethanol	1.11	1.09, 1.15, 1.13, 1.17

7. Match the appropriate analyte with the statement that most accurately denotes the variability within the reported values.

Statement	Analyte
a. The data are unbiased but not precise.	
b. The data are precise but biased.	
c. The data are accurate (both unbiased and precise).	
d. The data are not accurate.	

8. **Explain how ignoring the rules of rounding and significant figures can affect the data produced by a laboratory.**

9. **Determine the number of significant digits in each of the following data points.**

Reported Result	Significant Digits
1.0002	
2.3	
1	
127	
6.234	
0.0012	
100.002	
23.025	
0.002360	
4.000	

10. **Assuming the listed data points are used in a formula to compute a single result, how many significant digits should the result contain?**

Input Data	Significant Digits for Result
1.02, 2.3, 2.112	
1.0047, 1	
0.23, 1.0, 21.3	
100.02, 1.123	
0.2369, 10.2	
1.2, 2.1, 1.3	

11. **Round the numbers in the following table to the specified number of significant digits.**

Number	Significant Digits	Answer
0.1356	3	
123.14	4	
135	2	
0.1123467	1	
12.400	4	

12. Add the following numbers and express the answer to the proper number of decimal places.

Numbers	Answer
1.23, 2.45, 23.12, 16.7	
123.04, 234.112, 125.3	
0.12, 0.998, 0.9765, 1.23	
12, 23, 456, 112	
0.1, 0.22, 1.23, 2.3	

13. Multiply the following numbers and express the answer to the proper number of significant digits.

Numbers	Answer
2, 21.2, 1.23	
1.25, 23.4, 12.22	
0.11, 0.123, 0.1235	
0.11, 1.23, 10.2	
1.234, 123.5, 0.00235	

14. Identify which of the following data represent the total population, a sample group or a sub-sample.

Data Group	Total Population, Sample Group or Sub-sample
All registered voters	
All residents of the State of New York	
100 fields from a slide mount	
A 10 mL aliquot from 100 mL of eluent	
A set of air filters for a week of monitoring	
A collection of floor dust from a day's cleaning	
Factories that apply paint to steel	

15. What does the mean represent? What does the standard deviation represent?

16. Calculate the mean and standard deviation for each of the following data sets.

Data Points	Sample Mean	Standard Deviation
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6		
32, 33, 31, 32, 33, 34, 34, 36		
1.002, 1.009, 1.004, 1.001, 1.008		
20.7, 21.0, 20.3, 22.8, 21.1, 22.3		
0.23, 0.31, 0.33, 0.45, 0.52, 0.21		
34, 67, 98, 120, 111, 54, 65, 77		

17. Calculate the coefficient of variation (CV) and relative standard deviation (RSD) for each of the data sets in Problem 16.

18. If the above data represent multiple measurements of a group of analytes, based on the computed means and standard deviations, can you estimate which of the results are the most accurate? If not, what additional information would you need?

19. Determine the degrees of freedom associated with each of the data sets in problem 16.

20. Calculate a 95 percent confidence interval for individual observations from sample populations that produced the first 3 datasets in Problem 16. Use the following t values for the listed degrees of freedom:

Degrees of Freedom	95% t value
3	3.182
4	2.776
5	2.571
6	2.447
7	2.365
8	2.306
9	2.262

21. Calculate the confidence intervals for the sample means of these three datasets.

22. Which of the confidence intervals is the narrowest? Which is the broadest?

23. What techniques could be used to narrow a confidence interval that we believe is too broad?

Consider the following data for questions 24 and 25:

Concentration (µg)	Area of Curve
1.0	10
2.0	23
3.0	29
4.0	47
5.0	52
6.0	59
7.0	87

24. Calculate the equation representing the best-fitting line through the data points.

25. Calculate the correlation coefficient and coefficient of determination for the data. Would you use these results as a standard curve?

Additional Suggested Reading

Burkhart, J.A., L.M. Eggenberger, J.H. Nelson, and P.R. Nicholson: "A Practical Statistical Quality Control Scheme for the Industrial Hygiene Chemistry Laboratory." American Industrial Hygiene Association Journal 45(6):386-392, 1984.

Natrella, M.G.: "Experimental Statistics." NBS Handbook 91, Gaithersburg, MD.: National Institute of Standards and Technology, 1991.

Taylor, J.K.: "Quality Assurance of Chemical Measurements." Chelsea, Mich.: Lewis Publishers, Inc., 1987.

Taylor, J.K.: "Statistical Techniques for Data Analysis." Chelsea, Mich.: Lewis Publishers, Inc., 1990.

Chapter 8

Test Methods and Method Validation (5.4)

Learning Objectives

- After completing this chapter, the student should be able to:
- Understand requirements related to analytical methods
- Identify sources for published analytical methods
- Summarize the contents of NIOSH Analytical Methods
- Summarize the contents of OSHA Analytical Methods
- Identify the different phases of method validation
- Understand the requirements related to validation and verification of method performance

Introduction

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.4 of ISO/IEC 17025 and Section 2A.5.4 of AIHA-LAP, LLC Module 2A in conjunction with reviewing the sections presented in this chapter.

5.4 Test Methods and Method Validation

This section includes performance standards requiring appropriate procedures for sampling, handling, transport, storage and preparation of samples and for testing methods. The laboratory must have instructions on the use and operation of all relevant equipment and on the handling and preparation of samples for testing. All relevant instructions, standards, manuals and reference data must be kept up to date and be readily available to personnel. Deviation from these methods by the laboratory shall only occur if the deviation has been documented, technically justified, authorized and accepted by the customer. The Note in this section of the standard permits labs to use recognized methods as written, but only in rare instances do laboratories conduct methods exactly as written. Almost all laboratories have minor modifications, select various options and include additional AIHA-LAP, LLC requirements that mandate additional documentation above and beyond the published method. These additions/modifications can take the simplest form of a method modification that details the changes to the published method with laboratory approval along with a copy of the published method. Most laboratories choose to adopt a standard laboratory format for their methods and generate a laboratory specific analytical method SOP.

Section 5.4.4 Notes are also a good starting point for a laboratory analytical method SOP template whether utilizing a standard or laboratory developed method. Whatever source or format the laboratory decides to use, it is suggested that the following minimum elements be included or addressed within the laboratory analytical SOPs:

- Date, approval, and unique identification as a controlled document
- Type of material tested and analytes covered
- Adaptation to lab use (specific to laboratory instruments and equipment)
- Instrument operating conditions and performance checks
- Calibration standard preparation
- Calibration frequency/levels
- QC sample frequency/preparation/acceptance criteria/corrective actions
- Sample media/storage/handling/preparation/analysis
- Data reduction/calculations
- Data reporting (significant figures/reporting limits)
- Estimates of bias and precision stated

Section 5.4.2 of the standard stresses once again that the needs of the customer must be met by the test methods employed by the lab and there is a preference for methods published in international, regional or national standards. In all cases, the customer must be informed regarding the method chosen by the laboratory and if the customer specifies a method that is considered to be inappropriate or out of date, the lab must inform the customer. This review should be initiated during the review of requests, tenders and contracts (see Chapter 3) or at time of sample receipt.

It is important to note that AIHA-LAP, LLC does not specify methods for any FoTs with the exception of asbestos by PCM where the OSHA standard mandates either NIOSH Method 7400 or the OSHA reference method. All other methods are subject to agreement with the customer. Most frequently, laboratories utilize published NIOSH, OSHA, ASTM, International, ISO and/or EPA methods with only minor modifications. These are considered standard methods and full method validation is not required; however, verification of the appropriateness of modifications adopted must be documented. Since many laboratories are multi-functional and may use EPA methods for water and or solid waste analyses, many elect to utilize these analytical methods for industrial hygiene analysis. These analytical methods may be acceptable if appropriately modified by the laboratory to incorporate preparation/digestion of the sample matrix and appropriate reporting of air or surface concentrations.

Method Confirmation

Section 5.4.2 of the standard states that the laboratory shall confirm that it can properly operate standard methods before introducing the tests or calibrations. If the standard method changes, the

confirmation shall be repeated. Confirmation for such methods can be demonstrated by establishing and evaluating the reporting limit, media blanks, calibration range, recovery and precision through the analysis of laboratory control samples or certified reference materials, desorption efficiency determinations (as applicable), and through interlaboratory comparisons or proficiency testing. The manner in which this confirmation is accomplished should be defined in the laboratory quality system documentation. Results of method confirmations must be recorded.

Laboratory-Developed Methods

Sections 5.4.3 and 5.4.4 of the standard discuss laboratory-developed methods and non-standard methods. It is rare that laboratories develop their own methods unless they are a captive in-house laboratory, have a contractual agreement to develop methods for a customer, or are an environmental microbiology laboratory. In most circumstances, these sections of the standard are not applicable. When these sections do apply, Section 5.4.4 and the notes provide good guidance regarding the content and considerations for laboratory-developed and non-standard methods. Section 2A.5.4.1 of AIHA-LAP, LLC policies additionally states that laboratory-developed methods and non-standard methods may be used if the laboratory 1) has developed and documented procedures considering the topics a-k contained in the note in ISO/IEC 17025:2005, Section 5.4.4; and 2) has validated the method, considering the following topics as appropriate: minimum acceptance criteria, analyte specificity, linearity, range, accuracy, precision, detection limit, quantification limit, stability of samples and reagents, interlaboratory precision, and analysis robustness.

Method Validation

Section 5.4.2 of the standard states that laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. Section 5.4.5 of the standard further addresses method validation requirements. Be certain to address these relevant elements when validating laboratory–developed methods.

Note that AIHA-LAP, LLC Policy 2A.5.4.2 requires that the laboratory define the process utilized in the adoption and revision of analytical procedures employed by the laboratory including when and how these procedures are reviewed. Laboratories may address this requirement as part of their document control procedure or separately. The frequency of review is up to the laboratory. Many laboratories utilize an annual review, but this may not be practical for laboratories with a large number of analytical procedures.

The following sections discuss several sources of standard methods that are often used by industrial hygiene testing laboratories. Three organizations publish the most commonly used analytical methods for environmental and industrial hygiene sampling and analysis. The National Institute for Occupational Safety and Health (NIOSH) publishes the *NIOSH Manual of Analytical Methods (NMAM)*, the Occupational Safety and Health Administration (OSHA) publishes the *OSHA Analytical Methods Manual*; and the Environmental Protection Agency (EPA) has published the *EPA Compendium of Methods for*

the Determination of Toxic Organic Compounds in Ambient Air (TO methods) and the *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air* (IO methods). Links to these compendia are available at **www.epa.gov/ttn/amtic/methods.html**. EPA also publishes standard methods for testing other environmental samples such as the SW846 methods used for sampling and analyzing solid wastes that are available on-line at **https://www.epa.gov/hw-sw846**.

The ASTM International publishes numerous consensus testing methods (**www.astm.org**). The International Organization for Standardization (ISO) also publishes international consensus standards (**www.iso.org**). ASTM and ISO testing methods are copyrighted documents, while the NIOSH, OSHA and EPA methods are available for unrestricted use.

NIOSH Manual of Analytical Methods (NMAM)

The Purpose, Scope and Use of the NIOSH Manual of Analytical Methods (Fifth Edition) states in part: "The NIOSH Manual of Analytical Methods (NMAM) is a compilation of analytical methods for air, biological, surface (including dermal) and bulk samples that have been evaluated and validated in consideration of their fitness for purpose for workplace exposure monitoring [NIOSH 1995]. NIOSH sampling and analytical methods are intended to promote accuracy, sensitivity, and specificity in industrial hygiene analyses and related applications. NMAM, which is published online (available at: **www.cdc.gov/niosh/nmam**), is constantly updated as new methods are developed and validated and as revised methods are evaluated and their performance verified. The methods published in NMAM are relied upon by authoritative bodies such as accrediting organizations and regulatory agencies. Besides sampling and analytical methods, NMAM also includes chapters on quality assurance, portable instrumentation, measurement of fibers, aerosol sampler design, and other guidance on specific areas of interest."

These methods and chapters from the fifth addition are available online at **www.cdc.gov/niosh/nmam**. This page includes links to an alphabetical listing of all methods. The air and biological analytical methods contained in NMAM were evaluated by NIOSH and classified into three evaluation categories: full, partial and unrated. Fully evaluated methods have been tested and have met all the factors of the NIOSH evaluation protocol. Partially evaluated methods have been subjected to some of the evaluation experiments but have not received a full evaluation. Partially evaluated methods may also include methods that were fully tested but did not meet one or two of the evaluation criteria. Unrated methods have not been tested by NIOSH, but have been developed by a recognized independent source such as OSHA.

Method Format

The NMAM methods consist of three major parts: the front page, the instructions and supporting information. See Attachment 8-1 for an example of a NMAM method.

Front Page. The first page of each method summarizes sampling and measurement parameters and gives estimates of limits of detection, working range, overall and measurement precision, and interferences. The first page also lists the method classification (full, partial, or unrated), NIOSH Registry of Toxic Effects of Chemical Substances (RTECS) number, an estimate of accuracy at the OSHA permissible exposure limit (PEL), and references to other methods.

Instructions. The second page of each method begins with lists of required reagents and equipment. These reagents and equipment reflect the conditions under which the methods were evaluated, and there may be some room for variation in actual field use. Examples of typical tolerances are as follows:

- Glass tubing used to contain solid sorbents: the inside diameter is usually not critical within the range of 4 to 6 mm; the length should be sufficient to contain the specified mass of sorbent.
- Contents of sorbent tubes: mass of sorbent should be within ±10% of the specification; separators of either glass wool or cleaned polyurethane foam unless otherwise indicated; sorbent mesh size of 20/40 unless sampling efficiency dictates otherwise. Filled sorbent tubes should be sealed to protect from contamination.

The special precautions section gives safe practices to be observed during sampling and measurement. The sampling section gives step-by-step instructions for collecting the samples in the field, including calibration and flow rate information. The sample preparation section contains information about how to prepare the samples for analysis, including desorption or digestion techniques. The calibration and quality control section discusses the measures used for calibration and provides some details of quality control required during analysis.

The measurement section gives step-by-step instructions for performing the measurement. Note that this section typically refers the reader back to page one for instrument conditions (specific column, injection temperature, detector temperature, column temperature, ramping, etc.). The calculations section shows how to calculate an air concentration based on the analytical results and volume of air sampled.

Supporting information is discussed in the section titled Evaluation of Method. This section typically advises the reader of the range of concentrations over which the method was tested, average desorption efficiencies when applicable, mean recoveries, observed breakthrough, measurement precision, observed bias and overall accuracy. Sometimes the method will also contain appendices which may contain lengthy instructions for standards preparation, sample chromatograms or mass spectral data, or detailed instructions for the preparation of sampling media.

OSHA Analytical Methods Manual

The *OSHA Analytical Methods Manual* is available on line at **www.osha.gov/dts/sltc/methods/index**.html. Over a hundred analytical methods for organic compounds and a hundred analytical methods

for inorganic compounds are available. The methods were developed at OSHA's Salt Lake City Technical Center, which performs analyses for a wide range of toxic substances and provides analytical services for the federal OSHA program.

Method Format

The OSHA methods are generally much lengthier than the NIOSH methods because they include a great deal of development and background information. The following is a general description of an OSHA method.

Title Page. The title page typically contains general information such as the name of the analyte, method number, matrix, target concentrations, verbal description of the method, recommended sampling parameters, reliable quantitation limit, standard error estimate, status of the method, date the method was evaluated, and the chemist performing the evaluation.

General Discussion. Most methods contain a general discussion section that is further broken down into several sections. A history section gives the history of the method development and evaluation, toxic effects of exposure to the analyte, locations where exposure may be expected, and physical properties of the analyte. A section on limit defining parameters summarizes the detection limit for the analytical procedure and for the overall method, the reliable quantitation limit, precision of the method, recovery percentages and reproducibility. A section on sampling procedures includes discussions of equipment used, collection technique, sampler capacity, desorption efficiency, recommended volumes and sampling rates, interferences, and safety precautions. A section on analytical procedures includes discussions of required laboratory equipment, required reagents, standards preparation, sample preparation, analytical procedures and equipment conditions, interferences, calculations and safety precautions. Another section presents backup data used in determining detection limits, quantitation limits, and so forth. Each method also contains a reference section.

Attachment 8-2 includes an example OSHA Method (ID-215) for hexavalent chromium. It should be noted that this method includes wiping of the interior of the cassette as part of the sample preparation. Studies have demonstrated that a significant percentage of particulate can be lost to the interior cassette walls. OSHA is now including this requirement for newly developed particulate methods using two or three piece sampling cassettes. At this time, the AIHA-LAP, LLC does not require that laboratories include this additional step unless it is part of the referenced method followed by the laboratory.

Method Development and Validation

As stated in section 5.4.5, validation is required for non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The extent of the validation is dependent upon the application of the method. Sections 5.4.3 and 5.4.4 of

the standard discuss laboratory-developed methods and non-standard methods. As stated previously, Section 5.4.4 and the notes provide good guidance regarding the content and considerations for laboratory-developed and non-standard methods.

Several protocols have been established for developing and evaluating analytical methods and these can be adopted or adapted as laboratory procedures used for method validation. The protocols used by OSHA for the development and evaluation of its methods are available at the OSHA website and include:

- Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis (www.osha.gov/dts/sltc/methods/chromguide/chromguide.html)
- Evaluation Guidelines for Air Sampling Methods Utilizing Spectroscopic Analysis (www.osha.gov/dts/sltc/methods/spectroguide/spectroguide.html)
- Evaluation Guidelines for Surface Sampling Methods (www.osha.gov/dts/sltc/methods/surfacesampling/surfacesampling.html)

These guidelines are also included as Appendices 8-1 through 8-3. It must be noted that most OSHA occupational health standards do not required a specific sampling and analytical method to determine compliance with exposure limits. Any method can be used provided that it yields results that are within 25 percent of the actual concentration at the 95 percent confidence level.

The fifth edition of the NIOSH Manual of Analytical Methods includes a chapter entitled "Development and Evaluation of Methods." This chapter provides an outline of a generalized set of evaluation criteria prepared by NIOSH researchers for the evaluation of sampling and analytical methodology. The full protocol referenced and used by NIOSH to evaluate methods is contained in the NIOSH document "Guidelines for Air Sampling and Analytical Method Development and Evaluation" is available at www.cdc.gov/niosh/docs/95-117/ and is included as Appendix 8-4 with this chapter. This document details a recommended protocol used for method development and includes appendices with statistical considerations and assumptions. The NMAM "Development and Evaluation of Methods" and the following sections highlight the elements of the protocol used by NIOSH to develop and evaluate methods and include a number of important considerations when developing and evaluating methods.

Literature Search

A detailed literature search can provide useful information in several areas. The search may reveal that others have already developed analytical methodologies for the analyte of interest. Existing methodologies for sample collection and/or analysis can be reviewed and evaluated for acceptability. Additional information can be obtained during the literature search that can be used in the development of a validated method if no methods currently exist. Important information that can be obtained during the literature search related to the analyte of interest includes physical properties, chemical reactions, toxic properties of the analyte, methods for analytes with similar composition or

properties, work areas where exposure may occur, possible interferences and established exposure limits (OSHA PELs, recommended exposure limits, threshold limit values).

