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Draft Indian Standard
3-NITROTOLUENE — SPECIFICATION
(First Revision of IS 8398)
(ICS 71.080.30)

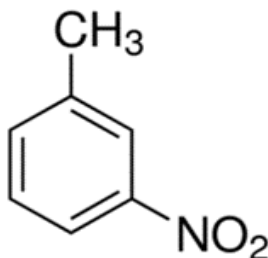
Dye Intermediates Sectional Committee,
PCD 26

Last date for comment
06 May 2024

FOREWORD

(Formal clauses to be added later)

3-Nitrotoluene (C₇H₇O₂N) is an important intermediate used in the manufacture of dyestuffs. It is obtained by nitration of toluene. It is also known as *m*-nitrotoluene and 3-nitro-1-methyl benzene. It is represented by the following structural formula:



3-NITROTOLUENE
Molecular mass 137
CAS Number 99-08-1

This standard was originally published in 1976. In this (*first*) revision, test methods for determination of 3-Nitrotoluene content (assay) and impurities content that are 2-Nitrotoluene and 4-Nitrotoluene and other impurities by gas chromatography have been modified. A new characteristic that is moisture content has been added. The requirement of Di-nitrotoluene and pH have been deleted.

The bags or 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 C, 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 testing for 3-Nitrotoluene.

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 indicated below:

<i>IS No.</i>	<i>Title</i>
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 in the form of clear colourless to yellow colour 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 in col 4 and 5 of Table 1.

TABLE 1 REQUIREMENTS FOR 3-NITROTOULENE
(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, <i>Min</i>	99.50	A	—
ii)	2-Nitrotoulene by GC, percent area, <i>Max</i>	0.10		
iii)	4-Nitrotoulene by GC, percent area, <i>Max</i>	0.20		
iv)	Other impurities by GC, percent area, <i>Max</i>	0.30		
v)	Moisture Content by Karl Fischer, percent, <i>Max</i>	0.10	B	—
vi)	Crystallization point ¹⁾ , °C, <i>Min</i>	15.7	—	7 of IS 5299

¹⁾ Crystallization point is optional requirement to be tested.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in galvanized iron drums and tanker or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

4.2 Marking

4.2.1 Each container 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) Month and year of manufacturing;
- f) Shelf life of the material; and
- g) Any other statutory requirement.

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 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 assay, 2-nitrotoluene, 4-nitrotoluene, other impurities, moisture content and crystallizing point shall be conducted on the individual sample.

5.3 Criteria for Conformity

5.3.1 The lot shall be declared as conforming to the requirements of assay, 2-nitrotoluene, 4-nitrotoluene, other impurities, moisture content and crystallizing point, if each of the individual test results satisfies the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the method prescribed in col 4 and col 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* 1070) 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), (ii), (iii) and (iv)]

DETERMINATION OF ASSAY AND IMPURITIES CONTENT BY GAS CHROMATOGRAPHY

A-1 GENERAL

Determination of 3-Nitrotoulene content (assay) and content of impurities that are 2-Nitrotoulene and 4-Nitrotoulene and content of other impurities by Gas Chromatography instrument through area percent calculation.

A-2 APPARATUS

A-2.1 Analytical Balance

A-2.2 Volumetric Flask — 10 ml

A-2.3 Glass Beaker

A-2.4 Sonicator

A-2.5 Gas Chromatograph — Gas chromatograph equipped with a flame ionization detector (FID) may be used with following accessories and typical operating conditions:

A-2.5.1 Column — (14 percent cyanopropyl-phenyl)-methylpolysiloxane with length 30 m, inner diameter 0.25 mm and film thickness 1.0 µm or equivalent.

A-2.5.2 Gas Chromatography Parameters :

Carrier Gas : Nitrogen
Injector Temperature : 275 °C
Carrier Gas Pressure : 96 kpa

Column Oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	100	2
10	230	15

Hydrogen flow : 30 ml/min
Zero air flow : 400 ml/min
Column flow : 1 ml/min
Purge Flow : 3.0 ml/min
Make up gas (N₂) flow : 25 ml/min
Split Ratio : 1 : 30
Detector Type : Flame Ionization Detector (FID)
Detector Temperature : 275°C
Injection Volume : 1.0 µl
Total 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, H₂, N₂) 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 Methanol — Solvent

A-4 PROCEDURE

Take 0.5 g of 3-Nitrotoluene (sample) and make up to 10 ml with methanol. Now, dissolve properly and take 1.0 µl of sample as prepared micro syringe. Confirm there are no air bubbles in the syringe and inject the sample and allow the run to complete the run time.

