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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

DIMETHYLAMINE, TECHNICAL — SPECIFICATION

(*First Revision* of IS 8874)

(ICS 71.080.30)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for Comments: **5 April 2024**

FOREWORD

(*Formal clauses will be added later*)

Dimethylamine is used in the manufacture of rayon tyre cord, styrene-butadiene rubber (SBR), acetone formaldehyde resins and nitro-compounds. It is also used in the tanning of leather, insecticides, soil disinfectants and fungicides, drugs and pharmaceuticals, ion-exchange resins, acrylic polishes, resin curing agents, weedicides, drilling of oil wells and in the catalyst for reaction polymerization.

This standard was originally published in 1977. In this (*first*) revision, the reference clause with updated cross reference standards have been incorporated. The requirement table has been bifurcated into two tables stating the requirements for anhydrous form and solution form, separately. Also, the sampling procedure has been modified, based on the type of material prescribed.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022. 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for dimethylamine, technical.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are

encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water- specification (*fourth revision*)
IS 4905 : 2015 / ISO 24153: 2009	Random sampling and randomization procedure (*first revision*)

3 TYPES

3.1 The material shall be of the following types:

a) *Type 1* – anhydrous form
b) *Type 2* – 40 percent solution.

4 REQUIREMENTS

4.1 Description

4.1.1 *Type 1*

The material shall mainly consist of dimethylamine [$(CH_3)_2$ NH] and shall be in the form of gas or colourless liquid under pressure and possess a characteristic odour of fish.

4.1.2 *Type* 2

The material shall be in the form of liquid and possess a characteristic odour of fish.

4.2 Solubility

The material shall be highly soluble in water, and fairly soluble in alcohols and glycols.

4.3 The material shall also comply with the requirements prescribed in Table 1 or Table 2, when tested according to the methods given in col 4 of Table 1 or col 4 of Table 2.

4.3.1 *Quality of Reagents*

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Dimethylamine, Technical – Anhydrous form (Type 1)
(*Clause* 4.3)

Sl No.	Characteristics	Requirement	Method of test, Ref to
(1)	(2)	(3)	(4)
i)	Dimethylamine content, percent by mass, *Min*	99.5	Annex A
ii)	Monomethylamine, percent by mass, *Max*	0.20	
iii)	Trimethylamine content, percent by mass, *Max*	0.20	
iv)	Moisture, percent by mass, *Max*	0.5	
v)	Ammonia, percent by mass, *Max*	0.01	

Table 2 Requirements for Dimethylamine, Technical – 40 percent Solution (Type 2)
(*Clause* 4.3)

Sl No.	Characteristics	Requirement	Method of test, Ref to
(1)	(2)	(3)	(5)
i)	Dimethylamine content, percent by mass, *Min*	40	Annex B
ii)	Monomethylamine, percent by mass, *Max*	0.1	
iii)	Dimethylamine content, percent by mass, *Max*	0.1	
iv)	Ammonia, percent by mass, *Max*	Traces	

5 PRECAUTIONS IN HANDLING

5.1 The material being flammable and corrosive, necessary precautions shall be taken while handling.

6 PACKING AND MARKING

6.1 Packing

6.1.1 The gaseous material shall be filled in gas cylinders under pressure. The material in the solution form shall be packed in mild steel drums.

6.1.2 Necessary safeguard against the risk arising from the storage and handling of this material shall be provided and precautions shall be taken to prevent accident by fire and explosion.

6.1.3 All containers for storage and transport of the material shall, in addition, comply with the requirements of applicable **Red Tariff No. for Rules and Rates for Conveyance by Rail of Explosives and Other Dangerous Goods** issued by the Indian Railways Conference Association with any additions and alterations made thereafter and the requirements laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids.

6.2 Marking

6.2.1 The containers/cylinders shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and type of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Batch number;
d) Net mass of the material in the container;
e) Month and year of manufacture; and
f) Any other statutory requirements

6.2.2 All the containers/cylinders in which the material is stored or transported shall be prominently and clearly marked:

DANGER!

EXTREMELY FLAMMABLE

HAZARDOUS LIQUID AND VAPOUR UNDER PRESSURE

LIQUID CAUSES BURNS

VAPOURS EXTREMELY IRRITATING

6.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the rules and regulations framed thereunder, and the products may be marked with the standard mark.

