BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

1,3-डाइनिट्रोबेंजीन – विशिष्टि

(IS 8399 का पहला पुनरीक्षण)

Draft Indian Standard

1, 3 – DINITROBENZENE — SPECIFICATION

(*First Revision of* IS 8399)

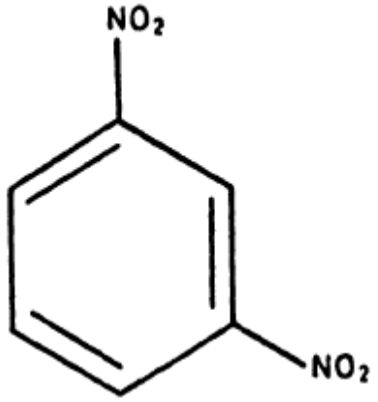
(ICS 71.080.30)

Dye Intermediates Sectional Committee, PCD 26	Last date for Comments 1st July 2024

FOREWORD

(*Formal clauses to be added later*)

1,3-Dinitrobenzene ($C_6H_4O_4N_2$), also known as *m*-dinitrobenzene, is an important dye intermediate used in the manufacture of dyestuffs. It has the following structural formula:



1, 3-Dinitrobenzene
Molecular Mass 168.1
CAS No. 99-65-0

This standard was first published in 1976. In this (first) revision, determination of assay and impurities content by gas chromatography has been modified. Two new characteristic that are moisture content and *p*H along with their requirements has been added. Requirement of matter insoluble in methanol have been deleted. An amendment has also been incorporated.

The containers in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as mentioned at Annex D, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines were 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 and the methods of sampling and test for 1, 3-Dinitrobenzene.

2 REFERENCES

The following Indian 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 agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given 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 for sampling and tests for dye intermediates *(first revision)*

3. REQUIREMENTS

3.1 Description

The material shall be in the form of clear yellow liquid, free from extraneous matter.

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

TABLE 1 REQUIREMENTS FOR 1, 3-DINITROBENZENE

(*Clauses* 3.2, 5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of tests, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay by GC, percent area, *Min*	99.70		—
ii)	Impurities			
	1,4-dinitrobenzene Content by GC, percent area, *Max*	0.20	A	—
	1,2- dinitrobenzene Content by GC, percent area, *Max*	0.20		—
	Nitrobenzene Content by GC, percent area, *Max*	0.05		—
iv)	Moisture Content by Karl Fischer, percent by mass, *Max*	0.50	B	IS 2362
v)	*p*H, *Min*	6.5	C	—
vi)	Crystallization Point[1)], °C, *Min*	87.5	—	**8** of IS 5299

[1)] Crystallization point is Optional Requirement.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in galvanized iron drums (*see* IS 2552) or tanker 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 trade-mark, if any;
c) Batch number;
d) Gross, net and tare mass;
e) Batch number, month and year of manufacturing;
f) Shelf life of the material; and
g) Any other statutory requirement.

'STRONG BLOOD POISON. CAUSES CYANOSIS, ABSOLUTELY AVOID ANY CONTACT WITH SKIN OR DIRECT INHALATION OF DUST OR FUMES.'

4.2.2 For supplies of material in bulk, a test certificate containing the details mentioned at **4.2.1** shall be provided for each consignment.

4.2.3 *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.

5 SAMPLING

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

5.2 Number of Tests

5.2.1 Tests for the determination of assay, impurities, crystallization point, *p*H and moisture content shall be conducted on each of the individual samples.

5.3 Criteria for Conformity

5.3.1 The lot shall be declared as conforming to the requirements of assay, impurities, crystallization point, *p*H and moisture content of each if the individual result satisfies the relevant requirements given in Table 1.

6 TEST

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col (4) and (5) of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed 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*.(i) and (ii)]
DETERMINATION OF ASSAY AND IMPURITIES CONTENT BY GAS CHROMATOGRAPHY

A-1 GENERAL

Determination of assay and content of impurities by Gas Chromatography instrument through area percent calculation.

