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Draft Indian Standard
2-NITROTOLUENE — SPECIFICATION
(First Revision of IS 8397)
(ICS 71.080.30)

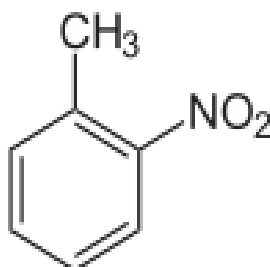
Dye Intermediates Sectional Committee,
PCD 26

Last date for comment
30th April 2024

FOREWORD

(Formal clauses to be added later)

2-Nitrotoluene (C₇H₇O₂N) is an important intermediate used in the manufacture of dyes and explosives. It is also known as 2-nitro-1-methyl benzene. It is obtained by nitration of toluene. It is represented by the following structural formula:



2-NITROTOULENE
Molecular mass 137.138
CAS Number 88-72-2

This standard was originally published in 1976. In this (*first*) revision, test methods for determination of 2-Nitrotoluene content (assay) and impurities content that are 3-Nitrotoulene and 4-Nitrotoulene 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 2-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:

IS No.	Title
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2552 : 1989	Steel drums (Galvanized and Ungalvanized) — Specification (<i>third revision</i>)
IS 5299 : 2001	Methods of Sampling and Tests for dye Intermediates (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of clear yellow color 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 2- 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>	99.7	A	—
ii)	3-Nitrotoulene, percent area, <i>Max</i>	0.20		
iii)	4-Nitrotoulene, percent area, <i>Max</i>	0.20		
iv)	Moisture Content by Karl Fischer, percent, <i>Max</i>	0.10	B	—
v)	Crystallization point ¹⁾ , ° C, <i>Min</i>	-9.5	—	7 of IS 5299

¹⁾ Crystallization point is optional requirement to be tested.

NOTE — 2-Nitrotoluene exists in two polymorphic forms, namely, alpha type crystals having crystallizing point of -9.5°C and beta type crystals having crystallizing point of -3.5°C . Generally the crystallizing point recorded is that of alpha form only. However, there are instances when there is another crystallizing point, namely, that of beta form. In such cases, the sample should be heated to its boiling point, cooled and then the crystallizing point redetermined. If the beta form persists, the crystallizing point determination should be further repeated by using seed crystals of some other sample that exists in alpha form.

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 manufacture;
- f) Shelf life of the material; and
- g) Any other statutory requirements

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, 3-nitrotoluene, 4-nitrotoluene, 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, 3-nitrotoluene, 4-nitrotoluene, 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)]

DETERMINATION OF ASSAY OF 2- NITROTOULENE BY GAS CHROMATOGRAPHY

A-1 GENERAL

Determination of 2-Nitrotoulene content (assay) and content of impurities that are 3-Nitrotoulene and 4-Nitrotoulene 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 : 120 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
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 2-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

2-Nitrotoluene	11.15
3-Nitrotoluene	11.81
4-Nitrotoluene	12.21

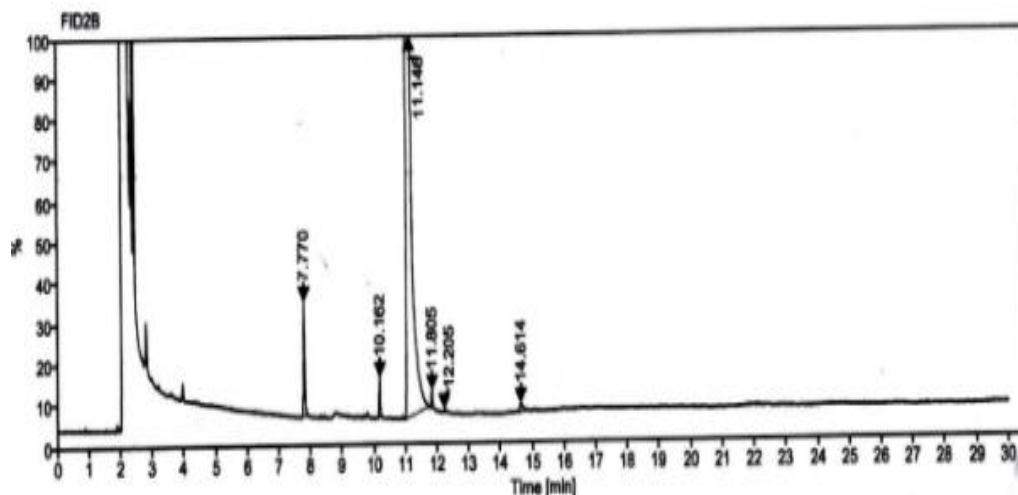


FIG 1 A TYPICAL CHROMATOGRAPH

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to 2-Nitrotoulene 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 2-Nitrotoulene.

$$\text{Assay, percent by area} = \frac{\text{2-Nitrotoulene peak area in the sample}}{\text{Sum area of all the peaks in the chromatogram}} \times 100$$

A-6.2 Similarly, 3-Nitrotoulene and 4-Nitrotoulene content shall be calculated.

ANNEX B

[Table 1, Sl No. (iv)]

DETERMINATION OF 2-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 2-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	WARNING	ENVIRONMENTAL HAZARD
Hazard Statement	H302 - Harmful if swallowed. H340 - May cause genetic defects. H350 - May cause cancer. H361f - Suspected of damaging fertility. H411 - Toxic to aquatic life with long lasting effects.		
Precautionary Statement	Precautionary statement(s) Prevention P201 - Obtain special instructions before use. P202 - Do not handle until all safety precautions have been read and understood. P264 - Wash hands thoroughly after handling. P270 - Do not eat, drink or smoke when using this product. P273 - Avoid release to the environment. P280 - Wear protective gloves, protective clothing. P301+P312 - IF SWALLOWED: Call doctor, a POISON CENTER if you feel unwell. P308+P313 - IF exposed or concerned: Get medical advice/attention. P330 - Rinse mouth. P391 - Collect spillage. 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.		