7 SAMPLING

7.1 The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex C.

ANNEX A

[*Table* 1, *Sl No.* (i) to (v)]

DETERMINATION OF DIMETHYLAMINE CONTENT IN ANHYDROUS FORM (TYPE 1) AND ITS IMPURITIES

A-1 GENERAL

Anhydrous dimethylamine is analyzed gas chromatographically by injecting a known volume of the gas and calculating the percentages by mass by the method of area normalization with response factors determined by injecting a standard mixture.

A-2 Apparatus

A-2.1 Gas *Chromatograph,* with thermal conductivity detectors (hot wire type).

A-2.1.1 *Column,* of stainless steel or glass, 185 cm long, 4 mm internal diameter and 6 mm external diameter packed with Porapak Q (500-842 microns) coated with 10 percent (*m/m)* of a mixture of 8.9 percent (*m/m)* tetraethylenepentamine and 1.1 percent (*m/m)* potassium hydroxide.

A-2.1.2 *Operating Parameters of Gas Chromatograph*

Column Oven Temperature	:	90 °C (isothermal)
Injection Port Temperature	:	150 °C
Detector Block Temperature	:	150 °C
Carrier Gas	:	Hydrogen with 50 ml/min flow rate
Delivery Pressure of Carrier Gas	:	137.3 kN/m^2
Bridge Current	:	200 mA
Chart Speed	:	30 cm/h

A-2.2 Potentiometric Strip Chart Recorder, full scale deflection 1 mV.

A-2.3 Syringe, 2 ml and 10 µl.

A-2.4 Sampling Bomb, stainless steel bomb 2.4 metres long and 3.75 cm diameter fitted with needle valves at both ends with 6 mm N.P.T. The bomb should be able to withstand pressure up to 1765 kN/m^2.

A-2.5 Electric Oven, thermostated, fitted inside with a stainless steel coil of 3 mm diameter with ends protruding out through holes on both the side walls of the oven.

A-3 REAGENTS

A-3.1 Ammonia, of known purity

A-3.2 Monomethylamine, of known purity

A-3.3 Dimethylamine, of known purity

A-3.4 Trimethylamine, of known purity

A-3.5 Methanol, of known purity

A-3.6 Water

A-3.7 Standard Mixture

A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water (*see* **A-3.1** to **A-3.6**) is prepared on *m/m* basis, taking *care* to see that the total vapour pressure of the mixture does not exceed 98.06 kN.

A-4 PROCEDURE

A-4.1 Check and adjust the chromatograph. Inject 1 µl of the standard mixture with the help of the hypodermic syringe. By suitably manipulating the attenuator switch, record all the peaks on the chart. Measure the area of all the individual peaks. Calculate the response factors of all the components, considering the factor to be one for monomethylamine.

A-4.1.1 *Determination of Response Factors*

A-4.1.1.1 Corresponding to each peak of the standard mixture, determine the amount of area produced by mass percent of the component.

A-4.1.1.2 Select one peak (monomethylamine) as a reference. Set its response factor (area by mass percent) equal to 1, and express all other respone factors relative to it.

A-4.2 Sample Injection

A-4.2.1 The bomb containing the sample under pressure is connected vertically to one end of the heated coil in the oven with swage lock metallic fittings. The other end (exit end) of the coil is connected with rubber tubing to a bubbler half filled with water. The exit end of the bubbler is again connected with a long rubber tubing which is taken outside the room as a vent. Now the bottom valve of the sample bomb is slowly opened. The sample gets immediately vaporized as it passes through the heated coil kept at 150°C. The vapour coming out through the outlet end of the coil is taken in a 2 ml syringe by piercing the needle through the connecting rubber tubing.

A-4.2.2 1 ml of the gaseous sample is now injected into the chromatograph and by suitably manipulating the attenuator all the peaks are recorded on the chart.

A-4.2.3 Measure the areas of all the peaks and calculate the percentage (*m/m)* with the help of response factor (*see* **A-4.1.1**).

A-5 CALCULATION OF MASS PERCENT OF COMPONENTS IN SAMPLE

A-5.1 For each peak, divide the measured area by the relative response factor to obtain corrected area.

A-5.2 Add up all the corrected areas and calculate each corrected area as a percent of the total corrected area. These percentages are the mass percentages of the components in the sample.