2 APPARATUS

A-2 APPARATUS

A-2.1 Analytical Balance

A-2.2 Volumetric Flask

A-2.3 Beaker

A-2.4 Pipette

A-2.5 Sonicator

A-2.6 Micro Syringe

A-2.7 Water bath

A-2.8 Gas Chromatograph — Any gas chromatograph equipped with a flame ionization detector (FID).

A-2.8.1 *Column* — (14% cyanopropyl-phenyl)-methylpolysiloxane column with length 30m, inner diameter 0.25mm and film thickness 0.25µm or equivalent.

A-2.5.2 *Gas Chromatography Parameters*:

Carrier gas	: Nitrogen
Injector temperature	: 275 °C

Column oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	100	2.0

Pressure : 95.8 kPa

Hydrogen flow : 30 ml/min

Air flow : 400 ml/min

Column flow :1.0 ml/min

Split ratio : 1:30

Detector type : FID

Detector temperature : 275 °C

Injection volume : 1.0 µl

Run time : 30.0 min

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 REAGENT

A-3.1 Acetone — Solvent

A-4 PROCEDURE

Take 1.0 g of 1,3-Dinitrobenzene (sample) and make up to 10 ml with acetone. Dissolve properly and take 1.0 µl sample in micro syringe. Confirm there are no air bubbles in the syringe and inject the sample and allow the run to complete run time.

NOTE — The weights and volumes given are the recommended amounts for routine quantitative analysis. Alternative amounts may be used, provided that the final concentrations remain the same.

A-5 PEAK TIME

1, 4-Dinitrobenzene : 16.13 min

1, 3-Dinitrobenzene : 16.77 min

1, 2-Dinitrobenzene : 17.75 min

Nitrobenzene : 10.30 min

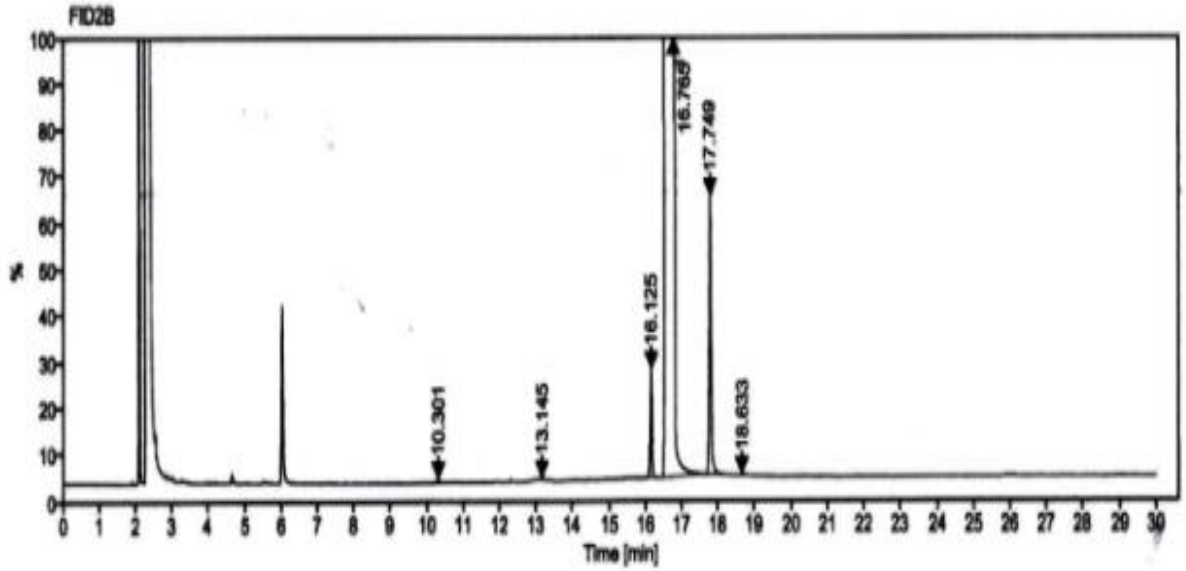


FIG. 1 A TYPICAL CHROMATOGRAM

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to 1,3-Dinitrobenzene 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 1,3-Dinitrobenzene.

$$\text{Assay, percent by area} = \frac{\text{1,3−Dinitrobenzene peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

A-6.2 Similarly**,** 1,2-Dinitrobenzene, 1,4-Dinitrobenzene and nitrobenzene content shall be calculated.