Proposed Sample Collection and Analytical Methods

Sample collection is typically performed in industrial settings where temperature, relative humidity and cross-contamination must be accounted for. The sample collection devices must be worn by the employees for OSHA compliance sampling. The sample collection media must be rugged enough to withstand the work environment, and must not be cumbersome so workers can wear the collection devices. Compounds that are too hazardous should not be used, and the method should not require constant attention by the sampler. The following questions should be answered in considering the collection method:

- Will the worker be able to wear the collection device and perform his or her work tasks?
- Will the collection device withstand the rigors of the work place (i.e., is it appropriate for the extreme temperatures in the work place, etc.)?
- Is the sampler appropriate for the work task (i.e. if the work task involves bending over repeatedly, an impinger may not be appropriate as the liquid solution can be pulled into the pump)?
- Will the proposed collection device be compatible with the proposed analytical method?

Preliminary Evaluation of Analytical Method

The proposed analytical method should be subject to preliminary evaluation. Such preliminary evaluation includes evaluation of the instrumental limit of detection and quantitation, the linear range of calibration, instrumental precision and potential interferences for the analyte of interest. The proposed analytical method should be able to quantify the analyte at one tenth of the exposure limit with consideration of the typical sample collection volume for the collection medium.

Useful Range of Measurement

The useful range of the measurement system is important in determining if the measurement system is appropriate for the intended use. For example, if the method will be used for determining OSHA compliance, the useful measurement range should be sufficient to allow analysis of samples ranging from one half to two times the permissible exposure limit for the analyte.

Defining the useful measurement range typically includes determining the limit of detection (LOD) and the dynamic range for the method. The LOD refers to the smallest concentration of an analyte that can be detected by the measurement system. The LOD is defined as the mass of analyte that gives a signal three times the standard deviation of the blank signal above the mean blank signal. The LOD is sometimes alternately described as the smallest single result that can be distinguished from a suitable blank with 95 percent probability or as three standard deviations from the mean of seven replicate

analyses of a blank sample. The dynamic range is defined as the range over which the sensitivity is considered constant. The dynamic range should also include an upper quantitation limit (UQL). The dynamic analytical range should be wider than the anticipated working range for the method to be acceptable.

The limit of quantitation (LOQ) refers to the smallest concentration of an analyte that can be quantified by the method. The LOQ is defined as the larger of: the mass corresponding to the mean blank signal plus ten times the standard deviation of the blank signal, the mass above which recovery is greater than 75 percent, or 5–10 standard deviations from the mean of 7 replicate analyses of a blank sample.

Note that AIHA-LAP, LLC IHLAP Policies 2B.4.1 through 2B.4.3 require laboratories to establish a reporting limit by processing media samples, spiked at or below the desired reporting limit, and carrying these samples through the entire analytical process. The acceptable recovery and/or precision for these samples must be defined by the laboratory. This requirement is further discussed in Chapter 10.

Precision of the Measurement System

Precision of the measurement system must be determined separately from the precision of the collection method. Precision of the measurement system can be determined by analyzing a minimum of six standards at three different concentrations. The precision is then determined by pooling the standard deviations from the measurements at three concentrations (pooled standard deviation). The pooled standard deviation should be sufficiently low (less than 7 or 8 percent) so that when combined with the imprecision of sampling and uncorrected bias, the overall accuracy is still within 25 percent of the true concentration.

Interferences

Potential interferences during the measurement process must be defined as well as interferences during sample collection. Some interferences during the measurement process can inhibit the analysis of the analyte resulting in reported concentrations that are lower than the concentrations actually present in the sampling atmosphere. Other interferences may enhance the analysis resulting in reported concentrations that are higher than the concentrations actually present in the sampling atmosphere.

The results from the literature search should be used to help define compounds that would be expected to interfere with the analysis and to identify interfering compounds that may be present in the sampling environment. Method development should include a determination of adjustments to measurement conditions that could help eliminate or minimize interference. Interferences that cannot be eliminated should be defined in the method.

Preliminary Laboratory Evaluation of Collection Medium

Existing methods or proposed newly developed methods are evaluated to establish the acceptability of the method in a phased approach. The first set of criteria that are evaluated include the recovery of the analyte from the collection medium, the stability of the collected analyte, and stability on the collection medium prior to use.

Recovery from the Collection Medium

The ability to recover the analyte from the collection medium after sample collection is important to method development. Recovery efficiencies are usually determined by spiking multiple sets of the sample media with known concentrations of the analyte (0.1, 0.5, 1.0, and 2.0 times the exposure limit) followed by desorption, extraction, or digestion and analysis. Spiking methods are discussed in Chapter 10 of this manual. Some of the sets are analyzed and some are retained for the stability test. The results are divided by the spiked concentrations resulting in desorption, extraction or digestion efficiencies. The media should have a minimum recovery of 75 percent at all concentrations tested (0.1 to 2.0 times the exposure limit). For concentrations at 0.5, 1.0 and 2.0 times the exposure limit 90 percent recovery is preferred.

Stability on the Collection Medium

Several sets of the recovery samples are retained at room temperature for 7 days and then extracted and compared to the results of the recovery efficiency samples analyzed at day 0. Any significant differences may indicate the need to investigate alternate storage conditions such as refrigeration or freezer storage prior to analysis.

Stability of the Collection Medium Prior to Sampling

Unused sample collection media are stored for extended periods of time to determine if the formation of artifacts or interferences occur that may affect sampling. This can be accomplished by the analysis of the blank media. The collection medium may also need to be evaluated to determine if it suffers any loss in collection capacity following storage. This is determined at a later date by sampling a known atmosphere.

Sampling of Known Atmospheres

If preliminary laboratory evaluations suggest an acceptable method, further evaluation is conducted by generating and sampling a known concentration of analyte. The generation of known atmospheres can vary significantly based upon the nature and properties of the analyte. It can be very difficult to generate homogenous particulates atmospheres and the reactivity of gases and vapors may also present significant challenges. In some circumstances, media fortified with the analyte by means of spiking with solutions, suspensions or aliquots of pure gases may be required. These fortified samples are then subject to sampling of clean atmospheres (with humidity) to determine characteristics of the collection medium. Tests conducted utilizing laboratory generated atmospheres include sampler

capacity and sampling rate, sample stability, interferences and overall method precision, bias and accuracy.

Sampler Capacity and Sampling Rate

The collection capacity of the sample media is its ability to capture the analyte as air is passed over or through the media. Collection capacity is typically reported as collection efficiency (%) or breakthrough volumes. Various sampling rates (necessary for short term, peak, and TWA sampling) are also evaluated. For sorbent tube methods, the proposed sampling tube (with primary and backup sections of sorbent) is used to sample a test atmosphere at two times the expected concentration with the air treated to obtain greater than 80% relative humidity. The sampling is repeated for a series of time frames and the primary and backup sections are analyzed. The capacity of the tube is exceeded when the amount of analyte in the backup section divided by the amount in the primary section exceeds some specified value (typically 5-10%). Breakthrough of 25% is typically used as a point of sample rejection since the capacity of the sample collection device may have been exceeded.

Passive (diffusive) sampling devices can be evaluated by sampling for times varying from 7.5 minutes to 12 hours. Sampler capacity is exceeded when the analyte uptake rate is no longer constant or predictable. Filters can be evaluated using the same method as sorbent tubes and monitoring the effluent concentration using a real-time particulate monitor or a series of filters used consecutively for short time periods. A maximum loading of 2 mg total dust is recommended for filters to avoid sample loss due to mechanical sloughing.

Sample Stability

The stability of the analyte after collection on the proposed media must be determined to ensure that errors are not introduced to the method because of delays in shipping. Sample stability is usually determined by using a large number of samplers to sample an atmosphere at 0.5 to 1.0 times the exposure limit and greater than 80% relative humidity. Typically, 4 to 6 sets of 6 to 10 samples are collected. The first set of samples is analyzed immediately, the second set is analyzed after a week and the rest are analyzed over a period of 30 days. Subsequent sample results are compared to the results from the first set. Sample stability is achieved if the subsequent average results are at least 90% of the first set of results. If the sample results after 30 days meet this criterion the samples are typically reported as "stable." If the criterion is met for only one of the shorter time frames, the samples may be reported as stable for the specified period time. If stability cannot be achieved at room temperature, the study can be repeated with refrigeration of samples to determine if the samples are stable with refrigeration.

Interferences During Sampling

Interferences will most likely be present during sampling in the field. Typically, interferences are thought of in terms of compounds that compete with the analyte of interest and inhibit the ability of the media to collect the analyte, resulting in lower collection efficiency. The interfering compound

may act as a co-adsorbent or may alter the chemistry of the media, resulting in lower affinity for the analyte of interest and/or resulting in breakthrough. Compounds that are likely to be present in the sampling environment should be defined, and their effect on sample collection should be investigated. Laboratory studies can be used to investigate the interference of various compounds (including moisture) after the expected compounds have been defined. Samples are collected from an atmosphere containing only the analyte of interest and compared with a second series of samples collected from an atmosphere containing the analyte where the suspected interfering compound is introduced to the atmosphere at varying concentrations. The samples are analyzed and the results are compared to the results from the first series of samples. These results can be used to define the extent of interference for each compound.

Precision

The precision is typically determined by collecting several samples over the working range of the method. Four concentrations are common (0.1, 0.5, 1.0 and 2.0 times the exposure limit).

Bias of the Method

The bias of the method is normally determined using the results from the precision sampling experiment. The mean concentration determined by the analyses must be within 10 percent of the concentration determined by an independent method.

Overall Accuracy of the Method

The guide includes an algorithm used to calculate the overall accuracy of the method using precision, bias, and stability data. All uncorrected bias in the method as well as imprecision in sample collection and analysis when added together must meet the ±25% accuracy criterion.

Field Testing of the Method

Although field testing is not always possible and practical, this testing is an important means to determine whether the method is appropriate for its intended purpose. When feasible, replicate area and personal samples are taken to determine field precision and bias.

The guidelines presented in this chapter should assist laboratories with considerations for method development and method validation. The laboratory should also consult the following reference before initiating method development or validation.

Harmonized Guidelines for Single Laboratory Validation cf Methods cf Analysis (Iupac Technical Report) *Pure Appl. Chem.*, Vol. 74, No. 5, pp. 835–855, 2002.

This reference is included as Appendix 8-5 for the student's convenience.

Chapter 8 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 168.

1. **Which agencies/organizations publish analytical methods for air sampling and analysis specifically related to occupational exposures? (select all that apply)**

    a. EPA
    b. FDA
    c. HUD
    d. NIOSH
    e. OSHA

2. **If an analytical procedure is tested by the NIOSH evaluation protocol and meets all the factors, it is rated:**

    a. Fully evaluated
    b. Partially evaluated
    c. Unrated

3. **In what section of a method published in NMAM would you expect to find an estimate of the limit of detection?**

    a. The Sampling Section
    b. The Front Page
    c. The Special Precautions Section
    d. The Calculations Section

4. **TRUE or FALSE: If the customer specifies an out-of-date method, the laboratory should conduct the tests without consulting the customer.**

    True
    False

5. **In which section of a method published in NMAM would you expect to find a warning that a compound may be absorbed through the skin?**

    a. The Sampling Section
    b. The Calculations Section
    c. The Calibration and Quality Control Section
    d. The Special Precautions Section

6. **The section in the OSHA analytical methods manual where you would probably find the detection limit for the method would be:**

    a. The History Section
    b. The Limit Defining Parameters Section
    c. The Precision Section
    d. The Sampling Procedures Section

7. **The reagents required for desorption in an OSHA analytical method would most likely be found in which section?**

    a. Sample Procedures
    b. Limit Defining parameters
    c. Analytical Procedures
    d. Backup Data

8. **TRUE OR FALSE: If a laboratory uses a standard method, such as an OSHA or NIOSH method, no confirmation of performance is needed other than following the method exactly as written.**

    True
    False

9. **TRUE OR FALSE: Non-standard methods or laboratory-developed methods must be validated by the laboratory before use.**

    True
    False

10. **When is a laboratory required to use NMAM methods? OSHA methods?**

11. **TRUE OR FALSE: Most laboratories operate standard methods exactly as written.**

    True
    False

12. Breakthrough volumes are typically reported to describe which of the following?

a. Collection Capacity
b. Sample Stability
c. Interferences
d. Precision

13. Four to six sets of samples are collected. One set is analyzed immediately, another after a week, and the other sets are analyzed over a period of 30 days. The latter analytical results are compared to the initial results. This is an example of testing performed to determine which of the following?

a. Collection Capacity
b. Sample Stability
c. Interferences
d. Precision

14. A compound alters the chemistry of the collection media resulting in a lower affinity for the compound of interest. This is an example of:

a. Low Precision
b. Interference
c. Low Accuracy
d. Bias

15. What precision is required for a method to be validated using the SCP protocol?

a. Within 25% of the true value 95% of the time
b. Within 95% of the true value 25% of the time
c. Within 25% of the true value 25% of the time
d. Within 95% of the true value 95% of the time

16. When sampling for an analyte at the PEL concentration what is the preferred minimum recovery efficiency?

a. 50%
b. 75%
c. 80%
d. 90%

17. A sample is spiked with 250 µg of an analyte representing an exposure at the PEL. Desorption and replicate analyses show an average result of 200 µg with a standard deviation of 5%. What is the recovery efficiency?

a. 50%
b. 75%
c. 80%
d. 90%

18. Is the recovery efficiency calculated for Question 17 acceptable for the NIOSH validation process?

19. Define the following terms; LOD, LOQ, Useful Range of Measurement.

20. Where are the requirements for reporting limits for the IHLAP program specified in AIHA-LAP, LLC policies?

Chapter 9

Estimation of uncertainty of measurement (5.4.6) and Control of Data (5.4.7)

Learning Objectives

After completing this chapter the student should be able to:

- Understand requirements related to estimation of uncertainty of measurement
- Identify sources of uncertainty and their relative significance
- Understand terms related to uncertainty of measurement
- Apply techniques for estimating uncertainty of measurement
- Understand requirements for control of data
- Identify several techniques for controlling data

Introduction

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.4.6 and 5.4.7 of ISO/IEC 17025 and Sections 2A.5.4.4 through 2A.5.4.6 and Appendix G "ESTIMATION OF UNCERTAINTY OF MEASUREMENT" of AIHA-LAP, LLC Policies in conjunction with reviewing the sections presented in this chapter.

5.4.6 Estimation of Uncertainty of Measurement

Estimation of uncertainty of measurement has one of the briefer requirements sections of the standard, but this is one of the more highly debated and potentially confusing elements associated with the standard. Estimation of uncertainty can be important in the evaluation of data, since very few measurements are absolute and all are affected by various sources of uncertainty.

Section 5.4.6.2 of the standard requires testing laboratories to have and apply procedures for estimating uncertainty of measurement. It states further that in certain cases where the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement that the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Many laboratories fail to consider components of uncertainty and often report results with excessive significant figures that can give a wrong impression of the uncertainty. Section 5.4.6.3 of the standard requires that all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

It is important that laboratories understand the following terms related to estimation of uncertainty of measurement.

The ISO VIM 3rd Edition (JCGM 200:2012(E/F))defines **Uncertainty of Measurement** as "non-negative parameter characterizing the dispersion of the quantity being attributed to the measurands, based on the information used."

The AIHA-LAP, LLC Module 9 glossary further defines **Uncertainty of Measurement** as "Result of the evaluation aimed at characterizing the range within which the true value of a test result is estimated to lie, generally within a given likelihood."

- **Measurand** – "The quantity intended to being measured" (JCGM 200:2012(E/F)) Example: ug/m^3 of lead
- **Standard Uncertainty** – "measurement uncertainty expressed as a standard deviation." (JCGM 200:2012(E/F))
  Note that uncertainties are generally expressed as a relative standard deviation so they can be subsequently combined.
- **Relative Standard Deviation** – "The estimated standard deviation of a population derived from a sample of n results divided by the mean of the n results for that sample. This quantity is often referred to as the coefficient of variation. It is frequently stated as a fraction or a percentage."
- **Combined Standard Uncertainty** – "standard measurement uncertainty that is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model." (JCGM 200:2012(E/F)) Since this may be a bit confusing, the following prior definition from the ISO GUM helps to clarify this term: "Standard uncertainty of the result y of a measurement when the result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variance or covariances of the other quantities weighted according to how the measurement varies with these quantities." (ISO GUM:93). This is the method used to estimate the overall (combined) analytical uncertainty using uncertainties from various components of the process. The combined standard uncertainty is calculated as follows: Combined standard uncertainty = $\sqrt{(s_1^2 + s_2^2 + \ldots s_n^2)}$ where sn = standard deviation (standard uncertainty) for each of n steps of the process
- **Expanded Uncertainty** – "product of a combined standard measurement uncertainty and a factor larger than the number one." (JCGM 200:2012(E/F)) The expanded uncertainty is the uncertainty associated with a confidence interval.
- **Coverage factor** – "number larger than one by which a combined standard measurement uncertainty is multiplied to obtain an expanded measurement uncertainty." (JCGM 200:2012(E/F)) The normal convention is to utilize a coverage factor of 2 to approximate the 95% confidence interval as the expanded uncertainty.

- **Type A evaluation (of uncertainty)** – "evaluation of a component of measurement uncertainty by a statistical analysis of measured quantity values obtained under defined measurement conditions." (JCGM 200:2012(E/F)) This is also defined in the ISO GUM as "Method of evaluation of uncertainty by the statistical analysis of a series of observations." (ISO GUM:93)

  This is the technique commonly applied to the statistical analysis of available quality control data (replicates, laboratory control samples, spikes, etc.) to estimate uncertainty in testing laboratories. This technique includes and accounts for the contribution of many individual components and contributors to overall uncertainty.
- **Type B evaluation (of uncertainty)** – "evaluation of a component of measurement uncertainty determined by means other than a Type A evaluation of measurement uncertainty." (JCGM 200:2012(E/F)) This technique typically uses stated or estimated uncertainties for each component and contributor to overall uncertainty. This approach is typical for calibration laboratories, but rarely used for testing laboratories.

As stated previously, Section 5.4.6.3 of the standard requires that all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

When developing the laboratory estimation of measurement uncertainty procedure, the following should be considered as sources of uncertainty:

- Sampling
- Matrix effects
- Environmental conditions
- Uncertainty of masses and volumetric equipment
- Reference values
- Approximations and assumptions
- Random variations
- Sample storage conditions
- Sample digestion/extraction variability
- Instrument effects
- Reagent purity
- Assumed stoichiometry of analytical reactions
- Measurement conditions (Temp. variations)
- Computational effects (Curve fitting)
- Blank correction (value and appropriateness)
- Operator effects

When developing the laboratory procedure for estimating uncertainty of measurement it is very important that the laboratory differentiate between field sampling uncertainty and analytical uncertainty. The laboratory generally has no control over field sampling strategies, procedures and the actual field sampling process itself. Matrix effects and environmental conditions can also contribute to the uncertainty and these cannot be evaluated short of the collection and analysis of multiple field samples. Field sampling is generally considered to be one of the largest components of uncertainty, especially when dealing with particulate materials. Because replicate field samples are rarely collected, the laboratory must rely upon its routine quality control efforts to estimate and report analytical uncertainty associated with its test methods.

Laboratory subsampling is generally limited to solid and liquid samples, and can also be a significant source of uncertainty, but can be estimated by duplicate samples subject to the entire analytical procedure.