ANNEX B

[*Table* 2, *Sl No.* (i) to (v)]

DETERMINATION OF DIMETHYLAMINE CONTENT, IN 40 PERCENT SOLUTION (TYPE 2) AND ITS IMPURITIES

B-1 GENERAL

The strength of the particular dimethylamine in solution is determined in two stages. In the first stage total alkalinity of the solution is determined by titrating against standard acid and the alkalinity is expressed in terms of percent of the particular amine. In the second stage the impurities in the solution are determined gas chromatographically and each impurity (ammonia and amines) is expressed as the corresponding amine of which the solution is made. The sum total of these impurities is then subtracted from the total amine content to get the percent of the dimethylamine.

B-2 DETERMINATION OF TOTAL ALKALINITY (*as* DIMETHYLAMINE)

B-2.1 Reagents

B-2.1.1 *Standard Hydrochloric Acid,* 1 N.

B-2.1.2 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

B-2.2 Procedure

Take about 100 ml of water in a 250 ml conical flask and weigh. Pipette 10 ml of sample into it, keeping the tip of the pipette dipped in water while releasing the sample. Weigh it again. The difference of mass gives the mass of the sample. Titrate the contents with standard hydrochloric acid using phenolphthalein solution as indicator.

B-2.3 Calculation

$$\text{Total alkalinity } X_1 \text{ (as dimethylamine)} = \frac{V \times N \times 0.045 \times 100}{M}$$

where

V = volume, in ml, of standard hydrochloric acid used in the titration with the sample solution,
N = normality of standard hydrochloric acid, and
M = mass, in g, of the sample taken for test.

B-3 DETERMINATION OF IMPURITIES BY GAS CHROMATOGRAPHIC METHOD

B-3.1 Apparatus

B-3.1.1 *Gas Chromatograph,* with thermal conductivity detector (hot wire type).

B-3.1.1.1 *Column,* of stainless steel or glass, 185 cm long, 4 mm internal diameter and 6 mm external diameter packed with a porous polymer composed of ethylvinylbenzene and divinylbenzene (500-842 microns) coated with a 10 percent (*m/m*) of a mixture of 8.9 percent (*m/m*) tetraethylene pentamine and 1.1 percent (*m/m*) potassium hydroxide.

B-3.1.1.2 *Operating parameters of gas chromatograph*

Column temperature	:	90°C
Injection temperature	:	150°C
Detector block temperature	:	150°C
Carrier gas	:	Hydrogen
Flow rate	:	50 ml/min
Delivery pressure	:	137.3 kN/m^2
Bridge current	:	200 mA
Chart speed	:	30 cm/h

B-3.1.2 *Potentiometric strip chart recorder,* full scale deflection 1 mV.

B-3.1.3 *Syringe,* 10 µl.

B-3.2 Reagents

B-3.2.1 *Ammonia*, of known purity

B-3.2.2 *Monomethylamine,* of known purity

B-3.2.3 *Dimethylamine,* of known purity

B-3.2.4 *Trimethylamine,* of known purity

B-3.2.5 *Methanol*, of known purity

B-3.2.6 *Water*

B-3.2.7 *Standard Mixture*

A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water (*see* **A-3.2.1** to **A-3.2.2**) is prepared on *m/m* basis, preferably in concentration similar to that expected in the sample, taking care to see that the total vapour pressure of the mixture does not exceed 98.06 kN/m^2.

B-3.3 Procedure

B-3.3.1 Check and adjust the gas chromatograph. Inject 1 µl of the standard mixture with the help of the syringe. By suitably manipulating the attenuator switch, record the peaks on the chart and measure the area of the individual peaks.

B-3.3.2 Under identical conditions, 1 µl of the sample is injected and peak area measurement is done for call individual peaks as in the case of standard mixture.

B-3.4 Elution Order

Elution order of the component is ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water.

B-3.5 Calculation

B-3.5.1 $$Ps = \frac{A_s \times P_{std} \times S_1}{A_{std} \times S_2}$$

where

Ps = percent by mass of the component in the sample;
P_{std} = percent by mass of the component in the standard mixture;
A_s = area of the component in the sample;
A_{std} = area of the component in the standard;
S_1 = attenuation used for sample, and
S_2 = attenuation used for standard.

