



Miskolc, 2015.szeptember 3.

## 1. számú melléklet az M15-KJ-11-016 számú szerződéshez

STP No. 017 B / 2009  
It replace STP 017b / 2003

### TECHNICAL SPECIFICATION FOR PRODUCT ISOPROPYLAMINE SOLUTION

#### 1. GENERALITIES

1.1. COMMERCIAL NAME: ISOPROPYLAMINE SOLUTION

1.2. CHEMICAL NAME: ISOPROPYLAMINE

1.3. FIELD OF USAGE: Used as corrosion inhibitor, manufacturing of colouring agents, drugs, pesticides.

#### 2. TECHNICAL CONDITIONS FOR QUALITY

No.	Property	MU	Conditions for Admissibility	Analysis methods
1.	Apperance	-	clear liquid without mechanical impurities	visually
2.	Colour	-	colourless to light yellowish	visually
3.	Isopropylamine content	%	min. 70	Annex 1
4.	Total chemical impurities from wich:	%	max. 0.21	Annex 2
	Ammonia	%	max. 0.21	
	Acetone	%	max. 0.14	
	Isopropylic alcohol	%	max. 0.07	
	Diisopropylamine	%	max. 0.07	
	Monoethylamine	%	max. 0.14	
	Other amines	%	max. 0.14	



### 3. PACKING

The product is packed in:

- steel railway tanks
- isocontainers
- special barrels

Maximum filling grade of packing is: 0.62 kg/l.

### 4. MARKING, HANDLING, STORAGE, TRANSPORT, DOCUMENTS

**4.1. Marking of packings** is made by templating/labelling at visible places with the following specifications:

- name of manufacturing company
- product name according to STP nr. 017B/2009
- lot number / tank number
- warning symbol
- warning signs in accordance with warning symbol
- inscription for warning symbol
- risk phrases (R...)
- safety phrases (S...)
- EC number corresponds with the EINECS inventory
- manufacturing date (except tanks and isocontainers)
- term of validity (except tanks and isocontainers)

**4.2. Product handling** requests cautions applicable for an extremely flammable product.

Product handling is made away from fire sources and open flame. Use only non-sparking handtools and explosion proof electrical equipment.

**Protective equipment used** is: gloves resistant to acid and alkali, overall, boots resistant to acid and alkali, self-contained breathing apparatus with full facepiece and organic vapor cartridge.

Avoid product handling, storage and transport together with: strong acids, strong oxidizing agents, bromine, mercury, halogens.

Vapours of product may form explosive mixture with air and oxygen.

Attacks the metals: copper, zinc, aluminium alloys.

**4.3. Product is stored** in steel cylinder and vertical pressure tanks, outside, away from heat action, connected to grounding belt. Product packed in barrels is stored in its original packing, in dry and clean rooms provided with ventilation.

Maximum recommended temperature for storage is of 40°C.

**4.4. Transport of product** is made by steel railway tank / isocontainers.

According to ADR / RID valid ed., the product is included in risk class 3/FC, identification number UN: 2733, identification hazard number HI: 338, hazard label applied is nr. 3.8.

**4.5.** Each lot of product will be accompanied by a document for quality certification elaborated according to legal provisions in force.

### 5. VALIDITY TERMS

Validity term for product is **24 months**, in conditions of handling, storage and transport provided at item 4.

## **6. RULES FOR QUALITY CHECKING**

**6.1** Checking of product quality is made on lots.

**6.2.** Size of a lot is correspondent to packing type used:

- capacity of a railway tank
- capacity of isocontainers
- in case of packing in barrels, lot size is of max. 15000 kg

**6.3.** At checking, product has to meet all the technical conditions for quality provided at point no 2.

**6.4.** Checking of product characteristics is made on a sample, sampled in the following mode:

- from railway tank – from each railway tank
- from isocontainer – from each isocontainer
- from barrels: checking, of product characteristics is made on a representative average probe sampled acc to scheme for checking quality AQL = 0,1%; severely level checking, simple sampling

Probes are sampled with a glass tube wich is introduced into package so that to be filled from the entire hight of product layer.

The sample is analyzed for determination of technical conditions for quality provided at item 2.

**6.5.** The inspection concerning net mass and the mode of product marking is made according to schedule for quality checking AQL =2,5 normal level checking, simple sampling.

## DETERMINATION OF ISOPROPYLAMINE CONTENT

### 1.1 Method principle:

Method consist in volumetric determination of isopropylamine content by total alkalinity titration of the sample with sulfuric acid solution 1n, in presence of bromine thymol blue indicator.

### 1.2 Reagents:

- sulfuric acid, solution 1N standardized
- bromine thymol blue indicator solution – dissolve 0.1g of indicator in 3.2 cm<sup>2</sup> of 0.05n sodium hydroxide solution, add 80 ml water and 20 ml ethanol (96%). The solution colour must be from blue greenish.