AIHA-LAP, LLC and other accrediting bodies have developed specific requirements and guidance regarding estimation of uncertainty of measurement. AIHA-LAP, LLC Policy G provides background and specific requirements for its accredited laboratories. AIHA-LAP, LLC Policy G includes sections addressing the scope, references, terms and definitions, background, policy, assessment for accreditation and guidance and examples. The specific requirements are in Section 5 of the policy. The student should thoroughly review these additional policy requirements as part of this course. AIHA-LAP, LLC has a guidance document "Guidance on the Estimation of Uncertainty of Measurement" and example Excel workbooks posted on its website to assist laboratories and data users with this topic. The 2016 versions of these documents are included as Appendices 9-1, 9-2, and 9-3. These documents are self-explanatory and provide excellent guidance in developing and applying procedures to estimate uncertainty of measurement. The student should carefully review and understand these documents. Since guidance and requirements change over time, the student is strongly encouraged to review the most current posted versions of these documents for updates. Note that these documents present several acceptable approaches.

One of the approaches acceptable to most accreditation bodies is a Type A approach using existing quality control data. This approach is described in the AIHA LAP, LLC guidance document and example workbooks and an overview is provided below.

Since laboratory control samples and/or duplicate laboratory samples are subject to most contributors to uncertainty including sample extraction, digestion, operator effects, environmental conditions, instrument calibration/standardization, and other variations, uncertainty can be generally be estimated by these QC samples. The only component that is not included in this approach is the uncertainty of the reference material(s) used to quantify results. This is typically insignificant when compared to the uncertainty of the QC data. The guidance document describes how the relative standard deviation of laboratory control samples (and replicate samples when laboratory sub-sampling is involved) can be used to estimate analytical uncertainty and how to report these estimates if

requested by the customer. In the approaches and examples provided, a confidence level of 95% with a coverage factor of 2 (K=2) is used for 30 or more observations. The laboratory can use the appropriate Student t value for a lesser number of observations.

Uncertainty (at 95% confidence level, K=2) can be expressed as:

$$X(1 \pm 2S_r)$$

where:

X is the result

S_r is the combined relative standard deviation of LCS data/replicate (as applicable)

For FoTs where laboratory control samples are not available, estimates of analytical uncertainty can be made using the results of inter-analyst quality control samples. The relative standard deviation of each set of inter-analyst samples is calculated and a pooled relative standard deviation can be calculated for samples of similar concentration. The pooled relative standard deviation can then be used in the same manner as the standard deviation of the LCS data in the formula above to estimate uncertainty.

Bias is also a component of uncertainty and must be considered. When a bias is corrected (such as through desorption efficiency correction) this bias is already accounted for. If a known bias for the test method exists and is not corrected for, the bias should be reported in addition to the estimate of uncertainty based upon random errors (standard deviation of data). Bias can be estimated through the analysis of certified reference materials (CRM) or laboratory control samples from the average percent recovery. If the average percent recovery of the CRM or LCS is 92%, the estimation of uncertainty should include a statement to the effect that the measurement results have a probable bias of -8%. Bias is further addressed in the guidance document and examples.

The following summarize the steps described in the AIHA-LAP, LLC Guidance document to estimate uncertainty of measurement:

- Define the measurand (e.g. ug/sample of lead)
- Review and identify the contributors to uncertainty
- Determine if contributors are accounted for with existing QC data
- Compile the applicable QC data and any other contributors and perform calculation of combined uncertainty where:

  $$\text{Combined uncertainty (SDc)} = \sqrt{\ [\ SD_1^2 + SD_2^2 + \ldots + SD_n^2\ ]}$$

  NOTE 1: It may be beneficial to use RSD instead of SD as it allows for the concentration dependence of SD.

  NOTE 2: Sources that have an SD of less than $^1/_3$ of the largest SD can be eliminated
- Calculate the expanded uncertainty

Apply the appropriate coverage factor 'k'. Calculate the expanded uncertainty by multiplying the combined standard uncertainty by the appropriate coverage factor (k) to give an expanded uncertainty with the desired confidence level. The factor k is the confidence interval Student distribution t-factor for n-1 degrees of freedom. For a confidence level of 95%, k is approximately 2 for a data set of 30 points or more, for normally distributed data sets. The expanded measurement uncertainty = k x SDc

- Reporting test results with the expanded measurement uncertainty

  Example: Total benzene concentration of 88 ug/sample ±11 ug/sample at the 95% confidence level (k=2).

- Where bias is present, report it along with the uncertainty as a probable bias in a manner such as the following example:

  Total lead concentration of 78 ug/sample ±12 ug/filter at the 95% confidence level (k=2). This method has an average recovery of 94%, or at this level, a probable bias of -5 ug/filter.

Note that alternate forms of reporting uncertainty and bias are acceptable as long as required information is clearly presented.

The approaches for estimating uncertainty of measurement as outlined in this chapter and detailed in the AIHA-LAP, LLC guidance document are acceptable to AIHA-LAP, LLC but other approaches may be acceptable as long as the method of calculating and reporting estimates of uncertainty of measurement are valid, adequately documented within the laboratory procedure, and understood by responsible laboratory staff.

(NOTE: Further discussion of statistical consideration of various distributions is contained in Appendix E of Appendix 9-4 "Quantifying Uncertainty in Analytical Measurement." Appendix 9-4 is a very good reference and includes other approaches to estimate the uncertainty of measurement.)

Additional references include:

1. A2LA Policy on Estimating Measurement Uncertainty For Testing Laboratories. https://www.a2la.org/policies/a2la_p103.pdf
2. Interpretation and Guidance on Estimation of Uncertainty of Measurement in Testing APLAC TC005. https://www.aplac.org/documents/tc/tc_005.pdf

5.4.7 Control of Data

This section of the standard requires that all types of calculation and data transfers are checked in a systematic manner. AIHA- LAP, LLC Policies 2A.5.4.5 and 2A.5.4.6 additionally require specific content of these checks and that the check is done by an independent reviewer and prior to the reporting of results. It is suggested that labs use a brief checklist, project tracking form, or some electronic method to document this independent review.

Section 5.4.7.2 mandates validation of computer software developed by the laboratory and procedures to protect electronic data. As stated in the Notes to the standard, "Commercial off-the-shelf software (e.g., word processing, database and statistical programmes) in general use within their designed application range may be considered to be sufficiently validated. However, laboratory software configuration/modifications should be validated as in 5.4.7.2 a)." Software such as chromatography software, data acquisition software and commercial LIMS systems can be considered sufficiently validated, however, modifications to LIMS, and the specific functionality (formulae) of spreadsheet and database applications developed by the lab must be verified. The use of manual calculation or a set of test data within known output can be used for such verifications. Be certain to maintain these records of verification. Laboratories are encouraged to protect formulae wherever feasible within spreadsheet template applications to prevent the unauthorized or unintentional revision of these formulae. Once protected, verification is not required unless the template is revised. Other methods of protecting the integrity and confidentiality of electronic data include password protection of files, data directories, and/or LIMS functions and confidentiality statements on reports, fax cover sheets and e-mail footers, and the use of pdf files in email transmittals.

For additional guidance on software validation consult "Software Validation in Accredited Laboratories, A Practical Guide." This document is available at **www.a2la.org/guidance/adequate_for_use.pdf** and is included as Appendix 9-5 for the student's convenience.

Chapter 9 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 169.

1. **Define uncertainty of measurement.**

2. **What is considered the largest source of uncertainty associated with the overall uncertainty of measurement for occupational health and environmental samples?**

3. **When estimating uncertainty, the following standard uncertainties (standard deviations) for different steps of the analytical process were determined. 2.2, 1.4, 3.0, 9.8, 0.7, and 1.4.**

    a. Calculate the combined standard uncertainty using these standard uncertainties.

    b. Calculate the expanded uncertainty using a coverage factor of 2.

4. **In general, when can a source of uncertainty be considered insignificant and removed from consideration?**

5. **What routine quality control sample data can be used to estimate analytical measurement uncertainty?**

6. **List the required elements that must be included in data review to satisfy AIHA-LAP, LLC requirements.**

7. **List several steps that laboratories can take to protect electronic records during storage or transmission.**

8. **True or False? Significant bias must be reported when reporting estimates of uncertainty.**

   True
   False

Chapter 10

Equipment (5.5) and Traceability of Measurement (5.6)

Learning Objectives

After completing this chapter, the student should be able to:

- Identify laboratory equipment that requires calibration and ongoing maintenance
- Establish a calibration and maintenance schedule for laboratory equipment
- Understand traceability requirements and how they impact laboratory operations
- Properly document equipment calibration
- Understand why proper equipment maintenance is important

Introduction

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.5 and 5.6 of ISO/IEC 17025 and Sections 2A.5.5 and 2A.5.6 and Appendix H "TRACEABILITY OF MEASUREMENT" of AIHA-LAP, LLC Policies in conjunction with reviewing the sections presented in this chapter.

5.5 Equipment

The accuracy of data produced during the measurement process is directly related to the bias and precision of the instruments used to perform the measurements. The bias and precision of measurement devices can vary greatly over time because of improper or inadequate maintenance procedures and improper calibration. Therefore, proper maintenance and calibration of laboratory instruments can help eliminate or reduce bias and improve precision during the measurement process.

All equipment associated with the analysis of samples that involves the measurement of some parameter will require calibration. Care must be taken to calibrate the equipment at regular intervals ensuring that all measurements made with the equipment are traceable to a known standard. The frequency of calibration will depend on the stability of the measurement instrument and will vary from instrument to instrument.

There are two basic types of calibration required in a laboratory—periodic calibration and at-use calibration. Periodic calibration is performed on instruments, such as thermometers and balances, that are fairly stable over time. These instruments require full calibration verification at specified frequencies with user checks in between the full calibrations. Less stable instruments, such as spectrophotometers and gas chromatographs, need at-use calibrations. These instruments require

full-range calibrations performed on a routine basis by the analyst with on-going checks during each sample run using initial and continuing calibration verification checks.

Requirements regarding instrument calibration and maintenance are detailed in Section 5.5 of the standard and Section 2A.5.5 of AIHA-LAP, LLC Policies. Additional calibration requirements relevant to the IHLAP program are included in Section 2B.3 of the AIHA-LAP, LLC Policies. The standard has a number of relatively straight forward requirements in Sections 5.5.1 through 5.5.12 of the standard. Several of these sections are expanded on in the following paragraphs.

Section 5.5.1 – It is rare that equipment is not under the direct control of the laboratory. This may occur when using shared equipment with other departments or laboratories. In those circumstances the laboratory must ensure that all requirements related to calibration and maintenance procedures and records are satisfied.

Section 5.5.3 – Requires that equipment be operated by authorized individuals as in Section 5.2.5 of the standard. A separate authorization for instrument operation is not required if such operation is part of an authorization to conduct a test method using that equipment. In other words, a specific authorization to operate a gas chromatograph is not required if the analyst is already authorized to conduct aromatic hydrocarbon analysis by gas chromatography.

Section 5.5.4 – Requires that equipment is uniquely identified. Major instrumentation is typically identified by a serial number or a capital asset tag. Laboratories must be sure that support equipment such as mechanical pipettes and thermometers are uniquely identified.

Section 5.5.5 – This section lists a number of specific requirements for equipment records. It is suggested that laboratories use these elements to develop a template for either a written or electronic logbook for each instrument. It should be noted that laboratories frequently fail to list current operating software as required by Sections 5.5.5 and 5.5.5.a. Section 5.5.5.f requires that records are maintained of the dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria and the due date of next calibration. For routinely calibrated instruments, maintenance of instrument calibration records within data packages or project files is considered adequate.

Section 5.5.5.g – Requires records of the maintenance plan, where appropriate, and maintenance carried out to date. Proper maintenance of equipment is critical to ensuring that the equipment performs as required and produces accurate and precise measurements. Written maintenance plans and schedules must be developed for each piece of major equipment. In many instances, these written plans may be obtained from the manufacturer of the equipment. Alternately, an outside maintenance firm may be contracted to perform ongoing maintenance for laboratory equipment. In such cases, a copy of the maintenance plan used by the outside contractor should be maintained in the laboratory.

The maintenance plans and schedules should be established based on the following considerations at a minimum:

- Type of equipment
- Frequency of use
- Type of use
- Manufacturer's recommendations
- Environment (heat load, relative humidity, foot traffic, etc.)
- Personal experience

AIHA-LAP, LLC Policy 2A.5.5.1 – Additionally requires that the name or initials of the person performing the maintenance or repair shall be recorded. It is also important to remember that routine maintenance performed by the instrument user must also be documented.

Section 5.5.8 – Major equipment, calibrated at time of use, does not need to be labeled with its calibration status. Calibration instructions and the records described under Section 5.5.5.f above are sufficient. This requirement, however, is applicable to support equipment such as balances, mechanical pipettes and temperature reading devices which require only periodic full calibration.

Section 5.5.9 – Although one may consider this section to be inapplicable for a fixed site laboratory, do not lose sight of the need to verify the calibration status of equipment after it may be sent out for repair or calibration by an external provider. Note that AIHA-LAP, LLC Policy 2A.5.5.3 additionally requires that when possible, any external calibration service used shall be a calibration laboratory accredited to ISO/IEC 17025:2005 by a recognized accreditation body.

Section 5.5.10 – This requirement is typically satisfied by continuing calibration verification checks of major equipment and periodic calibration checks of support equipment such as balances, pipettes and temperature reading devices. AIHA-LAP, LLC Policy 2A.5.5.2 additionally requires that the frequencies of calibration checks are specified by the laboratory.

Section 5.5.11 – Addresses correction factors. The use of correction factors is atypical with the exception of correction factors that may be applied to working temperature monitoring devices (thermometers) and inter-element corrections for ICP methods. Correction factors for temperature monitoring devices are discussed further in the traceability Section (5.6).

Section 2B.3 of the AIHA-LAP, LLC Policies – includes addition requirements for method reporting limits and instrument calibration specific to analytical procedures for industrial hygiene testing, with the exception of gravimetric and asbestos analyses. The student should review these carefully. The following paragraphs discuss these requirements.

Section 2B.3.1 – This section requires that minimum reporting limits are established initially by analyzing media spiked samples, prepared at or below the desired minimum reporting limit concentrations, and taken through the entire analytical process. Acceptance criteria must be documented. The reason for this requirement is that sampling media desorption characteristics and media and reagent analyte background concentrations and interferences can significantly affect quantitation at lower concentration ranges. The use of media-spiked samples at or below the desired minimum report limit takes these issues into account. Acceptance criteria of 75-125% recovery and a relative standard deviation of 25% are recommended in consideration of NIOSH method development guidelines. Note that a statistically based detection limit study (e.g., seven replicate samples) is not required to establish or verify the reporting limit, although these studies may be helpful in determining an appropriate target for the reporting limit. The laboratory should consider the typical air volume sampled, recommended or regulated exposure levels, and potential media and reagent background concentrations and interferences in establishing the reporting limit. A guideline of no less than three to five times the background (media blank) concentration of analyte should be considered for those analytes with a detectable background concentration.

Section 2B.3.2– During the analysis of samples, instrument performance at the minimum reporting limit concentration shall be verified with each analytical batch through the analysis of an analytical standard prepared at or below the analyte's minimum reporting limit concentration. Acceptance criteria shall be documented." This standard can be part of the calibration standard curve or can be a separate standard. An acceptance criterion of 80-120% or 75-125% is typical.

Section 2B.3.3 – States, "At least annually or when there is a change in methodology or instrumentation, minimum reporting limits shall be re-established by a process that requires analysis of a media spiked sample prepared at or below the minimum reporting limit concentration, and taken through the entire analytical process. Acceptance criteria shall be documented." This requirement is often overlooked by labs, especially for non-routinely analyzed analytes. If certain analytes are not requested for over a year, then the laboratory must include these checks upon the next request. Some laboratories include media spiked samples at the reporting limit with each batch of samples, although only once each year is required.

Section 2B.3.4 – Establishes the minimum requirements for calibration curves and a requirement for verification of the standard curve with an independent standard. Independent standards are either an independent preparation from a neat material or a preparation from a standard material other than the one used to prepare the calibration curve. Acceptance criteria for the standard calibration curve and the independent calibration verification standard shall be documented. Laboratories typically use a correlation coefficient (r) of 0.995 or greater or an RSD of the response factors of 20% or less for calibration curve acceptability. An acceptance criterion of 90-110% recovery for the independent calibration verification standard is also typical. Note that ICP-AES is an exception and where possible, a minimum of a two-point calibration plus a blank shall be performed. Multiple point calibration curves are possible and required for ICP-MS.

Section 2B.3.5 – Requires the analysis of an appropriate interference check standard at the beginning and at the end of each analytical run for inductively coupled plasma, emission spectroscopy (ICP-AES). Such solutions may contain only interfering elements, or a combination of interfering elements and target analytes to check interference correction. An acceptance criterion of 80-120% for low-level target analytes in the presence of interfering elements is typical.

Section 2B.3.6– The initial calibration curve can be utilized provided the instrument calibration/ standardization is verified each 24-hour period of use or at each instrument start-up if the instrument is restarted during the 24-hour period, by analysis of a continuing calibration verification (CCV) standard. Acceptance criteria shall be documented. Acceptance criteria of 90-110% for inorganic methods and 85-115% for organic methods are typical. There are no other frequency requirements for continuing calibration verification standards unless specified by the referenced method. Good laboratory practice includes a calibration verification standard at the end of the analytical run and generally a CCV every 10 to 20 samples.

Section 2B.3.7 – Requires that all sample extract concentrations are bracketed by the working quantification ranges (calibration standards) of the method. Any samples outside the working quantitation range require dilution and re-analysis. In addition, continuing calibration verification standards and continuing calibration blanks shall be analyzed in accordance with the specified test methods. Acceptance criteria of +/- ½ the absolute value of the reporting limit for blanks and 90-110% for continuing calibration verification standards are typical.

Although the standard and AIHA-LAP, LLC policies do not explicitly address support equipment, calibration and maintenance of this equipment is required as it falls under Section 5.5 of the standard. The following sections provide guidance in meeting AIHA-LAP, LLC expectations for support equipment.

Support equipment can be classified as equipment used to measure and/or regulate mass, volume, temperature, pressure, humidity and energy. This includes equipment such as balances, mechanical pipettes, dispensers, thermometers, pressure sensors and gauges, heating/digestion systems, autoclaves, etc. Each piece of equipment must have a documented procedure for calibration and/ or calibration checks. AIHA-LAP, LLC Policy H "TRACEABILITY OF MEASURMENT" addresses traceability requirements related to reference materials, reference standards, and calibration and verification checks of instruments and support equipment. Table 5-1 of this policy document includes a listing of reference standards and support equipment requiring calibration and verification. The following are recommendations:

Balances

- Typically subjected to annual servicing and calibration by an outside vendor. Although recommended, this is not required. Calibration is only required initially and if verifications fail.
- If outside calibration is used, it must be provided by an ISO/IEC 17025 accredited calibration

laboratory. Several balance manufacturers and a number of calibration laboratories provide this service.

- Balances should be verified with masses spanning the working range each day of use with documented acceptance criteria. Acceptance criteria are typically within no more than 0.1% of the actual mass for masses greater than 100 mg. Criteria are dependent upon use and are up to the laboratory.
- The laboratory must have a documented procedure and maintain records of balance calibrations and verification checks.