B-3.5.2 Conversion of ammonia, monomethylamine and trimethylamine in terms of dimethylamine:

X_1 = total alkalinity (as dimethylamine), percent by mass, (*see* **B-2.3**);
X_2 = ammonia content (NH_3) in terms of dimethylamine, percent by mass, in the sample (*see* **B-3.5.1**) = percent $NH_3 \times \frac{45}{17}$;
X_3 = monomethylamine (MMA) content in terms of dimethylamine, percent by mass, in the sample (*see* **B-3.5.1**) = percent MMA $\times \frac{45}{31}$; and
X_4 = trimethylamine (TMA) content in terms of dimethylamine, percent by mass, in the sample (*see* **B-3.5.1**) = percent TMA $\times \frac{45}{59}$.

B-3.5.3 *Dimethylamine Content*, percent by mass = $X_1 - (X_2 + X_3 + X_4)$

ANNEX C
(*Clause* 7.1)
SAMPLING OF DIMETHYLAMINE

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 The sampling instrument shall be clean and dry.

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.4 The samples shall be placed in suitable, clean, dry, airtight, metal, or dark or amber glass containers on which the material has no action.

C-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

C-1.7 Samples shall be stored in the dark.

C-2 SAMPLING INSTRUMENT

C-2.1 For Gas Samples

If the material is a gas, the sample should be taken by glass van syringe. After sampling of the material, the glass van syringe needle tip should be closed by rubber septum.

C-2.2 For Liquid Samples

C-2.2.1 The following forms of sampling instrument may be used:

a) Sampling bottle or can, for taking samples from tanks or drums; and

b) Sampling tube, for taking samples from bottles or small containers.

C-2.2.1.1 *Sampling Bottle or Can*, consists of a weighed glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 1). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

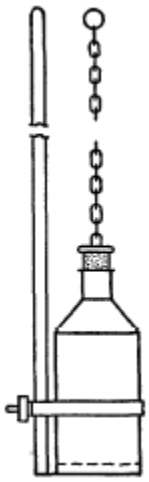

FIG. 1 SAMPLING BOTTLE OR CAN

C-2.2.1.2 *Sampling Tube*, made of metal or thick glass is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

NOTE — For small containers, the size of the sampling tube may be altered suitably.

C-3 SCALE OF SAMPLING

C-3.1 For Cylinders and Drums

Each cylinder or drum shall be sampled separately.

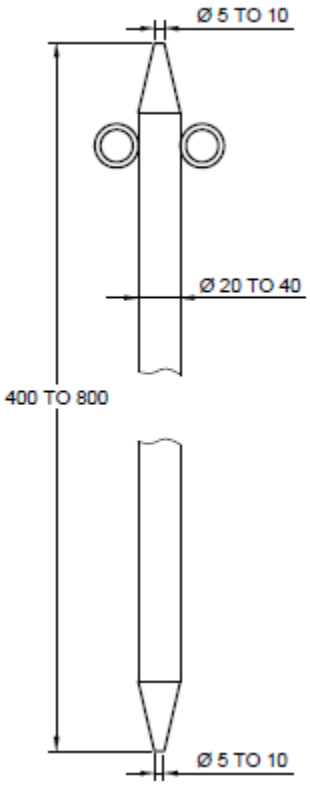

FIG. 2 SAMPLING TUBE

C-3.1.1 *Lot*

In any consignment, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

C-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with co1 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING
(*Clause* C-3.2)

Sl No.	**Lot Size** (*N*)	**No. of Containers to be Selected** (*n*)
(1)	(2)	(3)
i.	up to 15	3
ii.	16 to 40	4

iii.	41 to 65	5
iv.	66 to 110	7
v.	111 and above	10

NOTE – Where the size of the lot is three or less, all the containers shall be sampled.

C-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS 4905 for guidance to random selection procedures.

C-4 COMPOSITE SAMPLE

C-4.1 As far as possible, samples from a container or drum should be drawn during the operation of filling. In that case equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material about 1500 ml collected shall be thoroughly mixed and divided into 3 equal portions, one for the purchaser, another for the supplier and the third for the referee.

C-4.2 All the test samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

C-4.3 Tests for the determination of all the requirements given in this specification shall be performed on the test sample obtained in **C-4.1.**

C-5 CRITERIA FOR CONFORMITY

C-5.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed under **4**.