NOVAMAL

PN 10-056-14

KP 24.14.63

This company standard is valid for the production, testing and delivery of Novamal.

1. IN GENERAL

1.1 **CAS number:** 111-91-1 **EINECS number:** 203-920-2

1.2 Novamal (bis-(2-chloroethoxy)-methane) is produced by acetylation of parafomraldehyde with 2-chloroethanol under present of catalyst.

Chemical formula: $C_5H_{10}O_2Cl_2$

Relative molecular weight: 173,04 $g.mol^{-1}$

1.3 Novamal is pure, light yellow liquid with a typical odor. Novamal is soluble in aromatic and chlorinated hydrocarbons.

1.4 Novamal is used for production of polysulphides.

2. TECHNICAL PARAMETERS

Novamal must comply with the following technical requirements:

Parameter	Unit	Value	Test method
Content of Novamal, min.	%	80	FCH-PP-7-26-2014
Content of higher homologues, max.	%	19,8	FCH-PP-7-26-2014
Content of ethylene chlorohydrin, max.	%	0,30	FCH-PP-7-26-2014
Content of sulphuric acid, max.	%	0,05	FCH-PP-7-31-2014
Content of formaldehyde, max.	%	0,50	FCH-PP-7-31-2014
Content of iron, max.	ppm	5	FCH-PP-7-31-2014

Supply: PND 10-056-12 from 01.08.2012

Approved:	Ing. Tibor Žiak Head of Quality Management and Controlling Signature: Date: 5. 8. 2014	Ing. Jaroslav Horák Head of Technology Signature: Date: 6. 8. 2014

3. SAMPLING AND TESTING

3.1 Sampling

The sampling is performed according to STN 65 0512.

3.2 Testing

The following procedures for determination are shortened by the version of the FCH-PP-7-26-2014, which will, if necessary, provided to the customer´s request.

3.2.1 Determination of Novamal, ethylene chlorohydrin and homologs content

3.2.1.1 Principle of determination

The principle of determination is analysis of samples by capillary chromatography. Individual components in samples are identified on the basis of their typical retentive time. Flame ionization detector (FID) is used for detection of components eluting from chromatography column. Peak areas of individual components are in analysed range directly proportional to their concentration.

3.2.1.2 Chemicals, materials and apparatuses

Standard substances:

- diethylether, p. a.

Gases:

- nitrogen fluorescent
- hydrogen electrolytic
- compressed air, dried

Materials:

- microvolume syringe capacity 0,2 µl

Apparatuses:

- gas chromatograph for work with capillary columns
- autoinjector
- evaluation unit for quantitative data processing

3.2.1.3 Procedure for determination

Liquid sample is thoroughly mixed and dosed by microinjector directly into the column of gas chromatograph. The optimum dose is 0,2 µl.
Chromatography record showed the content of ethylene chlorohydrin, Novamal and higher homologs (sum of impurities X_1, X_2, X_5 and X_7) in % (surface).

3.2.1.4 Calculation

The content of X- component in % (surface) is given by the formula:

$$X = \frac{x_i}{\sum_{i=1}^{n} x_i} \times 100$$

where x_i is a surface (area) of peak of i-component

$\sum_{i=1}^{n} x_i$ sum of peak surfaces of individual components

i i-component in the sample

n total number of components in the sample (n = 6)

100 calculation to percentage

The result of determination is arithmetic average of two paralel determinations made step by step at the same method, the same person, with the same instrument under the same conditions.

3.2.2 **Determination of sulphuric acid content**

3.2.2.1 Principle

The principle of determination of sulphuric acid content is alkalimetric titration. The alkalimetric titration is the neutralization reaction of acid with the base to form the salt. The end of reaction is indicated by coloured transfer of the indicator.

3.2.2.2 Chemicals and materials

- sodium hydroxide, volumetric solution with the concentration c(NaOH) = 0,1 mol/l
- indicator – bromthymol blue 0,1 % solution in 20 % ethanol
- ethanol, denaturated, 96 %
- analytical balance capable of weighing to 0,01 g
- pipette capacity 25 ml
- Erlenmayer flask capacity 250 ml
- titration device with reading accuracy of 0,01 ml

3.2.2.3 Procedure for determination

10 g of the sample is weighed into the Erlenmayer flask. 25 ml of ethanol, the indicator are added and the content of the flask is titrated with the sodium hydroxide solution to blue colouring.

3.2.2.4 Calculation

The content of sulphuric acid (X) in % is given by the formula:

$$X = \frac{a_{NaOH} \times f_{NaOH} \times c_{NaOH} \times M_{H2SO4} \times 100}{n_1 \times 1000 \times 2}$$

where a_{NaOH} is the consumption of NaOH volumetric solution with the concentration c(NaOH) = 0,1 mol/l v ml
n_1 the mass of the test sample in g
f_{NaOH} factor of NaOH volumetric solution with the concentration c(NaOH) = 0,1 mol/l v ml
c_{NaOH} concentration of NaOH volumetric solution in mol/l
M_{H2SO4} molar weight of H_2SO_4 in g/mol (98,08 g/mol)
1000 conversion from mililitres to litres
2 coefficient resulting from stoichiometric aquation
100 counting to % (w/w)

The result of determination is arithmetic mean of two paralel determinations rounding-off to two decimal places.