Mechanical Pipettes/Dispensers

- May be subject to periodic servicing and calibration by an outside vendor. If outside calibration is used, it must be provided by an ISO/IEC 17025 accredited calibration laboratory. Several pipette manufacturers and number of calibration laboratories provide this service.
- Mechanical pipettes should be checked gravimetrically by replicate dispensing of volumes representing the volumes at all ranges of use. Adjustable mechanical pipettes used at less than 50% of maximum volume can be prone to greater errors. The technique used with mechanical pipettes is also important, so the volume dispensed should be checked for various operators.
- Manufacturers' specifications are recommended as acceptance limits.
- Monthly to quarterly frequency and 1-2% error/imprecision limits are typical.
- Documented procedure, records and acceptance limits for calibration checks are required.

Thermometers/Temperature Monitoring Devices

This equipment includes working thermometers, and dial-type and digital temperature monitoring devices used for monitoring the temperatures of refrigerators, freezers, incubators, Refractive Index (RI) fluids during PLM analysis (AIHA-LAP, LLC requirement), hotplates, block digestors, ovens, autoclave temperatures, water baths and other critical temperatures with specific method requirements.

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- Typical frequency for monitoring temperature of critical equipment or activities is each day of use or as needed. Continuous monitoring systems for refrigerators and freezers with alarm capabilities are best to alert the laboratory of problems (including off-hours and weekends) but are not required.
- Annual checks of temperature monitoring devices against a NIST or NMI traceable thermometer are recommended; a procedure is required.
- Documented procedures, records and acceptance limits for calibration checks are required.
- The NIST/NMI traceable standard thermometer must be initially and recalibrated as needed by an accredited calibration laboratory. A typical recalibration interval is every two to five years, depending on type and use.

Other Support Equipment

Other support equipment may include energy level indicators (microwave digestion systems) and pressure sensors.

- Typically manufacturer's recommended procedures, calibration/calibration check intervals and acceptance limits are used.
- Calibrations are performed by original equipment manufacturer (OEM) or an accredited calibration laboratory, where possible.
- Documented procedure, records and acceptance limits for calibration checks are required.

5.6 Measurement Traceability

Section 5.6.1 is simply an amplification of Section 5.5 requirements requiring that all equipment used for tests and/or subsidiary measurements that have a significant effect on the accuracy or validity of the result of the test must be calibrated before being put into service.

Section 5.6.2.1 is directed to calibration laboratories and requires that the calibration program is operated as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI). Metrological Traceability (traceability) is defined by the ISO VIM as: "property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty" (VIM 2.41 JCGM 200:2012).

This section also requires that when using external calibration services, that the traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories must contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification. The notes of this section explain how this may be accomplished.

Section 5.6.2.1.2 recognizes that there are certain calibrations that currently cannot be strictly made in SI units and provides several alternatives.

Section 5.6.2.2 addresses testing laboratories and mandates that the requirements of 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. AIHA-LAP, LLC Section 2A.5.5.5 requires that when possible, any external calibration service used must be a calibration laboratory accredited to ISO/IEC 17025 by a recognized accreditation body. This will provide the traceability required by this section; however, laboratories must ensure that they request calibration certificates that include required elements.

AIHA-LAP, LLC and other accrediting bodies have developed specific requirements and guidance regarding traceability of measurement to clarify terminology, applicability, and requirements for their accredited labs. AIHA-LAP, LLC Policy H "Traceability of Measurement" provides background and specific requirements for its accredited laboratories. AIHA-LAP, LLC Policy H includes sections addressing the scope, references, terms and definitions, background, and specific policy requirements. The specific requirements are in Section 5 of the policy. The student should thoroughly review these additional policy requirements as part of this course. AIHA-LAP, LLC has also created a guidance document "Guidance on Traceability of Measurement" that is posted on its website to assist laboratories and data users with this topic and implementing policy requirements. The 2016 version of this guidance document is included as Appendix 10-1. These policy and guidance documents are self-explanatory and provide excellent guidance in developing and applying procedures to support the traceability of measurement. The student should carefully review and understand these documents. Since guidance and requirements change over time, the student is strongly encouraged to review the most current posted versions of these documents for updates.

Section 5.6.3.1 of the standard addresses reference standards. It is important that laboratories understand these terms as "standards" are commonly used within the laboratory environment to calibrate instruments, when in fact these are reference materials. The VIM defines Reference Measurement Standard (reference standard): "measurement standard designated for the calibration of other measurement standards for quantities of a given kind in a given organization or at a given location (VIM 5.6 JCGM 200:2012). To provide clarity for testing laboratories, AIHA-LAP, LLC Policy H uses the term reference standard to be those related to physical attributes such as mass, length, and temperature that are defined by convention as traceable to the SI through an NMI such as NIST).

Typically only balance weights (masses), reference (primary) thermometers and stage micrometers are reference standards used by chemical testing laboratories. Section 5.6.3.1 requires a program (schedule) and procedure for the calibration of its reference standards and that reference standards are calibrated by a body that can provide traceability as described in 5.6.2.1. AIHA-LAP, LLC Section 2A.5.5.5 also applies here and requires that, where possible, the external calibration laboratory is ISO/IEC 17025 accredited.

It is suggested that balance weights (masses) and the reference thermometer are calibrated every two to five years, as dependent upon use, by an ISO/IEC 17025 accredited calibration laboratory. ASTM Class 1 (formerly Class S) masses are suitable for almost all applications. A lesser class may be suitable depending on the laboratory application. The reference thermometer utilized by the laboratory should have sufficient resolution and accuracy for the laboratory applications. Several reference thermometers in varying ranges may be required. All working thermometers and/or temperature monitoring devices should be checked (verified) against the reference thermometer(s) at least annually at the temperature of use according to a documented laboratory procedure. A correction factor can be applied to the working thermometers. In general, if the correction exceeds 1 degree centigrade, the working thermometer should be replaced although this depends on the application.

Section 5.6.3.2 addresses reference materials. The VIM defines Reference Material (RM) as: "material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties" (VIM 5.13 JCGM 200:2012). To provide clarity for testing laboratories, AIHA-LAP, LLC Appendix H uses the term reference material to be those related to chemical and microbiological references. Reference materials include neat materials, chemical solutions, and microbiologic cultures. Reference materials are what laboratories commonly refer to as standards (or calibration standards) and what are referred to in several AIHA-LAP, LLC requirements as standards. Reference materials include but are not limited to NIST Standard Reference Materials (SRM®s), certified reference materials (CRMs), analytical standard solutions, neat chemical materials and reference cultures (such as ATCC).

The term Standard Reference Material (SRM®) is registered by the National Institute of Science and Technology (NIST). A certified reference material is one that is traceable to a NIST SRM® or to consensus values. AIHA-PAT, LLC IHPAT, and ELPAT samples have consensus values assigned and are considered certified reference materials by AIHA-LAP, LLC.

Section 5.6.3.2 further requires that reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials. Internal reference materials (prepared by the laboratory) shall be checked as far as is technically and economically practicable. Reference materials are generally not traceable to the SI, but traceability to a National Metrology Institute (NMI) is considered acceptable. The National Metrology Institute for the United States is the NIST. AIHA-LAP, LLC Section 2A.5.6.5 states, "Reference materials shall have a certificate of analysis that shows specific NIST-traceability, or equivalent, and uncertainty, when possible. The certificate must show the specific SRM® used for traceability." A statement of "NIST traceable" without reference to a specific NIST SRM® is not sufficient. NIST SRM®s are available for a number of inorganic compounds and metals, but there are very few organic SRM®s. In any event, laboratories should request and maintain a certificate of analysis for all reference materials purchased by the laboratory. At this time, there is a rapidly expanding group of accredited reference material producers (RMP). Reference material producers are currently accredited to ISO Guide 34 in conjunction with ISO/IEC 17025. ISO Guide 34 includes provisions for homogeneity, testing and traceability. A new international standard (ISO 17034) is expected to replace Guide 34 sometime in 2016 or 2017 as the basis for the accreditation of reference material producers. The use of an accredited RMP with appropriate certificates of analysis provides acceptable traceability. AIHA-LAP, LLC requires that any new reference materials are purchased from accredited RMPs, when available, although existing stocks of reference materials may be used until exhausted.

Intermediate checks (Section 5.6.3.3) needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials are generally satisfied by the preparation and analysis of an independent calibration verification standard.

Section 5.6.3.4 requires the laboratory to have procedures for the safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration

and in order to protect their integrity. Good laboratory practices and reasonable safeguards often addressed in these procedures include:

- The need for adequate packing and protection of reference standards (masses, stage micrometer, reference standard) during shipping for external calibration and storage in the laboratory.
- Proper handling of masses with gloved hands or nylon tipped forceps to prevent scratching or contamination with hand oils.
- Protection of reference materials by avoiding direct pipetting out of reference material solution containers and discarding excess material instead of returning it to original container.

AIHA-LAP, LLC Policies 2A.5.6.1 through 2A.5.6.6 include additional specific requirements related to traceability of reference materials and reagents used in the laboratory. The following includes requirements and offers several recommendations:

2A.5.6.1 "Requirements for reagents and standards shall be specified by the laboratory." Specification within the SOP is considered adequate to meet this requirement.

2A.5.6.2 "Reagents and standards shall be inspected, dated and initialed upon receipt. Calibration standards and analytical reagents shall have an expiration or reevaluation date assigned." Containers must be physically dated and initialed upon receipt. If the supplier does not include an expiration date, the laboratory must assign one. The assigned expiration date is up to the laboratory and can be based upon experience. Expiration dates of five to ten years are typical for stable neat materials. Expiration dates for solutions are generally shorter.

2A.5.6.3 "Reagents and standards shall not be used beyond assigned expiration dates. Materials designated for reevaluation, which are determined to have adequate purity upon reevaluation, shall be assigned a new expiration date." Although reevaluation is permitted by AIHA-LAP, LLC policies, it is discouraged. If reevaluation is conducted, it is suggested that the concentration of a reevaluated material be statistically indistinguishable from another currently unexpired material.

2A.5.6.4 "Strict control and documentation of reagent solutions and calibration standards shall be maintained." This mandates detailed records of reagent and calibration standard preparation, lot numbers and usage.

2A.5.6.5 "Documentation of standard and solution preparations shall include:

- a description of the content,
- the date of preparation,
- concentration and/or purity of parent material,
- manufacturer and lot number of parent material,
- assigned expiration date, and
- the preparer's initials.

Solutions shall be adequately identified to trace back to preparation documentation." Once again, detailed records are needed. Laboratories are encouraged to develop a standardized logbook format (either written or electronic) to record this information. It is also suggested that the amount of parent material, final volume and final concentration of each component are detailed in these preparation records.

2A.5.6.6 "Laboratories shall comply with the requirements of the AIHA-LAP, LLC Policy on Traceability of Measurement, Policy Appendix H. Refer to the AIHA-LAP, LLC guidance document, Guidance on Traceability of Measurement on the AIHA-LAP, LLC website for additional information." The following sections include the requirements of the Traceability of Measurement Policy and with comments and/or recommendations as needed:

Policy H 5.1 "Laboratories accredited by AIHA-LAP, LLC shall demonstrate, when possible, that calibrations of critical equipment and hence the measurement results generated by that equipment, relevant to their scope of accreditation, are traceable to the SI through an unbroken chain of calibrations." This is accomplished through in-house or external calibrations using accredited calibration services, with traceable reference standards and/or reference materials with documented uncertainty of measurement.

Policy H 5.2 "External calibration services shall, wherever possible, be obtained from providers accredited to ISO/IEC 17025 by an ILAC recognized signatory, a CIPM recognized National Metrology Institute (NMI), or a State Weights and Measures Facility that is part of the NIST Laboratory Metrology Program. Calibration certificates shall be endorsed by a recognized accreditation body symbol or otherwise make reference to accredited status by a specific, recognized accreditation body, or contain endorsement by the NMI. Certificates shall indicate traceability to the SI or reference standard and include the measurement result with the associated uncertainty of measurement." Be certain that the service requested and calibration certificate includes the accreditation body symbol and laboratory accreditation number as some calibration laboratories offer several tiers of calibrations that may not meet these requirements. The AIHA-LAP, LLC "Guidance on Traceability of Measurement" provides additional discussion regarding purchasing external calibration services.

Policy H 5.3 "Where traceability to the SI is not technically possible or reasonable, the laboratory shall use certified reference material provided by a competent supplier, or use specified methods and/or consensus standards that are clearly described and agreed to by all parties concerned. A competent supplier is an NMI or an accredited reference material producer (RMP) that conform with ISO Guide 34 in combination with ISO/IEC 17025, or ILAC Guidelines for the Competence of Reference Material Producers, ILAC G12. Conformance is demonstrated through accreditation by an ILAC recognized signatory. Note: There are many gaps in the measurement traceability of the calibration infrastructure in the world and there are a relatively small, but increasing, number of accredited reference material producers. In recognition of this situation, AIHA-LAP, LLC requires the use of accredited reference material producers only for newly purchased reference materials with known accredited RMPs (e.g. many metals, inorganic anions, some organic mixture, some microbial organisms). Existing reference materials may be used until expired or exhausted. This requirement is not enforced for standards not readily available from an accredited RMP." Reference materials from accredited RMPs are now available for metals solutions and many inorganic and organic solutions and a number of neat materials. The AIHA-LAP, LLC "Guidance on Traceability of Measurement" provides additional discussion regarding purchasing reference materials.

Policy H 5.4 "Reference materials shall have a certificate of analysis that documents traceability to a primary standard or certified reference material and associated uncertainty, when possible. When applicable, the certificate must document the specific NIST SRM® or NMI certified reference material used for traceability." A certificate of analysis is required for all reference materials used by the laboratory, even if traceability and uncertainty cannot be provided. Accredited reference material producers can supply materials with documented traceability and stated uncertainty, but as is the case with calibration laboratories, some reference material producers provide various tiers of products that may not satisfy these requirements.

Policy H 5.5 "Calibrations performed in-house shall be documented in a manner that demonstrates traceability via an unbroken chain of calibrations regarding the reference standard/material used, allowing for an overall uncertainty to be estimated for the in-house calibration." Instrument calibration traceability requirements can be satisfied with documented analytical SOPs, trained personnel, and traceable reference materials. Equipment calibrations (other than analytical instrument calibrations) are not typically performed in-house by testing laboratories, but when done, must include statements of estimated uncertainty and be performed by trained personnel using traceable reference standards. The AIHA-LAP, LLC "Guidance on Traceability of Measurement" and CALA calibration examples provide additional information regarding in-house calibrations and associated traceability and uncertainty of measurement.

Policy H 5.6 "Calibrations shall be repeated at appropriate intervals, the length of which can be dependent on the uncertainty required, the frequency of use and verification, the manner of use, stability of the equipment, and risk of failure considerations. Table 5-1 includes a list of reference standards and support equipment, commonly found in AIHA-LAP, LLC accredited laboratories that

require calibration." The frequency of calibrations is up to the laboratory. Recommendations earlier in this chapter and the AIHA-LAP, LLC "Guidance on Traceability of Measurement" provide additional discussion and recommendations regarding procedures and frequencies of calibrations.

Policy 5.7 "Periodic verifications shall be performed to demonstrate the continued validity of the calibration at specified intervals between calibrations. The frequency of verifications can be dependent on the uncertainty required, the frequency of use, the manner of use, stability of the equipment, and risk of failure considerations." The frequency of verifications is up to the laboratory. Recommendations earlier in this chapter and the AIHA-LAP, LLC "Guidance on Traceability of Measurement" provide additional discussion and recommendations regarding procedures and frequencies of verifications.

Policy 5.8 "The laboratory shall have procedures describing their external and internal calibration and verification activities and frequencies, and the actions to follow if the equipment is found to be out of acceptable specification. Although the frequency of recalibration can be extended, it cannot be eliminated. The procedures shall describe the action(s) that will be taken when recalibrations or verifications fail to meet the established criteria, including the use of the nonconformance and corrective action system to identify the root cause, prevent recurrence, and evaluate the impact to data reported since the last passing calibration or verification, including data recall where appropriate." For simplicity, it is suggested that the laboratory document these procedures in a matrix similar to that in Table 5-1 of the "Guidance on Traceability of Measurement" with addition discussion of actions to be taken when calibrations or verifications fail to meet established criteria.

Policy H 5.9 "Laboratory staff performing in-house calibrations and verifications shall have received documented training." Training records and authorizations are required for these staff members.

Chapter 10 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 171.

1. Of the following equipment, which would never require calibration?

a. Ion chromatograph
b. 10 mL mechanical pipette
c. Bunsen Burner
d. Thermometer

2. Define traceability.

3. Name three reference standards common to many occupational health testing laboratories.

1.
2.
3.

4. The solutions used to calibrate an ion chromatograph, gas chromatograph, and atomic absorption spectrophotometer are prepared from what?

5. Which of the following methods would be the most appropriate way to verify a 1 mL mechanical pipette?

a. Fill the pipette with acetone, determine the quantity using a GC.
b. Fill the pipette with water, transfer the water to another pipette and compare the two measurements.
c. Fill the pipette with water, drain the water and weigh the water on an analytical balance.
d. Pipettes don't require verification.

6. **A. What records must be maintained for laboratory equipment?**

   B. Where are these requirements specified?

7. **TRUE OR FALSE: AIHA-LAP, LLC policies require laboratories to determine the reporting limit for all laboratory procedures.**

   True
   False

8. **What is the minimum number of calibration standards required by AIHA-LAP, LLC IHLAP policies?**

9. **An analyst runs initial calibration verification standards along with a set of samples. What errors might the results from these solutions be used to detect?**

10. **According to AIHA-LAP, LLC requirements, how must reporting limits be determined for analyses conducted under the IHLAP program?**

Chapter 11

Sampling (5.7) and Handling of Test Items (5.8)

Learning Objectives

After completing this chapter, the student should be able to:

- Understand requirements related to sampling
- Identify documentation used to protect sample integrity in the field
- Summarize the contents of the chain-of-custody form/analytical request forms and understand the need for these forms
- Summarize the procedures for receiving samples at the laboratory
- Identify the information that should be verified upon receipt of samples at the laboratory
- Identify sample log-in procedures at the laboratory including information that should be recorded for samples at log-in
- Identify sample storage and sample retention considerations

Introduction

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.7 and 5.8 of ISO/IEC 17025 and Sections 2A.5.7 and 2A.5.8 of AIHA-LAP, LLC Module 2A in conjunction with reviewing the sections presented in this chapter.

The laboratory analyses of samples and reporting of data represent the final steps in the sampling process. The reliability of the generated data depends on the laboratory procedures, but begins with the procedures used to collect the samples. If significant errors are introduced during the sampling procedures, the data produced may be inaccurate no matter how accurate and precise the analyses are. Therefore, any discussion of QA/QC procedures should address the sampling process from collection through sample disposal. This chapter will focus on procedures used to collect and send the samples to the laboratory and the laboratory sample handling procedures.

5.7 Sampling

In many circumstances laboratories are not involved in the collection of samples submitted to the laboratory. In those instances, laboratories are not required to comply with the requirements of ISO/IEC 17025:2005 Section 5.7. Exceptions are sub-sampling of customer samples prior to analysis (typically applicable only to bulk samples) and procedures for sampling potential laboratory contamination

(see Chapter 6). The sub-sampling of bulk samples is typically included in the preparative steps of the laboratory's analytical procedures and may involve mixing, grinding and/or drying to create a homogenous sample and representative sub-sample.