ANNEX B

[*Table* 1, *Sl. no.*(iv)]

DETERMINATION OF 1,3- DINITROBENZENE MOISTURE CONTENT BY KARL FISCHER

B-1 REAGENTS

B-1.1 Karl Fischer reagent

B-1.2 Methanol Dried

B-2 APPARATUS

B-2.1 Karl Fischer Moisture Analyzer

B-2.2 Dry Heating Block

B-2.3 Analytical Balance

B-3 PROCEDURE

Add approximately 40 ml methanol in titration vessel and stir with magnetic stirrer. Now, add Karl Fischer reagent to complete the neutralization of methanol. Now, enter sample details in the instrument and melt the sample, if required. After that, weigh 2.0 g of solid sample (2 ml, if liquid sample) and add in the titration vessel and press START to continue titration. Ensure proper and complete addition of sample in vessel. Once the sample is added, the instrument automatically starts addition of KF reagent in the titration vessel to titrate moisture content present in sample. Instrument will stop adding KF reagent automatically once it reaches the electrometric endpoint. Note down the burette reading.

B-4 CALCULATION

$$\text{Moisture Content, percent w/w} = \frac{\text{V} \times \text{ F} \times 100}{W \times 1000}$$

$$\text{Moisture Content, in ppm} = \text{Moisture (percent)} \times 1000$$

where

V = volume of karl fischer reagent consumed, in ml:
F = karl fischer reagent factor, in mg/ml and;
W = weight of sample taken, in g.

ANNEX C
[*Table* 1, *Sl. no.*(iv)]
DETERMINATION OF *p*H OF 1,3- DINITROBENZENE

C-1 APPARATUS

C-1.1 pH meter

C-1.2 Beaker

C-3 PROCEDURE

Take about 30 ml of 1, 3-Dinitrobenzene (sample) into 100 ml separating funnel. Add about 50 ml Distilled water (pH should be 7.00). Mix well for about 3.0 min and allow to separate the layer. Collect the aqueous layer into 50 mL beaker and measure the pH at 25°C temperature.

ANNEX D

(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :	
Signal Word :	**WARNING**
Hazard statement(s) :	H300+H310+H330 - Fatal if wallowed, in contact with skin or if inhaled. H373 - May cause damage to organs through prolonged or repeated exposure. H410 - Very toxic to aquatic life with long lasting effects.
Precautionary Statements	P260 - Do not breathe dust. P262 - Do not get in eyes, on skin, or on clothing. P264 - Wash hands thoroughly after handling. P270 - Do not eat, drink or smoke when using this product. P271 - Use only outdoors or in a well-ventilated area. P273 - Avoid release to the environment. P280 - Wear protective gloves, protective clothing. P284 - Wear respiratory protection. P301+P310 - IF SWALLOWED: Immediately call a doctor, a POISON CENTER. P302+P352 - IF ON SKIN: Wash with plenty of soap and water. P304+P340 - IF INHALED: Remove person to fresh air and keep comfortable for breathing. P310 - Immediately call a doctor, a POISON CENTER. P314 - Get medical advice/attention if you feel unwell. P320 - Specific treatment is urgent (see supplemental first aid instruction on this label). P321 - Specific treatment (see supplemental first aid instruction on this label). P330 - Rinse mouth. P361+P364 - Take off immediately all contaminated clothing and wash it before reuse. P391 - Collect spillage. P403+P233 - Store in a well-ventilated place. Keep container tightly closed. P405 - Store locked up. P501 - Dispose of container, contents to hazardous or special waste collection point, in accordance with local, regional, national and/or international regulation.