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

A-5 PEAK TIME

3-Nitrotoluene	: 11.87 min
4-Nitrotoluene	: 12.18 min
2-Nitrotoluene	: 11.08 min

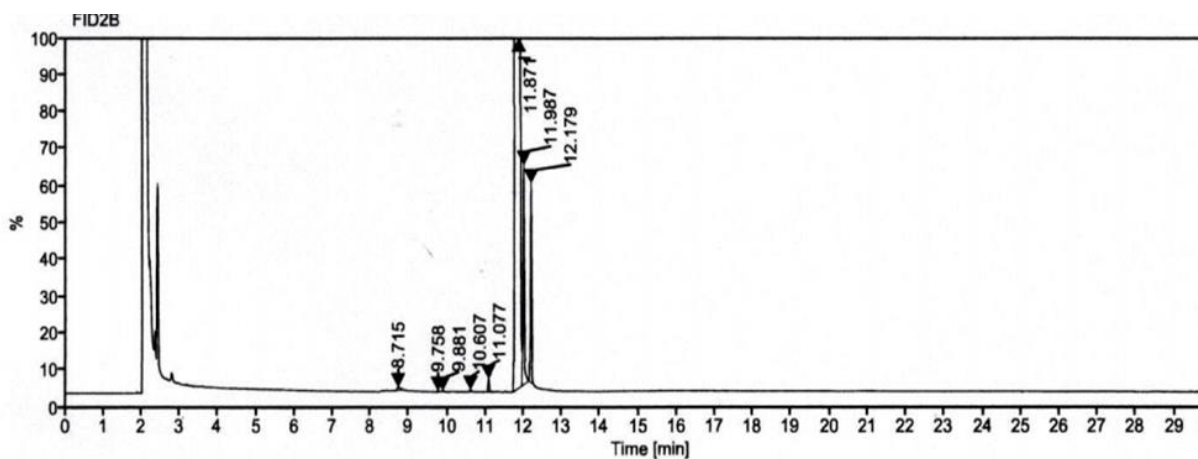


FIG 1 A TYPICAL CHROMATOGRAPH

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to 3-Nitrotoluene 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 3-Nitrotoluene.

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

A-6.2 Similarly, 2-Nitrotoulene, 4-Nitrotoulene and other impurities content shall be calculated.

ANNEX B

[Table 1, Sl No. (v)]

DETERMINATION OF 3-NITROTOLUENE MOISTURE CONTENT BY KARL FISCHER

D-1 APPARATUS

D-1.1 Karl Fischer Moisture Analyzer

D-1.2 Dry Heating Block

D-1.3 Analytical Balance

D-2 REAGENTS

D-2.1 Karl Fischer reagent

D-2.2 Methanol Dried

D-3 PROCEDURE

Add approximately 40 ml of methanol in titration vessel and stir with magnetic stirrer then add Karl Fischer reagent to complete neutralization of methanol. After that, weigh 2 g of 3-Nitrotoluene (sample) in titration vessel and dissolve it in methanol. Now, instrument automatically starts addition of Karl Fischer reagent in the titration vessel to titrate moisture content present in sample. Instrument will stop adding Karl Fischer reagent automatically once it reaches the electrometric endpoint. Note down the burette reading.

D-4 CALCULATION

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

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

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
(Foreword)

Pictograms, signal word, hazard statement and precautionary statement:

Pictogram(s)		
Signal Word	DANGER	ENVIRONMENTAL HAZARD
Hazard Statement	H301+H311+H331 - Toxic if swallowed, in contact with skin or if inhaled H411 - Toxic to aquatic life with long lasting effects.	
Precautionary Statement	Precautionary statement(s) Prevention P261 - Avoid breathing dust, fume, gas, mist, spray and vapours. 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. 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. P311 - Call doctor, a POISON CENTER. 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.	