It is suggested that laboratories assist their customers or field sampling staff with information regarding sampling materials, sampling containers, preservatives and shipping instructions. The laboratory is not required to provide sampling materials, sampling containers, sampling methods, or preservatives to the customer, but doing so does assist in making sure the appropriate sampling media is used. A number of laboratories have developed sampling guides for use by their customers and some laboratories provide complimentary sampling media and containers as a service to customers provided samples are returned to the laboratory for analysis. Others may provide sampling media for a fee.

AIHA-LAP, LLC Section 2A.5.9.1.1 states: "Laboratories shall advise customers to supply specimens of blank sampling media from the same source lot as was used for collecting the field samples." While customers cannot be required to provide blank sampling media, the laboratory must advise them to do so. It is suggested that this request is included with any sample shipping information or media provided to customers.

The following sections include the requirements of ISO/IEC 17025:2005 for laboratories that conduct field sampling.

Section 5.7.1 of the standard states: "Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the test and calibration results." Those responsible for developing the sampling plan should consult such references as the NIOSH "Occupational Exposure Sampling Strategy Manual" or EPA SW-846 for statistical considerations. Application of these strategies typically yields a very large number of samples to be collected. Most often, the professional judgment of the field sampler is used to select representative or "worst case" scenarios to evaluate actual or potential exposures.

Sections 5.7.2 and 5.7.3 require that customer required deviations, additions or exclusions from the documented sampling procedure are recorded in detail and communicated to the appropriate personnel. Laboratories conducting sampling must have procedures for recording relevant data and operations relating to sampling and the records of sampling must include:

- the sampling procedure used,
- the identification of the sampler,
- environmental conditions (if relevant),
- diagrams or other equivalent means to identify the sampling location as necessary, and
- if appropriate, the statistics the sampling procedures are based upon.

Appendix 11-1 provides useful information regarding considerations when selecting, conducting or recommending sampling media and methods.

Data reliability is dependent upon every step in the collection and analytical process. The data are only as reliable as the weakest element in the process. The reliability of the analytical data may be questioned if the integrity of the samples between field collection and analysis at the laboratory cannot be verified. Sample integrity is extremely important when the data will be used for litigation. Data have been ruled inadmissible in many court cases simply because the integrity of the samples could not be accounted for throughout the entire process. However, the importance of sample integrity should not be discounted in generating data that will not necessarily be used for litigation support. Bear in mind that these data are used to make health risk assessment decisions and sample and data integrity must be maintained. Therefore, all samples should be treated as if the analytical data could potentially be used in a court of law. There are several procedures that can be used to ensure sample integrity between collection in the field and analysis in the laboratory.

Field Sampling Forms

Standardized field sampling forms can help ensure that data are recorded that can be used to validate sample integrity. The field sampling form may be developed by the field personnel or by the analytical laboratory, as long as the form contains all the necessary information. The field sampling form should contain the following information at a minimum:

- Name, address, and contact number (telephone, Fax, E-mail) of the person collecting the samples
- Date the samples were collected
- Location where the samples were collected or project name/project number
- Sampling method used (e.g., NIOSH Method 7400) or a description of the sampling method
- Description of the sampling media used (type of media or manufacturer's identification number, manufacturer's name, lot number)
- Sample identification (a unique sample number assigned to each sample)
- Type of sample (air, bulk, personal, area, time weighted average, STEL, etc.)
- Sample location (name of employee sampled, identification of sample area, process name, machine identification, etc.)
- Identification of sampling device (pump number, etc.)
- Sample collection data (start time, stop time, flow rate, total volume collected, etc.)
- Analyses requested (some methods include analyses of multiple analytes)
- Date analytical results are requested
- Interferences that may be present in the sample environment
- Pertinent environmental conditions (relative humidity, temperature, etc.)

- Date samples were shipped
- Method of shipment
- Signature of sampler

Sample transport/shipping

Even samples collected with great care and adherence to established collection procedures can be ruined through improper shipping techniques. Samples can be destroyed and/or analyte(s) can be lost during shipping. The following shipping procedures should be considered:

- Always include a chain-of-custody/analytical request form with the shipment.
- Ship bulk samples in a separate package from air samples to avoid contamination of the air samples.
- Ensure that shipping containers are well padded to prevent breakage during shipping.
- Ensure that packing materials will not affect sample integrity (e.g., packing noodles or other static producing packing should not be used when shipping air samples for PCM analysis).
- Reference sampling methods to obtain special shipping requirements such as temperature requirements and sample stability. Some samples may require a temperature-controlled container such as a cooler or overnight delivery to the laboratory.

5.8 Handling of test items (samples)

Procedures used in every step between sample collection and report generation are important for ensuring the reliability of the data. This section of the chapter discusses procedures to be used by field personnel to submit samples to a laboratory and the procedures used by the laboratory receiving, handling and disposing of samples.

Section 5.8.1 of the standards requires that laboratories have procedures for the:

- transportation,
- receipt,
- handling,
- protection,
- storage,
- retention, and/or
- disposal of samples

The standard further specifies that these procedures include all provisions necessary to protect the integrity of the samples and to protect the interests of the laboratory and the customer. Some of these elements have been described previously under the sampling and field collection sections of this chapter. These and other required elements are typically included within the laboratory sample

handing and receipt standard operating procedure(s). The following sections address elements of these procedures that should be considered.

Chain-Of-Custody/Analytical Request Forms

Chain-of-custody forms are used to document the persons or groups that have handled samples from collection until the time of receipt by the laboratory, or in cases of legal chain-of-custody, track all handling and processing until analytical results are submitted to the client. This documentation can be used as evidence of the integrity of the samples during the process. The chain-of-custody is often part of the analytical request form provided by laboratories, or may be prepared as a separate document. While chain-of-custody forms vary from company to company, they should all contain some basic information. Appendix 11-2 contains an example chain-of-custody/analytical request form. Every chain-of-custody form should contain the following information at a minimum (some of the information is already present and does not need to be re-entered when the chain-of-custody is integrated with the analytical request form).

- Identification of the sampler (name, company name, telephone number)
- Identification of the sampling project (project number, project location)
- Identification of samples controlled by the chain-of-custody (sample id #, sample location description)
- Total number of samples
- Requested test(s) for each sample
- Date results are needed
- Area for signature of relinquishing party and date of signature
- Area for signature of receiving party and date of signature

The person collecting the samples should complete the chain-of-custody form/analytical request form in the field. The chain-of-custody form should be signed every time the samples change possession, including upon receipt by the laboratory. Under most circumstances, samples are considered under chain-of-custody after receipt at a secure laboratory facility. Laboratory preparation, analysis and review records are then typically sufficient to establish the chain of events and document the control of the samples and data while in the laboratory. When full legal chain-of-custody is required, a sample custodian may be assigned by the laboratory to maintain, monitor and record control of the samples and sample extracts/digestates. Under these circumstances, each analyst or technician must sign for custody of the sample or extract/digestate during each step of preparation and analysis.

Questions may arise concerning chain-of-custody during shipping when samples are not hand-delivered to the laboratory. Typically, the shipping company will not sign a chain-of-custody form, and the form is usually enclosed inside the shipping container. Historically, courts have held that chain-of-custody can be maintained if the individual delivering the package for shipping has signed the chain-of-custody,

the person receiving the package has signed the chain-of-custody form, the shipping form shows the delivery date, and the receiving party documents the condition of the package upon receipt. It is good practice to deliver the shipping container directly to the shipper and not to leave the package outside a drop-box, which could cause the integrity of the samples to be questioned.

Sample Seals and Labels

The integrity of the samples can be further protected when the sampler seals the samples immediately after collection. The seal must be placed on the sampling media in a way that any tampering with the samples would be apparent by visually inspecting the seal. Simply placing the end caps on a filter or the plastic caps on the ends of a sorbent tube will not suffice as a sample seal since they can be removed and replaced without detection. Typically, sample seals are long ribbon-like pieces of paper with an adhesive. The configuration allows the seals to be wrapped end-to-end around a sorbent tube or filter cassette and top-to-bottom around impingers. A well-designed seal will allow the sample to be labeled as to the sample number and date of collection and have a signature line. On many seals the signature line runs across the area where the two ends meet so that removal of the seal can be detected by the disruption of the signature. The seal should be wrapped around the sample tightly enough that it cannot be removed without breaking the seal. Some commercially available seals are designed as "shrink wrap" labels that dry after application, shrinking to fit tightly around the filter cassette. Figure 11.1 is an example of a sample seal (OSHA sample seal).

Figure 11.1

The use of sample seals is routine for OSHA compliance officers, but is rarely employed for routine sampling. Sample seals are typically only required in circumstances where evidentiary chain of custody has been requested.

All samples should be clearly labeled in the field immediately after sampling. The label should be legible, sturdy (so that it will not fall off during handling), and not contain adhesives that may contaminate the sample. Examples of information that may be included on the label include:

- Unique sample identification number (must match the sample identification number on the field sampling form and chain-of-custody form);
- Sample collection date; and
- Sampler's name.

Section 5.8.4 of the standard further requires that where a sample or a portion of a sample is to be held secure, the laboratory shall have arrangements for storage and security that protect the condition and integrity of the samples. AIHA-LAP, LLC Policy 2A.5.8.1 further requires that the laboratory shall have a written description of the chain-of-custody and sample receiving procedures followed in the laboratory. AIHA-LAP, LLC does not require full evidentiary (legal) chain-of-custody procedures unless required by the customer and agreed to by the laboratory. Samples are considered to be under the control and custody of the laboratory following receipt. AIHA-LAP, LLC does not require the use of sample seals unless requested by the customer and agreed to by the laboratory.

Sample Receiving Procedures

The procedures used to receive the samples at the laboratory are equally important in ensuring the integrity of the samples. Laboratories must develop standardized sample receipt and documentation procedures and train all personnel who will be receiving samples in their proper use. Section 5.8.3 of the standard requires that in the event that any abnormalities are noted upon receipt of samples, or if the requested test is not clearly indicated, that the customer is contacted for further instructions and the discussion is recorded.

AIHA-LAP, LLC Policy 2A.5.8.1 further requires that laboratory procedures include criteria for the rejection of samples. Laboratories should consider the following acceptance and rejection criteria as part of their sample receiving SOP and during the review of sample submissions and accompanying documentation:

- condition of the package, samples and the integrity of any sample seals, (if provided);
- manner of sample preservation, (if required);
- presence of chain-of-custody/analytical request form; and
- date of shipment and date of receipt at the laboratory.

The following should be verified through a review of the field sampling form/chain-of-custody or analytical request/chain-of-custody form and inspection of samples:

- date of sampling;
- sample collection method(s), (as applicable);
- type of analyses requested;
- total number of samples;
- appropriateness of flow rates and total volumes for air samples;
- appropriateness of collected mass for bulk samples;
- appropriateness of sample media for analyte(s) requested;
- whether sample identification numbers were assigned;
- whether samples were labeled with individual identification numbers;

- whether the identification numbers on the field sampling form/chain-of-custody match the identification numbers on the samples;
- whether all documentation and sample labeling are legible;
- length of time between sample collection and sample receipt;
- whether the sampling form has been signed by the sampler; and
- whether air samples were shipped with bulk materials.

In the event that any abnormalities noted above are observed upon the receipt of samples, the customer must be contacted and the results of the discussion must be recorded. Many laboratories require written confirmation of any additional customer information or requested changes in the form of a revised analytical request/chain-of-custody form or an email request.

Sample log-in/Accessioning

Section 5.8.2 of the standard states that the laboratory shall have a system for identifying samples and that this identification is retained throughout the life of the sample in the laboratory and is sufficient so samples and resulting laboratory records cannot be confused. The standardized sample receipt and log-in procedures may be reflected in a sample receipt form or may be a part of a computer-based sample log-in system. When hand-written receipt forms are used, completed forms should be bound into a log book with sequentially numbered pages. Computer log-in systems must include backup systems in accordance with procedures for protection of electronic records (Section 4.13).

The person receiving the samples should perform the following steps prior to placing the samples in storage:

- enter each sample into the laboratory's log-in system;
- assign and record a unique laboratory identification number to each sample;
- note any discrepancies on the sample receipt log;
- ensure preservation chemicals have been added to samples (as required);
- sign and date the chain-of-custody form, as applicable (noting any discrepancies on the form); and
- sign and date the sample receipt log.

Sample Storage and Disposal

Section 5.8.4 of the standard requires procedures and appropriate facilities for avoiding deterioration, loss or damage to the samples during storage, handling and preparation. When items have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded. This requirement is most applicable to specific sorbents and/or samples that may require refrigeration or storage at freezer temperatures prior to analysis. In these cases,

refrigerator and freezer temperatures must be maintained, monitored and recorded (See Chapter 10, Section 5.5).

All incoming samples should be received and entered into the laboratory's log-in system as soon as possible. Care must be taken when unpacking samples from the package to prevent damage. Some compounds are light- or temperature-sensitive. These samples should not be left out in the general laboratory environment for an extended period of time. Compounds requiring storage in a refrigerator or freezer should be placed in the proper storage area as soon as possible.

Storage considerations are also extremely important in maintaining sample integrity. Air samples should not be stored in the same package, cabinet, refrigerator or freezer as bulk samples or bulk containers of volatile liquids. Samples should be stored in a manner that prevents breakage during storage. Samples with limited storage life should be prioritized for analysis.

It is suggested that laboratories consider retaining any remaining amount of bulk samples, sample containers and sample extracts until some specified time following the receipt of results by the customer. This permits the laboratory to investigate potential misidentification of samples or extracts/digests in the event that the customer has questions regarding the results. The utility of retesting retained samples is dependent upon the nature and stability of the residual sample or sample extract/digestate or a specific customer sample retention requirement. A retention of 14 days to 30 days is typical for many laboratories.

Laboratories must also have appropriate procedures and arrangement for the disposal of hazardous wastes generated by laboratory operations. The laboratory is often required to sign a certification to this effect upon application for accreditation by accrediting authorities such as AIHA-LAP, LLC.

Chapter 11 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 172.

1. Which of the following is not normally a purpose of the field sampling form?

a. Documenting the sampling method
b. Assigning laboratory QC samples
c. Documenting who collected the samples
d. Stating which analysis is requested

2. At a minimum, who should sign the chain-of-custody/analytical request form? (Indicate all that may apply)

a. The person receiving samples for the laboratory
b. The person submitting the samples
c. All shipping personnel
d. Everyone who has control of the samples at some time

3. What is the primary purpose of sample seals?

a. Document sample flow rates
b. Document chain-of-custody
c. Indicate sample tampering
d. Protect the samples from breakage

4. What items should the person receiving a set of samples check to verify sample integrity? (Indicate all that apply)

a. Sample seals, as applicable
b. Condition of shipping container
c. Sampling method used
d. Presence of chain-of-custody

5. When should the samples be entered into the laboratory's log-in system?

a. After receipt and prior to placing the samples in storage
b. Any time prior to analysis
c. Any time prior to release of the final report
d. The samples do not have to be logged in

6. What should the sample receipt personnel do if a damaged sample package is delivered to the laboratory? (Indicate all that may apply)

a. No action is required
b. Record the damage on the sample receipt log
c. Determine if the samples are damaged
d. Note the damage and contact the customer for instructions

7. What actions should the sample receipt personnel take if the chain-of-custody/analytical request form is not included with a set of samples? (Indicate all that apply)

a. Create a chain-of-custody/analytical request form
b. Note the discrepancy on the sample receipt log
c. No action is required
d. Contact the customer before processing the samples any further and record the results of communication

8. Which of the following aspects might lead to a sample being rejected prior to analysis?

a. The sample was collected on silica gel sorbent when the method called for activated charcoal sorbent
b. The sample volume was four times the recommended maximum in the published method
c. The sample volume was 10% of the recommended minimum in the published method
d. All of the above

9. What storage rules should be used for air and bulk samples? Why?

10. Which of the following factors might affect sample retention times?

a. The type of analysis
b. Sample stability
c. Customer request
d. All of the above

11. If a laboratory is not responsible for the collection of field samples, do any of the requirements of ISO/IEC Section 5.7 apply?

Chapter 12

Assuring the quality of test results (5.9), Data reduction and validation, and Reporting the test results (5.10)

Learning Objectives

After completing this chapter, the student should be able to:

- Explain how each of the types of QC samples is used to check the accuracy and precision of data generated by the laboratory
- Explain how to generate or utilize QC samples
- Develop statistical quality control limits for accuracy and precision
- Identify statistical trends in QC data
- Identify some common proficiency testing programs for laboratories
- Explain how proficiency testing programs operate and how data generated by the programs can be used by the laboratory
- Identify the information that should be reviewed in order to correctly evaluate the quality of the data
- Understand how to resolve any discrepancies discovered during the informational review process
- Identify the basic contents that should be included on a final report
- Summarize the contents of a final report
- Present data on a final report in a clear, concise manner
- Understand the basic reporting units used for different types of analytical data

Introduction

AIHA-LAP, LLC Module 2A and program specific Modules 2B-2F, as appropriate, include additional accreditation requirements. The student should read Sections 5.9 and 5.10 of ISO/IEC 17025 and Sections 2A.5.9 and 2A.5.10 of AIHA-LAP, LLC Module 2A in conjunction with reviewing the sections presented in this chapter.

5.9 Assuring the quality of test results

This section is one of the shortest sections of ISO/IEC 17025. Despite its brevity, this section outlines what are most commonly referred to as the quality control requirements of the standard. Two

paragraphs contain all requirements. The following sections discuss these and additional AIHA-LAP, LLC policy requirements.

Section 5.9.1 of the standard states, "The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results." This section therefore mandates quality control procedures, the application of statistical techniques in reviewing data, and recording data in such a way that trends are detectable. AIHA-LAP, LLC Policy 2A.5.9.1 requires laboratories to determine, where feasible, the accuracy and precision of all analyses performed. AIHA-LAP, LLC Policy 2A.5.9.1.2 requires laboratories to establish acceptance limits for each method based on statistical evaluation of the data generated by the analysis of quality control check samples, unless specific acceptance limits are established by the method. The calculation procedures for statistically derived acceptance limits must be documented. It is suggested that method specified acceptance limits are only be used initially and are replaced when laboratories have generate sufficient data to establish their own statistical limits.

Recording data in such a way that trends are detectable essentially requires the use of control charts, or a sophisticated database that can detect statistically significant trends. AIHA-LAP, LLC Policy 2A.5.9.1.3 further requires that laboratories have documented procedures that are used to monitor trends and the validity of test results. This requires the definition of and monitoring of statistically significant trends in laboratory data. There are many interpretations regarding statistically significant trends. The following are generally accepted:

- One result outside the upper or lower control limit
- Two consecutive results outside either the upper or lower warning limit
- Four to five consecutive results either steadily increasing or decreasing in magnitude
- Seven consecutive results on one side of the mean value

When a trend is observed, the laboratory must implement the nonconforming work process and investigate the cause of the trend. Data falling within control limits may still be reportable, but the trend indicates a change in the statistical process control. Appropriate corrective action may include recalculation of statistical limits for the test method since something within the laboratory process may have changed.

