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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

N, N-DIMETHYLANILINE – SPECIFICATION

(*Second Revision* of IS 8111)

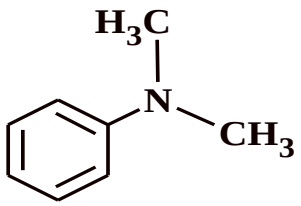
(ICS 71.080.30)

Dye Intermediates Sectional Committee, PCD 26	Last date for Comments 05 January 2024

FOREWORD

(*Formal clause to be added later*)

Dimethylaniline ($C_8H_{11}N$) is an important dye intermediate which finds extensive use in the manufacture of dyes, paper sensitizers, etc. It has the following structural formula:



N, N-Dimethylaniline
(Molecular mass 121)
(CAS No. 121-69-7)

This Standard was first published in 1976 and subsequently revised in 1986. The first revision was taken up to modify the requirement of freezing point and the test method for assay. GLC method was also introduced for determination of monomethylaniline content. In this (*second*) revision, Gas Chromatography method for determination of assay has been incorporated and new characteristics such as moisture content and aniline content and their requirements are added.

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as given in Annex C, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated.

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, the methods of sampling and test for N, N-dimethylaniline.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water - Specification (*fourth revision*)
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be clear light yellow coloured liquid which tends to darken on storage.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed col 4 and col 5 of Table 1.

Table 1 Requirements for N, N-Dimethylaniline
(*Clause* 3.2, 5.3.1, 5.3.2 and 6.1)

Sl. No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	Cl of IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Moisture content by KF, percent by mass, *Max*	0.2	—	**10.2**
ii)	Assay by GC[1)], percent by area, *Min*	99.0	A	—
	Or			
iii)	Assay (by titration), percent by mass, Min	98.0	B	
iv)	Impurities			
	a) Monomethylaniline content, percent by area, *Max*	0.7	A	—
	b) Aniline content, percent by area, *Max*	0.05	A	

v)	Distillation range, °C	95 percent shall distill between 191°C and 193 °C	—	**7**
vi)	Freezing point, °C, *Min*	1.8	—	**8**

[1)]When assay tested by GC, distillation range is optional requirement and in case of disputes, determination of assay by GC shall be the referee method.

4. PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trademark, if any;
c) Batch number;
d) Gross, net and tare mass;
e) Month and year of manufacture;
f) Shelf life of the material; and
g) Any other statutory requirements

4.2.2 *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.'

5. SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for assay and its impurities shall be conducted on each of the individual samples.

5.2.2 Tests for determination of remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For individual samples*

The lot shall be declared as conforming to the requirements assay and its impurities such as monomethylaniline and aniline if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For composite sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in col 4 and col 5 of Table 1.

6.2 Quality of Reagents

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

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

ANNEX A
[*Table* 1, *Sl* No. (ii) *and* (iv)]

DETERMINATION OF ASSAY, MONOMETHYL ANILINE AND ANILINE CONTENT BY GAS CHROMATOGRAPHY

A-1 GENERAL

Dimethylaniline and impurities like monomethylaniline and aniline are determined using gas chromatography (GC).

A-2 APPARATUS

A-2.1 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

A-2.1.1 *Column***,** 5 percent phenyl-methylpolysiloxane and 95 percent dimethylsiloxane phase with length 30 m, inner diameter 0.53 mm and film thickness 1.5 µm or equivalent.

A-2.1.2 Gas Chromatography Parameters

Carrier Gas	:	Nitrogen
Flow rate of carrier gas	:	2.0 ml/min
Fuel gas and flow rate	:	Nitrogen: 25 ml/min; Hydrogen: 40 ml/min; Air: 400 ml/min
Injection volume	:	0.2 µl
Run Time	:	17.0 min
Split	:	1 :10
Detector	:	FID

A-2.1.3 Temperature programme of oven, detector and injector are given below:

Injector temperature	Detector temperature	Oven		
		Temperature, °C	Hold time, min	Ramp rate, °C/min

280°C	290°C	135	7	—
		250	5	25

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-3 REAGENTS

A-3.1 N, N-Dimethylaniline, Reference standard

A-3.2 Monomethylaniline, Reference standard

A-3.3 Aniline, Reference standard

A-4 PROCEDURE

A-4.1 Standard Analysis

Inject standard monomethylaniline and aniline, continuing the chromatogram up to the end of the temperature program.

A-4.2 Sample Analysis

Inject sample, continuing the chromatogram up to the end of the temperature program.