### 1.3 Working mode:

In a 100 cm<sup>3</sup> iodometric flask, introduce 20 ± 30 cm<sup>3</sup> neutralised water against bromine thymol blue. Weigh the flask with a precision of 0.0002 g. Quickly introduce 2 cm<sup>3</sup> sample (with the top of pipette very close to aqueous layer) and stirr slightly the flask content for amine dissolution. Stopper the flask and weigh it with the same precision.

The difference between the two weighings represents the mass of the sample.

Rinse the stopper with neutralised water, add 2-3 drops of bromine thymol blue and titrate the sample, with stirring, with 1n sulphuric acid solution to the bromine thymol blue end point (changing of colour from blue to green).

### 1.4. Calculation:

Calculate the total alkalinity (expressed as isopropylamine):

$$\text{Total alkalinity (expressed as IPA) } = \frac{V \times 0,059 \times f \times 100}{m}, \quad [\%]$$

where:

V = volume of sulphuric acid solution 1n, used for sample titration, in cm<sup>3</sup>

0.059 = isopropylamine mass, coresponding to 1 cm<sup>3</sup> sulphuric acid solution n, in g

m = mass of the sample, in g

f = factor of sulphuric acid solution 1n

Isopropylamine content, % (IPA) = Total alkalinity (IPA content), % - Total chemical impurities, %

## DETERMINATION OF CHEMICAL IMPURITIES CONTENT

### 2.1 Method's principle:

Method consist in determination of solution isopropylamine content by gas chromatographic analysis using two capillary column and two detectors (of thermal conductivity for inorganic compounds detection and flame ionization for organic compounds detection).

The quantification is made by normalization (corrected area).

The normalization technique consists in multiply the areas associated with the component by an appropriate calibration factor. The calibration factors are obtained from standard mixtures of known composition and should be determined for each apparatus.

### 2.2 Instruments and materials:

- gas chromatograph equipped with thermostate for capillary column, a thermal conductivity detector and flame ionization detector
- Computer for registration and data processing or other type of registrator
- Capillary columnes type CP-Volamine -7448 type (length = 60m, internal diameter = 0.32 mm, film thickness = 0.45  $\mu\text{m}$ )

### 2.3 Working conditions:

#### - Injector:

- injector temperature: 250<sup>0</sup> C
- column flow: 1,61 ml/min
- split ratio: 1: 50

#### - Column:

- programated condition
- initially temperature of column: 80<sup>0</sup> C (maintain during 8.50 min)
- finaly temperature of column: 200<sup>0</sup> C (maintain during 8.50 min)
- growing ratio temperature: 20<sup>0</sup> C/min

#### - FID detector:

- detector temperature: 250<sup>0</sup> C
- make-up flow: 30 ml/min
- hydrogen flow: 40 ml/min
- air flow: 400 ml/min

#### - TCD detector:

- detector temperature: 250<sup>0</sup> C
- intensity electrical power: 70 mA
- make-up flow: 8 ml/min

### 2.4 The order of elution:

- air
- ammonia
- water
- monoethylamine
- monoisopropylamine
- acetone
- isopropylic alcohol
- diisopropylamine
- other amines

## 2.5 Procedure:

After fulfil the working conditions of apparatus, there are established the calibration factors for each compound, using a standard mixture of known concentrations, and in a similar concentration of the sample being analyzed.

Inject the sample into the chromatograph with microsyringe. Sample volume is 1 µl.

Record chromatogram.

## 2.6 Calculation:

It is calculate each chromatogram as follows:

For each compound ( $C_i$ ), in percent, is calculate as follows:

$$C_i = A_i \times f_i \times 100 / \sum (A_i \times f_i) \quad , \quad [ \% ]$$

where:

- $A_i$  = area of compound "i" from the sample being analyzed
- $f_i$  = calibration factor corresponding to compound "i" , established by standardization
- $\sum (A_i \times f_i)$  = sum of areas for every compounds from the chromatograms, corrected with calibration factors.

The final integration, after calculation of both chromatogrames (one from FID, other from TCD) is made as follows:

- ammonia content, % = from TCD chromatogram
- monoethylamine cont., % (MEA) = MEA content from FID chromatogram x organic content /100
- acetone content, % = acetone cont. from FID chromatogram x organic cont. /100
- isopropyl alcohol content, % (AIP) = AIP cont. from FID chromatogram x organic content /100
- diisopropylamine content, % (DIPA) = DIPA cont. from FID chromatogram x organic content /100
- other amines content, % = other amines cont. from FID chromatogram x organic content /100
- total chemical impurities content, % = (ammonia + monoethylamine + acetone + isopropyl alcohol + diisopropylamina + other amines) content, %

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