3.2.3 **Determination of formaldehyde content**

3.2.3.1 Principle

The principle of this determination is alkalimetric titration of the sample with sodium hydroxide by using methyl orange indicator.

3.2.3.2 Chemicals and materials

- sodium hydroxide, volumetric solution with the concentration c(NaOH) = 1 mol/l
- hydroxylaminehydrochloride, 10 % solution
- methyl orange, 0,1 % water solution
- analytical balance capable of weighing to 0,01 g
- Erlenmayer flask capacity 250 ml
- pipette capacity 10 ml
- titration instrument with accuracy of reading on 0,01 ml

3.2.3.3 Procedure

Blank test: methyl orange indicator is added into the 10 ml of hydroxylaminehydrochlorid and it is titrated with the NaOH volumetric solution.

20 g of the sample is weighed into the Erlenmayer flask. 10 ml of hydroxylamine-hydrochloride is added, the solution is shaked and allowed to stand for 5 minute. The methyl orange indicator is added and titrated with the NaOH volumetric solution to orange colouring.

3.2.3.4 Calculation

The content of formaldehyde (Z) in % is given by the formula:

$$Z = \frac{(a - b) \times c_{NaOH} \times f_{NaOH} \times M_{HCHO} \times 100}{n \times 1000} - (X \times 0{,}306)$$

where n is the mass of the test sample in g
a consumption of NaOH volumetric solution with the concentration c(NaOH) = 1 mol/l na vzorku v ml
b consumption of NaOH volumetric solution with the concentration c(NaOH) = 1 mol/l na vzorku v ml – the blank test in ml
c_{NaOH} concentration of NaOH volumetric solution in mol/l (1 mol/l)
f_{NaOH} factor of NaOH volumetric solution
M_{HCHO} molar mass of formaldehyde in g/mol (30,027 g/mol)
1000 conversion from mililitres to litres
100 counting to % (w/w)
X content of sulphuric acid determinated according to FCH-TS-O-xx-ÚRKK-63/2013 in %

$$0{,}306 = \frac{M_{HCHO}}{M_{H2SO4}}$$

where M_{HCHO} is the molar mass of formaldehyde in g/mol (30,027 g/mol)
M_{H2SO4} molar mass of sulphuric acid in g/mol (98,08 g/mol)

The result of determination is arithmetic average of two paralel determinations rounding-off to two decimal places.

3.2.4 **Determination of iron content**

3.2.4.1 Principle

Determination of iron content is based on the reaction of trivalent iron with ammonium thiocyanate in alcoholic medium under formation of red colouring suitable for colorimetric determination. The method is used for determination of Fe in concentrations over 0,05 mg/l.

3.2.4.2 Chemicals and materials

- Standard calibration solution with the iron content of 1,000 g/l (CRM)
- 15 % solution of nitric acid in ethanol
- 10 % solution of ammonium thiocyanate in ethanol
- ethyl alcohol (denatured alcohol)
- destilled water

- volumetric flasks capacity 50 ml, 1000 ml
- pipettes capacity 5 ml, 10 ml
- spectrophotometer with accessories
- analytical balance

3.2.4.3 Procedure for determination

20-30 g of the sample is weighed into the 50 ml volumetric flask and dissolved in a small amount of denatured ethanol. 10 ml of 15 % ethanolic solution of nitric acid is added and the content is allowed to stand for about 5 min. Then 10 ml of 10 % ethanolic solution of ammonium thiocyanate is added and allowed to stand for about 10 min. and filled up to the line with the denatured ethanol. After mixing the solution is poured into the cell, it is inserted into the spectrophotometer and the extinction is measured at wavelenght of 520 nm. At the same time the blank test ist prepared at the same way but without the sample. The blank test is used for zero adjustment of the device.

NOTE: The spectrophotometer is regularly (min. 1 per year) calibrated to the desired concentration range. At the same time, the calibration curve is constructed, where the Fe content in mg in 50 mL volumetric flasks is on the x-axis and the extinction is on the y-axis.

3.2.4.4 Calculation

The Fe content in the directly weighed sample into the 50 ml volumetric flask is given by the formula:

$$Fe[\%hmot.] = \frac{a}{n * 10}$$

$$Fe[ppm] = 10^4 . * Fe[\%hmot.] = 10^4 * \frac{a}{n * 10}$$

where a is the Fe content calculated from the calibration curve in mg
n the mass of the test sample in g

4. PACKAGING, TRANSPORT, LABELLING AND STORAGE

4.1 Novamal is delivered in stainless railway tanks.

4.2 Each pack unit must be labelled with the following informations:

a) producer name
b) product name
c) net weight
d) production date
e) number of standard

4.3 Novamal is storage in the steel tanks surface of which is treated by popypropylene.

4.4 Storage time of the product upon observation of the above terms is 1 year from the date of dispatch.

5. SAFETY STATEMENTS

Detailed data are mentioned in Safety Data Sheet.

6. CITED STANDARDS

STN 65 0512	Sampling of liquids