The use of statistical techniques is described in Chapter 7. Generally a minimum of thirty (30) data points should be used to establish statistical limits. It is recommended practice to utilize initial points to establish statistical control limits and then to evaluate and chart subsequent quality control data for comparison with these limits. Some laboratories use a moving average where new limits are calculated and applied with each set of quality control data. While this method provides up-to-date statistical limits, it can obscure trends. It is suggested that laboratories recalculate statistical limits when a sufficient number of additional data points are accumulated and following any significant changes in

procedure. Consequently, high throughput test methods should be recalculated much more frequently than occasional tests. Laboratories may utilize LIMS based statistical quality control applications, third party statistical analysis programs, or control charts and limits generated using database or spreadsheet applications such as Microsoft Access and Microsoft Excel. AIHA-LAP, LLC expects laboratories to generate statistical limits for both accuracy (bias) utilizing the recovery of matrix-based certified reference materials or media spiked samples and for precision using data from either replicate certified reference materials, replicate media spiked samples, or replicate customer samples. In all cases, these samples must be processed through the entire analytical procedure.

The laboratory can create these QC samples by spiking media or samples with known concentrations of an analyte. Spikes of clean media are commonly referred to as method spikes, whereas spikes of customer samples are commonly called matrix spikes. Several types of spiked samples may be created in the laboratory. AIHA-LAP, LLC Policies 2A.5.9.1.1 and 2B.3.8 require the preparation of media blanks, replicate samples (or replicate spikes) and media based spikes with each batch of analytical samples. This requirement applies to all types or media including filters, sorbent tubes, diffusive samplers, and whole air samples. Manufacturers of diffusive samplers have been known to supply sorbent sections of their samplers to laboratories at a reduced cost when designated for this purpose.

Liquid or Solid Spiked Samples

In larger laboratories, the quality manager or a designee may spike the same sample media as submitted by the customer with known concentrations of the analyte(s) and submit the sample(s) as part of the customer's sample set. Neat solutions of analytes or dilutions of neat solutions are spiked directly onto the front section of sorbent tubes or deposited onto a filter. Sorbent tubes should be allowed to sit at room temperature for at least four hours or stored overnight before desorption. Filter samples should be allowed to dry thoroughly before analysis. QC sample submittal is most effective when the analyst does not know which samples are from the client and which are QC samples. Although the routine submission of samples on a blind basis is desirable, it is not required. The preparation analyst is typically responsible for preparing spikes. Figure 12.1 shows an example of liquid spiking of a sorbent tube to create a media spiked (method spike) QC sample.

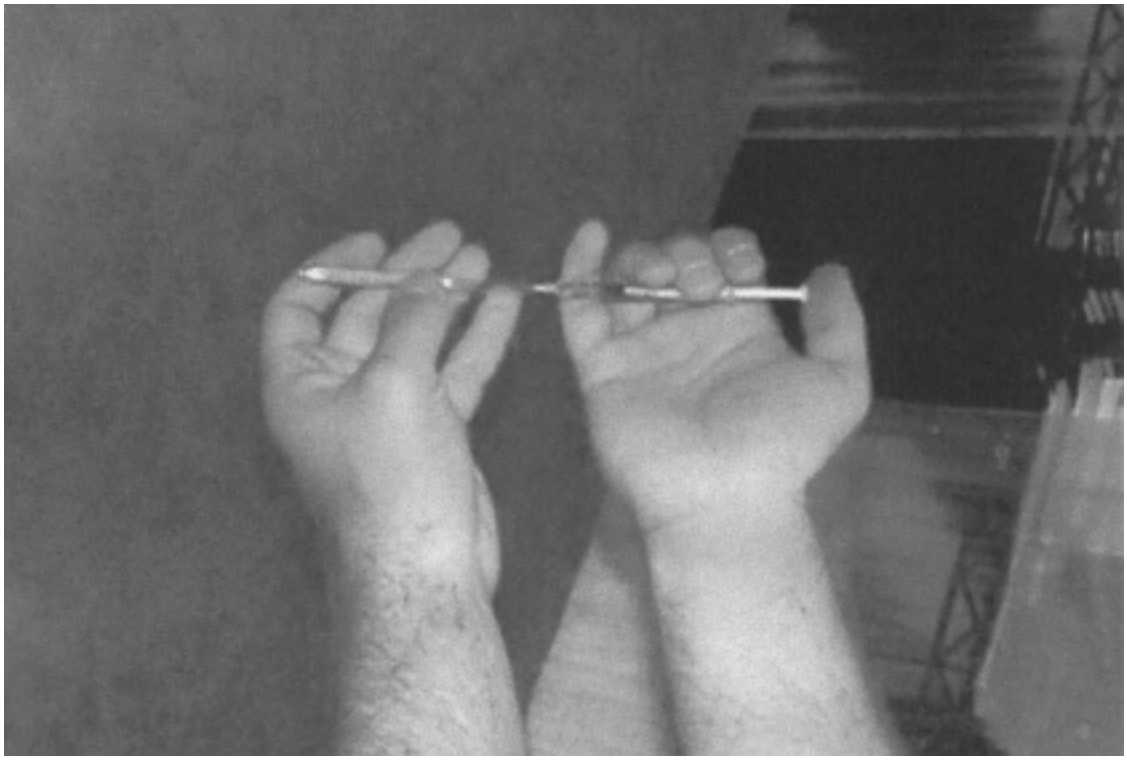

Figure 12.1 - Liquid Spiking for QC Samples

Vapor Spiked Samples

Vapor spiking can be an effective method to test not only the accuracy of an analysis, but also the collection and desorption efficiencies of a validated method. Vapor spiking requires the set up of some specialized equipment. It can be performed by creating special devices where a concentration of liquid is added, vaporized or flashed, and collected on a tube by drawing the air through the tube or drawing air onto a badge by allowing the badge to set inside the spiking chamber. One of the simplest systems is a stainless steel T with compression fittings. A "scrubbing" sorbent tube is attached to one end of the T and the tube to be spiked is attached to the opposite end of the T. The top of the T is fitted with a compression fitting containing an injection septum. A sampling pump is attached to the spike sorbent tube and air is drawn through the "scrubbing" sorbent tube to remove any of the analyte in the laboratory air, through the T, and into the spike tube. A liquid or gas can be injected through the septum into the T and drawn through the spiking tube. The stainless steel T can be wrapped with heating tape to assist in vaporizing the analyte. Alternately, some laboratories use a gas chromatograph to perform vapor spiking. The sample tube is connected to the injection port outlet of the GC with airflow through the tube. The liquid is injected into the heated injection port resulting in the liquid "flashing" onto the tube. The sample is submitted for analysis and the results are compared to predicted concentrations.

Field Spiked Samples

Although not required by the standard or AIHA-LAP, LLC policies, spiked samples can also be prepared by the laboratory or the client before collection of the field samples. As discussed in Chapter 8, this is a desirable part of the method development and validation. During this process, the identical sample media is spiked with a known concentration of the analyte(s) of interest prior to the start of air sampling. An air sample is then collected through the spiked sample side-by-side with a real sample. The two samples are submitted to the laboratory for analyses. When the results of the two samples are compared the spiked sample result(s) should equal the real sample results plus the spiked concentration(s). This comparison which will require a cooperative effort between the field investigator and the laboratory, can be used to test the accuracy of the laboratory results by comparing the difference in the two concentrations.

Accuracy (bias) is typically evaluated using the mean recovery with warning limits at plus and minus two (2) standard deviations from the mean recovery and control limits at plus and minus three (3) standard deviations from the mean recovery. The mean recovery represents the bias of the laboratory. In the case of sorbent methods, this bias also represents the desorption efficiency. Some laboratories use the ongoing mean recovery of media spiked samples for desorption efficiency correction.

Precision is generally calculated and evaluated as the range or relative percent difference between replicate (most often duplicate) samples. Range is most applicable if the magnitude of samples is constant (e.g., replicate spiked media always spike at the same concentration) while relative percent difference normalizes range data by taking both the range and concentration of the replicates into account. In industrial hygiene laboratories, precision is most commonly evaluated through duplicate media spiked samples, but precision can also be evaluated through the analysis of different aliquots of customer supplied bulk samples.

Examples of both accuracy (Table/Figure 1) and precision (Tables/Figures 2 through 5) control limits and control charts are included in Attachment 12-1. The Excel spreadsheet used to generate these limits and charts is included as Appendix 12-1 for the student's convenience. An AIHA-LAP, LLC guidance document is included as Appendix 12-2 and provides examples of several quality control techniques applicable to environmental microbiological methods as well as application of the techniques described in Attachment 12-1.

Section 5.9.1 further states the following requirements for quality control monitoring: "This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a. regular use of certified reference materials and/or internal quality control using secondary reference materials;
b. participation in interlaboratory comparison or proficiency-testing programs;
c. replicate tests or calibrations using the same or different methods;
d. retesting or recalibration of retained items;
e. correlation of results for different characteristics of an item."

AIHA-LAP, LLC policies expand on these general requirements and include additionally prescriptive requirements. These requirements are addressed in the sections that follow.

5.9.1.a) "Regular use of certified reference materials and/or internal quality control using secondary reference materials"

Certified reference materials (such as prior round IHPAT samples or NIST SRMs) or laboratory-generated laboratory control samples (media spikes, laboratory control samples) are required with each analytical (preparation) batch of samples by AIHA-LAP, LLC Section 2B.3.8 to monitor and evaluate accuracy (bias). Since most industrial hygiene samples are totally consumed (completely digested or extracted during the analytical process), true matrix spiked samples cannot be subject to the complete analytical procedure, so media spiked samples are accepted as a surrogate.

5.9.1.b) Participation in interlaboratory comparison or proficiency-testing programmes

AIHA-LAP, LLC requires participation in specified proficiency testing (PT) programs as indicated in Module 6. Proficiency testing programs are designed to test continuing laboratory proficiency for accreditation programs. A set of "unknowns" is sent to each participating laboratory. The unknowns may contain one analyte of interest (as with a filter) to mount and count for fiber concentrations, or there may be more than one analyte (as with samples for metals and organics). Each laboratory analyzes the samples and returns the results within a specified time period. All laboratories' results are statistically analyzed and acceptance limits are established. Typically, the mean and standard deviation for all reported results are computed (after removing outliers), and warning and acceptance limits are set at the mean plus or minus two and three standard deviations, respectively. Each laboratory is given a rating of acceptable or unacceptable on their reported results. Requirements are established for acceptable performance for each round and the number of unacceptable rounds a laboratory can receive before being rated as non-proficient. In general, laboratories must demonstrate acceptable performance in two of the three most recent consecutive rounds to maintain proficient performance.

Laboratories are required to analyze PT samples in the same manner as customer samples. This means that AIHA-LAP, LLC expects the following practices when analyzing and reporting PT samples:

- PT samples are rotated among current analysts
- A single analyst's results are reported
- Averages or multiple analyses may not be reported unless this is the laboratory's procedure for customer samples
- Additional QC samples are not included with PT samples

If an approved proficiency testing program is not available, laboratories may participate in a round robin interlaboratory exchange or demonstrate proficiency through statistical quality control and analysis of internally generated blind proficiency sample submissions once every six months. These specific requirements are detailed in AIHA-LAP, LLC Policy Module 6. If the laboratory chooses the round

robin or internal demonstration of proficiency methods, the AIHA-LAP, LLC policy requirements must be carefully reviewed and laboratory procedures related to the programs must be well documented.

5.9.1.c) Replicate tests or calibrations using the same or different methods

AIHA-LAP, LLC section 2B.3.8 mandates the analysis use of duplicate portions of client samples where subsampling is performed and where positive test results are expected or replicate laboratory control samples (replicate tests) to monitor and evaluate precision. Once again, since most industrial hygiene samples are totally consumed (completely digested or extracted during the analytical process) true duplicate samples cannot be subject to the complete analytical procedure, so replicate media spiked samples are accepted as a surrogate to monitor precision. Duplicate samples can be analyzed in circumstances where sufficient bulk material is provided.

5.9.1.d) Retesting or recalibration of retained items

Since few IH samples (with the exception of bulk samples) are retained following analysis, this requirement is generally not applicable, although the retesting of residual bulk samples (described in section 5.9.1.c, above) is essentially the same process.

5.9.1.e) Correlation of results for different characteristics of an item

Samples submitted to industrial hygiene laboratories are generally analyte or analyte group specific, so correlation of results for different characteristics of a sample is frequently not applicable. Where this may apply is the correlation of total or respirable dust measurements with subsequent specific analyte or analyte group testing such as total dust and silica, or total welding fumes and individual metals.

The AIHA-LAP, LLC policies additionally address blanks. Policy 2A.5.9.1.1 requires the use of media blanks (blank sampling media and analytical reagents) with each batch of samples and encourages customers to supply specimens of blank sampling media from the same source lot used for sample collection.

Typically, two types of blanks can be analyzed along with customer samples: field blanks and laboratory blanks. Field blanks are media that are treated the same as the sample collected for analysis except that they are not exposed to the sample environment. For example, when charcoal sorbent tubes are used to collect organic vapors in a work area, a number of charcoal tubes should have both tube ends opened and then the ends will immediately be capped to prevent exposure to the work environment. These blanks should accompany the samples throughout the entire sample collection, shipment and analysis process. Field blanks are used to detect contamination in the sample media from the manufacturer and any contamination introduced during collection and shipping (such as can occur when shipping samples in the same package as bulk samples). Many field investigators submit field blanks with samples. Some identify blanks but others label them as regular samples so the laboratory will not know which samples are blanks. The field blank concentrations should not be automatically subtracted from the sample results.

Laboratory blanks, on the other hand, are prepared within the laboratory from unused media. A laboratory blank accompanies the samples through the analytical process and is designed to detect contamination from the manufacturer, contamination from laboratory chemicals used in the analysis, and contamination that is introduced in the laboratory (such as can occur when samples are stored with bulk reagents). The laboratory should develop procedures for addressing high laboratory blank concentrations. Laboratory blank concentrations may be discussed with the customer and the analytical results rejected, or the blank concentrations may be subtracted from the sample results. Ideally both field and laboratory blanks should be used and analyzed with each analytical batch of samples.

Section 5.9.2 of the standard states, "Quality control data shall be analysed and, where they are found to be outside pre-defined criteria, planned action shall be taken to correct the problem and to prevent incorrect results from being reported." Laboratories must therefore define acceptance criteria and include planned actions to be taken in the event that quality control data are outside these pre-defined criteria. These planned actions are typically included in test method SOPs and may include such actions as:

- Continuing calibration verification (CCV) standard outside of defined acceptance limits—check and correct problem, recalibrate and reanalyze all sample extracts/digestates since the last valid CCV;
- Continuing calibration blank (CCB) outside of defined acceptance limits— check and correct problem, re-zero instrument (as applicable), check CCB and CCV, reanalyze all sample extracts/digestates since the last valid CCB; and
- Laboratory control sample/laboratory control sample duplicate outside of defined acceptance limits—check instrument calibration. Correct if necessary, recalibrate and reanalyze all sample batch QC and any extracts/digestates analyzed with that preparation batch. If still unacceptable, include comment on analytical report.

The AIHA-LAP, LLC general policy requirements for routine quality control are summarized below. The AIHA-LAP, LLC Modules 2B through 2F include additional quality control requirements specific to those AIHA-LAP, LLC accreditation programs and should be reviewed dependent upon program application.

- Evaluate contamination, bias, and precision with each batch of samples utilizing media blanks, spikes, replicates, and/or CRMs.
- Carry media spikes/duplicates through all preparation steps to evaluate and monitor bias and precision.
- Develop statistical control limits for both accuracy and precision using media spikes and media spike duplicate or duplicate samples.
- Monitor quality control data for statistical trends.

Data reduction and validation

All laboratory data must be reduced and validated prior to release to the customer. Data validation is required to ensure that analyses were conducted properly and that quality control data are within established limits. Data reduction and validation should be performed as soon as possible after the data are generated so that corrective actions can be implemented in a timely fashion. This is especially important with samples that have limited storage life. The first level of data reduction and validation should be conducted by the analyst and include the following:

- Did the analyst document the analytical procedure properly? Do the laboratory notes include all analytical parameters? Are the analytical parameters appropriate for the method? Are the analyst and date of preparation and analysis documented in laboratory records?
- Did the analyst use the appropriate reagents and reference materials during the analysis? Had the reagents and reference materials passed their expiration date?
- Were measurement instruments and other laboratory equipment properly calibrated? Did the analysis include verification of continued calibration throughout the analytical procedure?
- Were the calculations performed correctly? The analyst should calculate representative results from the raw data and ensure the data are accurate. The laboratory records should include enough information to allow another reviewer to calculate the results.
- Was analyte breakthrough, as applicable, observed in any samples? If so, are those samples appropriately noted?
- Were appropriate quality control samples included with the preparation and analytical batch? Were the QC sample results within laboratory control limits?
- Are all electronic files and analytical printouts such as chromatograms included with the laboratory records? Are the electronic files or printouts suitably marked to identify the batch of samples analyzed?
- In some instances, analysts either enter data into a LIMS or reporting format. Some laboratories have implemented direct data uploading to LIMS from instrument data files. In all cases, were the data transcribed or transferred accurately from the raw data to the LIMS or final report?

AIHA-LAP, LLC Policies 2A.5.4.5 and 2A.5.4.6 include specific data review requirements. As noted in prior chapters, this must be an independent review conducted by a qualified individual other than the analyst and completed and documented prior to the reporting of results to the customer. If this cannot be completed prior to reporting, results must be clearly indicated as preliminary. The data reduction and review process must include, but is not limited to:

- comparison of quality control data against established acceptance limits
- computation verification;
- transcription of data verification;
- adherence to the procedures established in the laboratory management system documents; and
- review of correlation of results if more than one parameter in a sample is tested.

A checklist, notation on data sheets or LIMS are typically used to document this review.

Any discrepancies discovered during the review process should be discussed and resolved with the analyst. Corrective actions should be taken to correct erroneous data and to prevent a recurrence during future analyses. All discrepancies and corrective actions should be documented and resolved prior to reporting to the customer.

5.10 Reporting the test results

The requirements and content of reports are well described within this section of the standard. Section 5.10.2 includes required content. As discussed, in Section 5.10.1, simplified reports may be provided for internal customers or those customers with specific written agreement. In such cases, all other information normally included in the report must be maintained, and made available, as necessary.

Several comments are provided regarding some of the report content requirements:

5.10.2.e) Identification of the method used

This is typically the referenced method or can be the laboratory SOP number reference. Laboratories should indicate referenced methods as modified, if modifications to the desorption, digestion, chemistry or analysis/detection method are made.

5.10.2.f) A description of, the condition of, and unambiguous identification of the item(s) tested

This element is often overlooked by laboratories. A statement to the effect that all samples were received in acceptable condition, unless otherwise indicated, is acceptable.

5.10.2.h) Reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

This element is not required unless the laboratory was responsible for field sample collection.

5.10.2.i) The test or calibration results with, where appropriate, the units of measurement;

Review of the final report should not only include review of final test results, but also review of the units of measurement. There are a number of appropriate units for reporting various analytes, and the laboratory should carefully review the customer request for any desired reporting units. Customers often request that only total mass of analyte per sample is reported, while others may request the reporting of air concentrations based upon the sample volume information provided by the customer. Examples of typical reporting units for various types of analytes and media are included in Table 12-1.

If airborne concentrations are reported, the laboratory is cautioned to clearly qualify such results as being based upon information provided by the customer.