A-5 PEAK TIME

N,N-dimethylaniline : 7.6 min

Monomethylaniline : 6.8 min

Aniline : 5.3 min

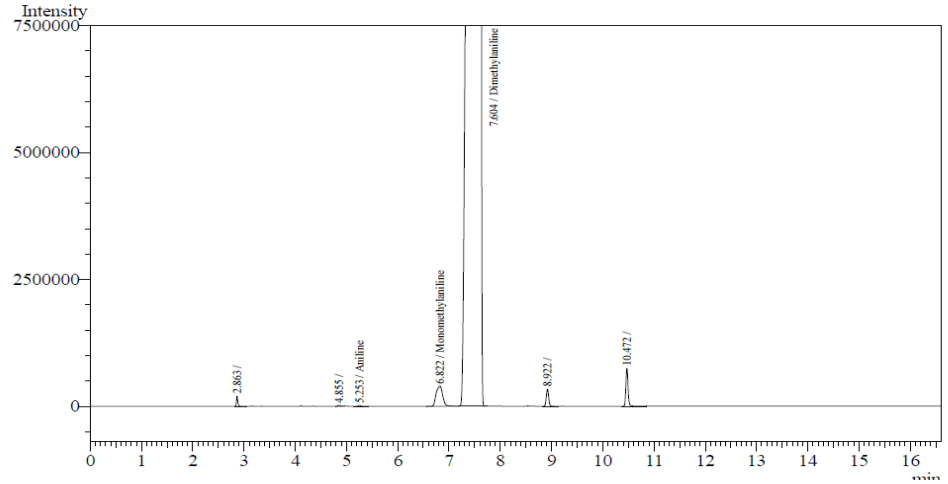


FIG 1 TYPICAL CHROMATOGRAPH

TABLE 2 TYPICAL PEAK TIME

Component	Retention time	Area	Area percent
	2.86	374059	0.05
	4.86	48245	0.01
Aniline	5.25	41958	0.01
Monomethylaniline	6.82	3493462	0.45
N,N-Dimethylaniline	7.60	777256524	99.07
	8.92	1045326	0.13
	10.47	2292478	0.29
		784552052	100.00

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to N,N-dimethyl aniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with standard N,N-Dimethyl aniline

$$\text{N,N-Dimethylaniline, percent by area} = \frac{\text{N,N−Dimethyl aniline Peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

A-6.2 Similarly, contents of monomethylaniline and aniline shall be calculated.

ANNEX B

[*Clause* 5.1 *and Table* 1, *S. No*. (iii)]

ASSAY (BY TITRATION) FOR N, N-DIMETHYLANILINE

B-1. ASSAY

B-1.1 Reagents

B-1.1.1 *Acetic Acid*

B-1.1.2 *Acetic Anhydride*

B-1.1.3 *Methyl Violet Indicator* — Dissolve 30 mg of methyl violet in 100 ml of monochlorobenzene.

B-1.1.4 *Acetous perchloric acid* — 0.1 N. Mix 8.5 ml of 72 percent perchloric acid (AR grade) in 500 ml of glacial acetic acid in 1 000-ml volumetric flask. Add 20 ml of pure acetic anhydride (AR grade) and swirl the contents of the flask to ensure thorough mixing. Make up to the mark with glacial acetic acid and allow to stand overnight to ensure complete reaction of the acetic anhydride with the water present.

B-1.1.4.1 Standardization of acetous perchloric acid — Weigh accurately about 0.5 g potassium hydrogen phthalate (AR grade) in 100 ml beaker and add about 50 ml glacial acetic acid. Warm until dissolved and cool to room temperature. Add a few drops of methyl violet indicator and titrate the solution with Perchloric acid. The colour changes from violet to blue. A very sharp end point is obtained by potentiometric titration using a glass and either a calomel or silver/silver chloride electrode.

NOTES

1 Crystal violet indicator may also be used.

2 The acetous perchloric acid may also be standardized by 0.1 N sodium acetate solution with the same procedure as above.

B-1.2 Procedure - Weigh accurately 0.3 g of the material in a 250 ml beaker and dissolve in 50 ml of glacial acetic acid. Add 3 drops of methyl violet indicator. Run in the acetous perchloric acid from a burette, while stirring, until the colour changes from violet to green. A blank titration may be made with 50 ml glacial acetic acid in the presence of the indicator and a correction applied, if necessary.

B-1.3 Calculation

Assay, percent by mass = $\frac{V \times N \times M_1}{M_2 \times 10}$

where

V = Volume in ml of acetous perchloric acid used,
N = Normality of acetous perchloric acid,
M_1 = Molecular mass of amine, and
M_2 = Mass in g of sample taken for the test.

ANNEX C
(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s)	**:**			
Signal Word	**:**	**Danger**	**Health Hazard**	**ENVIROMENTAL HAZARD**
Hazard Statement	**:**	Toxic if swallowed, in contact with skin or if inhaled. Suspected of causing cancer. Toxic to aquatic life with long-lasting effects.		
Precautionary Statement:		Avoid release to the environment. Wear protective gloves / protective clothing / eye protection / face protection / hearing protection. If swallowed: Immediately call a POISON CENTER / doctor. If inhaled: Call a POISON CENTER / doctor if you feel unwell. Store locked up. Dispose of contents / container in accordance with local / regional / national /international regulations.		