Table 12-1 - Typical Reporting Units

Analyte	Medium	Units
Gases and Vapors	Air (sorbents)	mg/m^3, ppm, $\mu g/m^3$, ppb, µg/sample, mg/sample
Particulates	Air (filters)	mg/m^3, $\mu g/m^3$, µg/sample, mg/sample
Particulates	Dust (filter or wipe)	$\mu g/ft^2$, µg/100 cm^2 , $\mu g/cm^2$, µg/wipe
Environmental	Solid Medium (Bulk Sample)	mg/kg, µg/kg, ppm, ppb, %
Environmental	Liquid Medium (Bulk Sample)	mg/L, µg/L, ppm, ppb
Lead	Paint chip	mg/cm^2, mg/kg, %, ppm
Asbestos, Fibers	Air (filter)	f/cc, f/mm^2,str/cc, str/mm^2
Asbestos, Fibers	Dust (filter)	f/ft^2, str/ft^2, f/cm^2, str/cm^2
Asbestos	Bulk Insulation Sample	%
Microbials (culturable)	Air (agar)	cfu/m^3
Microbials (culturable)	Solid Medium (Bulk Sample)	cfu/g, cfu/kg
Microbials (culturable)	Liquid Medium (Bulk Sample)	cfu/g, cfu/kg, cfu/L, cfu/mL
Microbials (fungal direct examination)	Air (inertial impactor/spore trap sample)	Str/m^3, spores/ m^3
Microbials (fungal direct examination)	Surface (Tape lift)	str/cm^2, spores/ cm^2

ppm = parts per million
ppb = parts per billion
mg/m^3 = milligrams of analyte per cubic meter of air
$\mu g/m^3$ = micrograms of analyte per cubic meter of air
mg/kg = milligrams of analyte per kilogram of sample
µg/kg = micrograms of analyte per kilogram of sample
f/cc = fibers per cubic centimeter of air
str/cc = structures per cubic centimeter of air
str/ m^3 = structures per cubic meter of air
f/cm^2 = fibers per square centimeter of area
str/cm^2 = structures per square centimeter of area
mg/L = milligrams of analyte per liter of solution
$\mu g/ft^2$ = micrograms per square foot of area
cfu/m^3 = colony forming units per cubic meter of air

5.10.2.j) The name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report or calibration certificate

Printed name(s) and function(s) are sometimes overlooked by laboratories. Remember that the individuals reviewing and signing reports must be authorized for report review and signature per section 5.2.5 of the standard.

5.10.2.k) Where relevant, a statement to the effect that the results relate only to the items tested

This is always considered relevant by AIHA-LAP, LLC since laboratories only analyze a sample provided by the customer or possibly collected by the laboratory. This is really a safeguard for the laboratory as the laboratory cannot reasonably extrapolate any results beyond those for the sample itself. Accordingly, this statement is required on all AIHA-LAP, LLC accredited reports.

Section 5.10.3.1 includes other elements when relevant. These are not typically required for industrial hygiene laboratory analyses. The following specific comments are provided:

5.10.3.1.a) Deviations from, additions to, or exclusions from the test method and information on specific test conditions, such as environmental conditions

Inclusion of the referenced test method and inclusion of "modified" is considered sufficient. This section would only be utilized when a laboratory significantly deviates from its internal test method SOP.

5.10.3.1.b) Where relevant, a statement of compliance/non-compliance with requirements and/or specifications

Some laboratories calculate TWA concentrations for customers and provide a comparison with OSHA limits or recommended TLVs. Laboratories are cautioned to include disclaimers regarding such calculations and comparisons since they are relying on customer supplied information to make these comparisons. Observation of work practices, the representativeness of sampling, work shift variations and consideration of potential exposures during un-sampled times involve decisions and interpretation that require observation and understanding of the operation being evaluated. This is the typically the responsibility of on-site sampling personnel.

5.10.3.1.c) Where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit

At this time, AIHA-LAP, LLC does not require inclusion of a statement on the estimated uncertainty of measurement unless specifically requested by the customer. The one exception is for fiber count data by NIOSH Method 7400. Intra-laboratory Sr values must be included on the report as required by the method and AIHA-LAP, LLC Policy 2B.4.1.2.

5.10.3.1.d) Where appropriate and needed, opinions and interpretations (see 5.10.5)

Since testing laboratories are only reporting the results of analysis, opinions and interpretations are not appropriate. Opinions and interpretations are more typically appropriate where results are interpreted, such as failure analysis where the results of physical tests on samples are used to predict potential failure of a system or product.

Additional AIHA-LAP, LLC reporting requirements (2A.5.10.1 through 2A.5.10.5) mandate the inclusion of such elements as:

- the date received,
- report page numbering,
- the reporting limit,
- method of reporting of non-detectable results,
- identification of tests not covered by the lab's accreditation,
- a statement regarding blank correction, and
- reporting appropriate number of significant figures.

Laboratories sometimes overlook the requirement to indicate whether or not blank correction has been applied, even when it has not been applied to the samples. Laboratories are also encouraged to indicate when no discernable blank is submitted.

The excessive use of significant figures is sometimes observed and overstates the precision associated with the analysis. The student should review Chapter 7 regarding significant figures. Quite often, some measurement or step of the analytical process is limited to two significant figures so, accordingly, the final result should not include any more than the least number of significant figures. The best determination of the number of significant figures is by estimating the uncertainty of measurement of the test method and reporting accordingly. It is generally recommended that one significant figure is reported at the reporting limit and two (but no more than three) significant figures are reported for quantified results significantly above the reporting limit.

Issuance of a report is one of the last steps performed by the laboratory. Reporting occurs after samples have been collected, shipped to and received by the laboratory, analyzed using appropriate methods, and the data have been validated. It is easy to overlook the generation of the report as important to data quality because the data has already been generated using good QA/QC procedures. However, errors may occur during report generation.

Final report review should also include information provided by the customer as well as any information generated during the analytical process. The review of information provided by the customer is just that: a review. The laboratory is not expected to conduct an investigation to ensure

that the information accurately reflects what the customer did in collecting the samples. In addition to the analyst reviewed elements and required report elements discussed above and in earlier sections of this chapter, a final report informational review should include the following areas:

- Chain-of-Custody. Did the customer provide a chain-of-custody with the samples? Was sample custody properly documented on the form? This information should have been reviewed and any problems discussed with the client upon receipt of samples.
- Collection Information. Was the method selected by the customer appropriate for the requested analyte(s)? Did the customer use the appropriate media for sample collection? If required by the method, were the samples preserved properly prior to shipment, including the addition of the proper preservatives? Was the sample volume adequate for the selected method? This information should also have been reviewed and any problems discussed with the client upon receipt of samples.
- Does the final report accurately reflect the final analytical results and include all required reporting information? Does the final report reference the analytical method, cross-reference the customer's sample identification numbers with the laboratory's identification numbers, and reference any modifications to the analytical method? Are any discrepancies discovered during receipt, analysis, data validation and report generation noted on the final report? Does the final report include a reference to the reporting limit for the method? Were the reporting limits calculated correctly for each sample?
- Will the final report be issued to the customer within the time specified on the field-sampling sheet or within laboratory guidelines?
- Do the results appear reasonable based upon the experience of the laboratory?

Section 5.10.3.2 of the standard includes additional reporting requirements applicable to sampling. These only apply when sampling is conducted by the laboratory.

Sections 5.10.4 (Calibrations) and 5.10.5 (Opinions and Interpretations) are not applicable to testing labs. The requirements of section 5.10.6 are technically only applicable to subcontracted test results that are within the laboratory's accredited fields of testing (FoT), although it is good practice for laboratories to always clearly indicate any subcontracted result. Identification of subcontracted results is also required under AIHA-LAP, LLC Policy 2A.5.10.2 whenever the laboratory report makes reference to the laboratory's accreditation and sub-contracted results are not within the laboratory's scope of accreditation.

Sections 5.10.7 through 5.10.9 of the standard include requirements for electronic transmission of results, the format of reports and amendment of reports. Electronic reporting is now commonplace via e-mail and attachments or customer ability to either review or download reports or data from laboratory websites. When laboratories use these techniques, care must be taken to include all required reporting information and to adequately maintain data integrity and confidentiality.

Reporting in portable document format (pdf) helps protect data integrity since these files are more difficult to alter. Confidentiality can be maintained by including confidentiality statements on all forms of results transmission and customer user identification and password protection for access to web based information.

Amended reports must be clearly indicated and reference the report that it is either amending or replacing. Records requirements of Section 4.13.2.1 of the standard are interpreted by AIHA-LAP, LLC to require that laboratories maintain a record of the report originally issued to the customer. Laboratories that rely on electronic records for these reports are cautioned to be sure that they maintain the original report as some LIMS systems only retain the final amended or replaced report.

Chapter 12 Competency Exercises

Complete these questions to test your understanding of the chapter. Check your answers in the Competency Exercise Answer Key, starting on page 173.

1. Which of the following methods would describe the preparation of a spiked sample?

a. Running two samples side-by-side in the field
b. Injecting a known concentration of an analyte into a sorbent tube
c. Breaking both ends of a sorbent tube and immediately recapping the ends
d. All of the above

2. The laboratory analyzes two blank samples provided by the client along with 18 regular samples. The blank samples are found to contain the analyte at concentrations that are approximately 25% of the regular samples. What should the laboratory do?

- ☐ a. Subtract the average results of the two blanks from the regular samples.
- ☐ b. Ignore the blank results.
- ☐ c. Report the results to the customer indicating the actual results of the blank samples with an indication of whether or not the results were corrected for the blank.
- ☐ d. Discard all the samples and report the results as unusable to the client.

3. Which of the following may specify the QC sample required for a particular analytical method?

a. The method itself
- ☐ b. Accrediting agencies
- ☐ c. Regulatory agencies
- ☐ d. Any of the above

4. Name two types of calibration verification standards. Define each.

1.
2.

5. Explain the difference between matrix spikes and media spikes.

6. A QC chart is established for recovery efficiency for benzene based on replicate analysis of spiked samples. The mean from the replicate analysis is 96% recovery and one standard deviation is 1.2%. Which of the following would be an appropriate lower control limit for the QC chart?

a. 75%
b. 92.4%
c. 99.6%
d. 90%

7. A QC sample is spiked with 22 µg of benzene. The analytical results show that the recovery was 20.5 µg. Using the QC chart in question 6, is the QC result within the control limits, warning limits, or outside both limits?

a. Inside both warning and control limits
b. Inside control limit, outside warning limit
c. Inside warning limit, outside control limit
d. Outside both warning and control limits

8. A laboratory control sample is prepared on six consecutive days by spiking with 22 µg of benzene. Analysis shows results of 20.6, 20.8, 20.9, 21.0, 20.6, and 20.9 µg.

a. Are these results within the warning limits for the QC chart used in Problem 6?

b. Are the results within the control limits?

c. Are there any QC issues with these data?

9. In the following table calculate the mean, standard deviation, control limits (±3 SD) and warning limits (±2 SD) for each set of data.

Data	Mean	SD	Control Limits	Warning Limits
1.0, 1.1, 1.4, 1.5, 1.4, 1.5, 1.2				
23, 23, 24, 25, 26, 26, 23, 24				
96, 96, 97, 98, 101, 100, 100				

10. Which of the following programs is administered by the American Industrial Hygiene Association Proficiency Analytical Testing Program, LLC? (Indicate all that apply)

a. EMPAT
b. IHPAT
c. ELPAT
d. NVLAP

11. The laboratory analyzes a set of proficiency samples for an accreditation program. One laboratory result was reported as 23.2 µg. The PT program data indicates the mean for that sample as 19.0 µg with a standard deviation of 0.5 µg. If the program sets the control limits at + 3 SD, what can be said about the laboratory's result?

a. It is acceptable
b. It is unacceptable
c. Not enough data provided

12. Which of the following programs might a laboratory be interested in if they analyzed paint chip samples and dust for lead content?

a. ELPAT
b. NVLAP
c. IHPAT
d. NIST

13. The NVLAP program is administered by?

a. AIHA
b. EPA
c. OSHA
d. NIST

14. To participate in the IHPAT program, the laboratory must be accredited by the AIHA-LAP, LLC.

True
False

15. Where would be the best place to obtain the sample identification numbers assigned by the client during sample collection?

a. Laboratory log-in sheet
b. Field sampling sheet
c. Chain-of-custody
d. Laboratory notebook

16. Which of the following information should be reviewed prior to releasing the data to the client?

a. Batch QC data
b. Proper media used for sample collection
c. Customer sample identification numbering
d. All of the above

17. What method could the reviewer use to detect transcription errors in data?

18. How should the reviewer handle discrepancies discovered during the informational review?

a. Resolve with the analyst prior to releasing the data
b. Resolve with the analyst prior to performing the analysis the next time
c. Just document and cover during the next training session
d. No action would be required

19. Sample analysis for tricholoroethylene shows 28 µg of tricholorethylene in the primary section and 10 µg of tricholorethylene in the backup section.

a. Should the sample be rejected for breakthrough?

b. Why or why not?

c. What should the laboratory do?

20. What are some of the possible reasons that tricholorethylene was found on the backup section of the tube in Problem 19? (Identify all that apply)

a. Flow rate was too low
b. Flow rate was too high
c. Tricholorethylene concentration was too high
d. Sample was collected in a highly humid atmosphere

21. The client sends eight mixed cellulose ester filter samples for lead analysis. Seven of the filters have volumes reported and one is listed as a blank. The blank was opened during the sampling and immediately capped. This is an example of:

a. A field blank
b. A laboratory blank
c. A spiked sample
d. A diffusive badge sample

22. Analysis of the above samples yields the following results for the seven samples; 1.2, 1.3, 2.6, 3.0, 1.0, 0.2, and 0.3 µg. Analysis of the blank supplied by the customer indicates a result of 5.2 µg.

a. Is this a problem?

b. Why or why not?

c. What should the analyst do in response to the results for the blank?

d. What are some of the potential causes for the concentration on the blank sample?

23. If the laboratory also assigned a blank to the set of samples upon analysis what kind of sample would that represent?

a. Field blank
b. Laboratory blank
c. Spiked sample
d. Certified reference material

24. The blank supplied by the laboratory is analyzed and shows a result that is less than the LOD for the method. What does this information combined with the result from the client's blank indicate?

25. What should the analyst do with the additional data from the analysis of the blank supplied by the laboratory?

26. After considering all of the information above the analyst talks with the client who states that he is unaware how the analyte could have gotten on the blank he supplied. In your opinion what should be done with the analytical results for the seven samples?

27. What are some of the advantages of using a standardized report format?

28. Which of the following dates should be included on the final report?

a. Date the report was generated
b. Date the samples were received by the lab
c. Date of analysis
d. All of the above

29. What is the purpose of the unique sample identification number assigned by the laboratory?

a. To create more paper work
b. To confuse the client
c. It is required by OSHA regulations
d. To help track samples through the analytical process and avoid confusion regarding customer sample identifications

30. When the client assigns a unique field sample identification number and the laboratory assigns a unique lab identification number, what must happen in the final report?

a. Only the lab identification number is listed
b. Only the field generated identification number is listed
c. Both numbers are listed and related to each other
d. No numbers are listed so no one is confused

31. After a final report was issued, the laboratory supervisor discovers that two identification numbers were reversed in the report. How should the correct results be reported to the client?

a. Cross out the wrong numbers on the report, write in the correct numbers, and send the report
b. Issue an addendum with the correct data
c. Just call the client and let them change their copy
d. No changes is necessary if the client did not catch the problem

32. In the table below indicate any reporting units that may be expected for the types of analytes listed.

Analyte	Medium	Possible Units
Lead	Paint chip	
Culturable Bacteria	Air (Agar sample)	
Acetone	Air (Charcoal tube)	
Fibers (Asbestos)	Air (MCEF)	
Asbestos	Bulk Insulation Sample	
Fungal Spores	Air (spore trap sample)	
Lead	Surface Wipe	
Chromium	Bulk Sample	

33. A sample is analyzed for sulfuric acid mist and all of the results were less than the LOD and reporting limit for the method. The LOD for the method was 1.0 µg and the reporting limit was 2.0 µg. The reported volume for the sample was 100 L. Which of the following would be an appropriate way to report the result on the final report? (Indicate all that are appropriate)

a. 0.0 mg/m^3
b. 0.01 mg/m^3
c. 0.02 mg/m^3
d. ND (reporting limit 0.02 mg/m^3)

Competency Exercises Answer Key

Chapter 1: Laboratory Quality Management System

Answers to Competency Exercises

1. The "quality management system" is used to ensure the quality of data and to provide evidence of data quality to the producer or end user of the data. It can also help the laboratory balance data production activities with activities that reduce or eliminate errors in the produced data. A central component of the quality management system is the written quality management program.
2. "Quality assurance" refers to the overall system and program related to quality. "Quality control" describes the activities and procedures utilized to produce data of known accuracy and precision. "Quality assessment" describes the activities and procedures used to evaluate the applicability and overall quality of the management system and the laboratory data produced.
3. International Standard ISO/IEC 17025:2005, "General requirements for the competence of testing and calibration laboratories."
4. Subsections of Management Requirements are:
   - 4.1 Organization
   - 4.2 Management system
   - 4.3 Document control
   - 4.4 Review of requests, tenders and contracts
   - 4.5 Subcontracting of tests and calibrations
   - 4.6 Purchasing services and supplies
   - 4.7 Service to the customer
   - 4.8 Complaints
   - 4.9 Control of nonconforming testing and/or calibration work
   - 4.10 Improvement
   - 4.11 Corrective action
   - 4.12 Preventive action
   - 4.13 Control of records
   - 4.14 Internal audits
   - 4.15 Management reviews

5. Subsections of Management Requirements are:
   - 5.1 General
   - 5.2 Personnel
   - 5.3 Accommodation and environmental conditions
   - 5.4 Test methods and method validation
   - 5.5 Equipment
   - 5.6 Measurement traceability
   - 5.7 Sampling
   - 5.8 Handling of test and calibration items
   - 5.9 Assuring the quality of test and calibration results
   - 5.10 Reporting the results

6. A "policy" is a written statement of commitment to implement a management program element or, in other words, "what" the organization intends to do. A "procedure" is a written set of instructions that describe how to implement a policy requirement, or how to carry out a specific task. An "arrangement" is a general description of how an outcome will be achieved; it must be included in a written or electronic document.

Chapter 2: Laboratory Organization and Management System Components

Answers to Competency Exercises

1. To provide testing to meet the requirements of ISO/IEC 17025:2005 and to satisfy the needs of its customers, regulatory authorities (e.g. OSHA, states) and authorities providing recognition (e.g. AIHA-LAP, LLC).
2. The organization chart provides a summary of the reporting structure of the laboratory and inter-relationships of laboratory staff with other organizational (e.g. corporate) entities and/or support operations.
3. The primary positions are Laboratory Director/Technical Manager, Quality Manager, Analyst, and Technician. Sample Custodian, LIMS Manager, etc. may also be separately identified positions. The relationships between quality management, technical operations and support services must be defined in the quality manual (Section 4.1.5.e) as well as the roles and responsibilities of technical management and the quality manager, including their responsibility for ensuring compliance with ISO/IEC 17025 (4.2.6). Other duties and responsibilities may be defined in either the quality manual or in job descriptions.
4. C
5. ISO/IEC 17025:2005 Sections 4.2.2, 4.2.6, AIHA-LAP, LLC Section 2A.4.2.1.
6. See ISO/IEC 17025:2005, Sections 4.2.2a-e.
7. Top level laboratory management
8. False. Only policies related to quality must be included in the quality manual (See ISO/IEC 4.2.2). Quality system procedures may be included in the quality manual or they may be in separate documents referenced in the quality manual.

Chapter 3: Document Control through Complaints

Answers to Competency Exercises

1. A, B, D, E, G, and H. C, F, and I are records, not documents.
2. The master document list allows affected staff to determine the current approved revision of the controlled documents related to their responsibilities and the distribution (location) of the documents. Both internally generated and external source documents must be included.
3. With an Analytical Request Form
4. The laboratory must maintain records of review of requests, tenders, and contracts and records related to the customer's requirements or the results of the work.
5. False. Laboratories must advise the customer in writing and gain the approval of the customer.
6. Commonly used criteria are:
   - QS registration (ISO 9000/9001)
   - Accreditations (ISO/IEC 17025, ISO/IEC 17043, ISO/IEC Guide 34)
   - Original Equipment Manufacturer (OEM)
   - Past Experience
   - Industry standard
   - Sole source
   - Meets requirements
7. E. Printer paper would not be considered a supply that affects the quality of testing by the laboratory.
8. True
9. Complaining parties may include, among others:
   - Employees
   - End users of data
   - Subcontractors
   - Suppliers
   - Accreditation bodies
   - Regulatory agencies
   - Laboratory neighbors

Chapter 4: Non-conforming Testing Work through Preventive Action

Answers to Competency Exercises

1. False. Nonconformities can apply to any aspect of a laboratory's testing work, or the results of this work, that do not conform to its own procedures or the agreed requirements of the customer. The nonconformity may not affect the quality of the final data. Evaluation of the significance of a nonconformity is a key element of the laboratory's control of nonconforming testing work policies and procedures.
2. C
3. Data review process and internal audits
4. Nonconforming work procedures must ensure:
   - The responsibilities and authorities for the management of nonconforming work are defined.
   - Actions are defined and taken when conforming work is identified.
   - Who has the authority to stop and resume affected work activities is identified.
   - Withholding of test reports is done when necessary.
   - A determination is made of when customers must be notified.
   - The significance of each nonconformity is evaluated.
   - Immediate action is taken to correct the nonconformity.
   - A decision on when a formal corrective action process must be initiated is made.
5. True. However, the form and details of documentation may vary depending on the significance of the nonconformity.
6. Identifying the root cause(s) of the nonconformity.
7. The corrective action process includes:
   - Identifying potential corrective actions.
   - Selecting and implementing the action(s) most likely to eliminate the problem and to prevent recurrence.
   - Ensuring that corrective actions are appropriate to the magnitude and the risk of the problem.
   - Documenting and implementing any required changes resulting from corrective action investigations.
   - Monitoring the results to ensure that the corrective actions taken have been effective.
   - Auditing of the areas of activity (may be required based upon the significance of the nonconformity).

8. Preventive action is a pro-active process that occurs before a nonconformity occurs. Corrective action is a reactive process that occurs after a nonconformity has been identified.
9. False. Preventive action plans must be documented.
10. The following can be used to continuously improve a laboratory's quality management system:
    - Its quality policy and objectives
    - Audit results
    - Analysis of data
    - Corrective and preventive actions
    - The management review

Chapter 5: Control of Records, Internal Audits and Management Review

Answers to Competency Exercises

1. B, C, D & F. A & E are documents not records
2. A, C, and F
3. Procedures must address how records are:
   - Identified
   - Collected
   - Indexed
   - Accessed
   - Filed
   - Stored
   - Maintained
   - Disposed

   A convenient way to address these is through the use of a records management matrix.
4. Consult federal, state, and local regulations, and customer and accrediting organizations' requirements.
5. Records may be stored as:
   - Hard copy files in properly protected storage areas
   - Scanned copies stored electronically
   - Original electronic files with proper backup and protection from alteration
6. Requirements from:
   - ISO/IEC 17025
   - AIHA-LAP, LLC policies (and/or other applicable accreditation requirements)
   - The laboratory's internal policies and procedures
   - Customer requirements (if they differ from those stated above)
7. Audit records should include:
   - Documentation of both conformity and nonconformity with requirements (often through a detailed notated checklist)
   - Documentation of the specific laboratory policies and procedures audited and personnel interviewed and/or observed
   - Listing of records reviewed to support conformity or nonconformity with requirements
   - Brief narrative report to summarize the overall findings is also recommended.

8. True
9. Follow-up audits are typically performed to verify that corrective actions have been implemented and are effective.
10. The management review is a review by top level management to determine the continued suitability and effectiveness of the management system and to introduce necessary changes or improvements while the internal audit is an evaluation of activities versus defined requirements.
11. Review of the laboratory's overall quality objectives.

Chapter 6: General, Personnel, and Accommodation and Environment

Answers to Competency Exercises

1. Factors are:
   - Human factors
   - Accommodation and environmental conditions
   - Test methods and method validation
   - Equipment
   - Measurement traceability
   - Sampling (generally limited lab control)
   - Handling of test items
2. Management personnel responsibilities include:
   - Ensuring that competent personnel are used to perform critical job functions
   - Providing appropriate supervision for personnel
   - Formulating goals with respect to personnel education, training and technical skills
   - Authorizing personnel who have met required training requirements to perform critical job functions.
3. D
4. E
5. A & D. ABIH certification is not required and the technical manager cannot be a consultant. The technical manager must be an employee of the laboratory.
6. Personnel training records must include:
   - Job descriptions
   - Relevant authorizations, showing dates
   - Educational and professional qualifications
   - Training, including demonstrations of competency
   - Description of training program(s), including content and duration
   - Skills and experience
7. Demonstrations of competency must be documented through the successful analysis of proficiency testing (PT) samples, certified reference materials (CRMs) or in-house quality control samples. Demonstrations of proficiency must be documented at a minimum of every six months.

8. Laboratories must:
    - Monitor, control and record environmental conditions that may affect analytical results
    - Effectively separate areas in which incompatible activities occur to prevent cross-contamination
    - Control access to areas affecting the quality of tests
    - Ensure good housekeeping within the lab.
9. Analyses requiring environmental monitoring include:
    - Bulk asbestos identification by PLM – temperature to determine impact on RI fluids
    - Gravimetric analysis – temperature and humidity
    - Environmental lead analysis – surface wipes for lead (Pb) contamination control
    - Microbiological analyses – air and/or surface samples for contamination control.

Chapter 7: Elementary Statistics

Answers to Competency Exercises

1. B
2. A and B (counting data are a subset of discrete data)
3. B
4. Differences in counting techniques, subjective calls on what is or is not a colony, human error, analyst training, and subjectivity in organism identification.
5.

Experimental Process	Data Type
Recording Successive Rolls of a Die	Discrete
Concentration from Gas Chromatography Run	Continuous
Fiber Concentrations Using NIOSH 7400	Discrete and Counting
Lead Concentrations from Atomic Absorption Analysis	Continuous
Polling Lab Analysts to Determine Their Favorite Analyte	Qualitative

6. Accuracy is the ability to measure close to the true value of an analyte. Precision is the ability to replicate or reproduce a measured value. Bias is the difference between a reference value and the measured value.
7.

Statement	Analyte
a. The data are unbiased but not precise.	Ethanol
b. The data are precise but biased.	Benzene
c. The data are accurate (both unbiased and precise).	Acetone
d. The data are not accurate.	Chloroform & Benzene

8. If rounding is performed too early in the analytical process the end result may be inaccurate. Reporting too many significant digits would imply that a reported number is more accurate than it actually is, whereas reporting too few significant digits would imply that the number is not as accurate as it really is.

9.

Reported Result	Significant Digits
1.0002	5
2.3	2
1	1
127	3
6.234	4
0.0012	2
100.002	6
23.025	5
0.002360	4
4.000	4

10.

Input Data	Significant Digits for Result
1.02, 2.3, 2.112	2
1.0047, 1	1
0.23, 1.0, 21.3	2
100.02, 1.123	4
0.2369, 10.2	3
1.2, 2.1, 1.3	2

11.

Number	Significant Digits	Answer
0.1356	3	0.136
123.14	4	123.1
135	2	140
0.1123467	1	0.1
12.400	4	12.40

12.

Numbers	Answer
1.23, 2.45, 23.12, 16.7	43.5
123.04, 234.112, 125.3	482.5
0.12, 0.998, 0.9765, 1.23	3.32
12, 23, 456, 112	603
0.1, 0.22, 1.23, 2.3	3.8

13.

Numbers	Answer
2, 21.2, 1.23	50
1.25, 23.4, 12.22	357
0.11, 0.123, 0.1235	0.0017
0.11, 1.23, 10.2	1.4
1.234, 123.5, 0.00235	0.358

14.

Data Group	Total Population, Sample Group, or Sub-sample?
All registered voters	Population
All residents of the State of New York	Population
100 fields from a slide mount	Sub-sample
A 10 mL aliquot from 100 mL of eluent	Sub-sample
A set of air filters for a week of monitoring	Sample Group
A collection of floor dust from a day's cleaning	Sample Group
Factories that apply paint to steel	Population

15. The mean represents the arithmetic average of all data points in a data set. The standard deviation represents the dispersion of the individual data points around the mean.

16.

Data Points	Sample Mean	Standard Deviation
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6	2.0	0.77
32, 33, 31, 32, 33, 34, 34, 36	33	1.6
1.002, 1.009, 1.004, 1.001, 1.008	1.005	0.0036
20.7, 21.0, 20.3, 22.8, 21.1, 22.3	21.4	0.97
0.23, 0.31, 0.33, 0.45, 0.52, 0.21	0.34	0.12
34, 67, 98, 120, 111, 54, 65, 77	78	29

17.

Data Points	CV	Relative Standard Deviation
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6	0.39	39%
32, 33, 31, 32, 33, 34, 34, 36	0.048	4.8%
1.002, 1.009, 1.004, 1.001, 1.008	0.0036	0.36%
20.7, 21.0, 20.3, 22.8, 21.1, 22.3	0.045	4.5%
0.23, 0.31, 0.33, 0.45, 0.52, 0.21	0.35	35%
34, 67, 98, 120, 111, 54, 65, 77	0.37	37%

For example: First set of data is calculated as follows:

CV = standard deviation/mean

= 0.77 / 2.0

= 0.39

RSD= CV X 100

= 0.39*100

= 39%

18. You should be able to determine the most precise measurements by looking at the relative standard deviations which represent the spread around the mean. Thus the data set with a relative standard deviation of 0.36% would be the most precise and the set with the relative standard deviation of 37% would be the most imprecise. You cannot determine accuracy until you know the true value of the analyte. If the run represented analysis of a spiked QC sample you could compare the mean to the calculated spiked concentration to determine accuracy.

19.

Data Points	Degrees of Freedom
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6	6
32, 33, 31, 32, 33, 34, 34, 36	7
1.002, 1.009, 1.004, 1.001, 1.008	4
20.7, 21.0, 20.3, 22.8, 21.1, 22.3	5
0.23, 0.31, 0.33, 0.45, 0.52, 0.21	5
34, 67, 98, 120, 111, 54, 65, 77	7

Degrees Of Freedom = number of observations in each set minus 1

20.

Data Points	Individual Point Confidence Interval
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6	2.0 ± 1.9
32, 33, 31, 32, 33, 34, 34, 36	33 ± 3.6
1.002, 1.009, 1.004, 1.001, 1.008	1.005 ± 0.010

For example: First set of data calculated as follows:

CI(x) = xbar+/- (t * s)

= 2.0 +/- (2.447 * 0.77)

= 2.0 +/- 1.9

21.

Data Points	Sample Mean Confidence Interval
1.2, 1.6, 2.0, 3.2, 1.1, 2.4, 2.6	2.0 ± 0.71
32, 33, 31, 32, 33, 34, 34, 36	33 ± 1.3
1.002, 1.009, 1.004, 1.001, 1.008	1.005 ± 0.004

For example: First set of data calculated as follows:

CI(x) = xbar+/- $[(t * s)/(n^{1/2})]$

= 2.0 +/- $[(2.447 * 0.77)/(7)^{1/2}]$

= 2.0 +/- [(1.88/2.65)]

= 2.0 +/- 0.71

22. The third data set has the narrowest confidence intervals and the first data set has the broadest.

23. Analyze more samples (increase sample population) or eliminate some of the error in the analytical process.

24. y = 11.6x - 2.7

25. r = 0.978 , r^2 = 0.956 NO, r is less than 0.995

Chapter 8: Analytical Methods and Method Validation

Answers to Competency Exercises

1. D & E
2. A
3. B
4. False. The laboratory must advise the customer if an inappropriate or out of date test method is requested.
5. D
6. B
7. C
8. False. The laboratory must confirm that it can properly conduct standard methods prior to use on customer samples.
9. True
10. Laboratories are only required to use a specific method only when specified in a Federal, State or local regulation or by an accrediting organization. A client may request a specific method.
11. False. Standard methods typically include various options that laboratories must document and minor changes related to specific instrument and/or calibration ranges that must be documented as part of the laboratory analytical procedure.
12. A
13. B
14. B
15. A
16. D
17. C
18. It is acceptable as above the minimum recovery percentage (75%); however, a higher recovery percentage at the PEL concentration (90% or greater) would be preferred.
19. The LOD is the smallest concentration of an analyte that can be detected by the measurement system. The LOQ refers to the smallest concentration of an analyte that can be quantified by the method. The useful range of measurement refers to the concentrations that can be determined by the measurement system (usually between the LOD and UQL).
20. Sections 2B.4.1 through 2B.4.3.

Chapter 9: Estimation of uncertainty of measurement (5.4.6) and Control of Data (5.4.7)

Answers to Competency Exercises

1. Result of the evaluation aimed at characterizing the range within which the true value of a test result is estimated to lie, generally within a given likelihood. OR

   Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used.

2. Sampling

3. A: combined standard uncertainty:

   $= [(2.2)^2 + (1.4)^2 + (3.0)^2 + (9.8)^2 + (0.7)^2 + (1.4)^2]^{1/2}$

   $= [(4.84) + (1.96) + (9.0) + (96.0) + (0.49) + (1.96)]^{1/2}$

   $= [114.3]^{1/2}$

   $= 10.7$

   B: expanded uncertainty:

   = (combined standard uncertainty) x (coverage factor)

   = (10.7) * (2)

   = 21.4

4. When a component of uncertainty is less than one-third of the largest component, it can be ignored.

5. Laboratory control samples (or CRMs) and laboratory duplicate sample results.

6. Required elements include:
   - comparison of quality control data against established acceptance limits
   - computation verification
   - transcription of data
   - adherence to the procedures established in the laboratory management system documents
   - If more than one parameter in a sample is tested, then the correlation of results shall be reviewed

7. Methods of protecting electronic data include:
   - protecting formulae within spreadsheet template applications
   - password protection of data directories and/or LIMS functions
   - confidentiality statements on reports, fax cover sheets, and email footers,
   - use of pdf files in email transmittals
   - regularly scheduled backing up of electronic databases
8. True

Chapter 10: Equipment (5.5) and Traceability of Measurement (5.6)

Answers to Competency Exercises

1. C
2. Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty
3. Balance calibration masses (weights), a NIST traceable reference thermometer, and stage micrometer
4. Reference materials. The reference materials may include Standard Reference Materials (SRM) from NIST, and certified reference materials (CRM).
5. C
6. A: Equipment records must include:
   - the identity of the item of equipment and its software;
   - the manufacturer's name, type identification, and serial number or other unique identification;
   - checks that equipment complies with the specification (see 5.5.2);
   - the current location, where appropriate;
   - the manufacturer's instructions, if available, or reference to their location;
   - dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;
   - the maintenance plan, where appropriate, and maintenance carried out to date;
   - any damage, malfunction, modification or repair to the equipment.
   - The name or initials of the person performing the maintenance or repair

   B. ISO/IEC 17025 Section 5.5 and AIHA-LAP, LLC policy 2A.5.5.1
7. False. Laboratories are required to determine the reporting limits for all test methods except asbestos and gravimetric methods (see AIHA-LAP, LLC Policy 2B.3)
8. Three plus a blank (Two, where possible, plus a blank for ICP-AES) (see AIHA-LAP, LLC Policy 2B.3.4)
9. An error in the concentration of the original reference material used to prepare instrument calibration standards or an error in the preparation of instrument calibration standards prepared by the analyst.
10. Through the analysis of media spiked samples at or below the reporting limit and carried through the entire analytical procedure.

Chapter 11: Sampling (5.7) and Handling of Test Items (5.8)

Answers to Competency Exercises

1. B
2. A and B
3. C
4. A, B, and D
5. A
6. B, C, and D
7. B and D
8. D
9. They should not be stored together because of the possibility of contamination of the air samples.
10. D
11. Only procedures related to subsampling as part of the testing process or procedures for collection of samples to monitor the laboratory environment, as applicable, are required.

Chapter 12: Assuring the quality of test results (5.9), Data reduction and validation, and Reporting the test results (5.10)

Answers to Competency Exercises

1. B
2. C
3. D
4. Continuing calibration verification and initial calibration verification standards (CCV and ICV). ICVs are used to check the initial calibration of the instrument and CCVs are used during an analysis to verify the continuing calibration of the instrument.
5. Matrix spikes are spikes of actual customer samples while method or media spikes are spikes of clean (unused) media.
6. B
7. B
8. A. Yes
   B. Yes
   C. Yes The results indicate a statistically significant trend. Seven consecutive points on one side of the mean.
9.

Data	Mean	SD	Control Limits	Warning Limits
1.0, 1.1, 1.4, 1.5, 1.4, 1.5, 1.2	1.3	0.2	0.7 to1.9	0.9 to 1.7
23, 23, 24, 25, 26, 26, 23, 24	24	1.3	20 to 28	21 to 27
96, 96, 97, 98, 101, 100, 100	98	2.1	92 to 104	94 to102

10. A, B, and C
11. B
12. A
13. D
14. False
15. B or C
16. D

17. A review of raw data and final report by an independent reviewer prior to release to the customer.
18. A
19. A. Yes
    B. Breakthrough exceeds 25%.
    C. The breakthrough percentage should be reported to the client.
20. B, C, and D
21. A
22. A. It may be.
    B. The customer could have spiked the sample as a QC sample so there may not be a problem. If the client did not spike the sample the source of the contamination should be found.
    C. The laboratory should report the concentration on the blank to the customer.
    D. The customer may have spiked the sample, the sample was contaminated in the field, or the sample was contaminated in the lab, or the samples were mislabeled in the field and the "blank" is really a sample.
23. B
24. If the sorbent tube is from the same lot as the sample the results would indicate that the contamination was not present in the tube when received from the manufacturer. The results would also indicate the contamination is not in the lab's reagents/solvents/standards.
25. Supply the information to the customer.
26. The analytical results should be reported to the client along with the results from the field blank. The client can make a determination as to the use of the data based on the supplied information. The laboratory should also considering flagging or commenting on these data within the report.
27. A standardized report format simplifies report review by the laboratory and assists in understanding by the customer.
28. D
29. D
30. C
31. B

32.

Analyte	Medium	Possible Units
Lead	Paint chip	mg/cm^2, mg/kg, %, ppm
Culturable Bacteria	Air (Agar sample)	cfu/m^3
Acetone	Air (Charcoal tube)	mg/m^3, ppm, ug/m^3, ppb, ug/sample
Fibers (Asbestos)	Air (MCEF)	f/cc, f/mm^2
Asbestos	Bulk Insulation Sample	%
Fungal Spores	Air (spore trap sample)	Str/m^3, spores/ m^3
Lead	Surface Wipe	ug/ft^2, ug/cm^2, ug/wipe
Chromium	Bulk Sample	mg/kg, ppm, %

33. C or D