BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

पी–नाइट्रोटोल्यूइन – विशिष्टि

(IS 3562 का दूसरा पुनरीक्षण)

Draft Indian Standard

p-NITROTOLUENE, TECHNICAL — SPECIFICATION

(*Second Revision* of IS 3562)

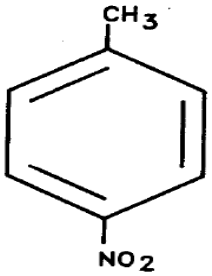
(ICS 71.080.30)

Dye Intermediates Sectional Committee, PCD 26	Last date for Comments **16th July 2024**

FOREWORD

(*Formal clauses to be added later*)

p-Nitrotoluene ($C_7H_7NO_2$) or 4-Nitrotoluene or 1-Methyl-4-Nitrobenzene is an intermediate used for the manufacture of *p*-toluidine, fuchsine and various other synthetic dyes, apart from its use in the manufacture of dinitrostilbene compounds. It is also a drug intermediate being the starting material for the preparation of *p*-nitrobenzoic acid which is used in the preparation of synthetic drugs like folic acid. It has the following structural formula:



p-NITROTOLUENE, TECHNICAL
Molecular Mass: 137
CAS no.: 99-99-0

This standard was first published in 1965 and subsequently revised in 1997. The Committee responsible for preparation of this standard felt the need to revise it to keep it in line with present industrial practices. In this (second) revision, determination of *p*-nitrotoluene content by Gas Chromatography has been updated and a new characteristic that is moisture content has

been added. Requirements of dinitrocresols content, dinitrotoluene content, unnitrated hydrocarbons content and matter insoluble in methanol 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 D, which are derived from GHS guidelines. At the time of publication, 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

This standard prescribes the requirements and methods of sampling and testing for *p*-nitro-toluene, technical.

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 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 *(fourth revision)*
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) *(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 dry yellow powder, free from lumps and extraneous substances.

3.2 The materials 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 *p*-Nitrotoluene, technical
(*Clause* 3.2, 5.3.1 and 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay by GC, percent area, *Min*	99.60		
ii)	Impurities			
	m-Nitrotoulene Content by GC, percent area, *Max*	0.15	A	—
	o-Nitrotoulene Content by GC, percent area, *Max*	0.20		
iii)	Moisture content by Karl Fischer, percent by mass, *Max*	0.10	B	IS 2362
iv)	Crystallization Point[1)], °C	50.5	C	**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 in jumbo bag or in tanker or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each bag or 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 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 Tests for assay, impurities, crystallization point and moisture content shall be conducted on each of the individual sample.

5.3 Criteria for Conformity

5.3.1 The lot shall be declared as conforming to the requirements of all tests mentioned if each of the individual test results satisfies the relevant requirements given in Table 1.

6 TESTS

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 otherwise specified, 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.

ANNEX A
[Table 1, *sl.no.* (i) and (ii)]
DETERMINATION OF ASSAY OF *p*-NITROTOLUENE, TECHNICAL BY GAS CHROMATOGRAPHY

A-1 GENERAL

Determination of assay shall be carried out by Gas Chromatography instrument through area percent calculation.

A-2 APPARATUS

A-2.1 Analytical balance

A-2.2 Volumetric flask

A-2.3 Glass beaker

A-2.4 Sonicator

A-2.5 Water bath

A-2.6 Gas Chromatograph — Any gas chromatograph equipped with a flame ionization detector (FID).

A-2.6.1 *Column*, (14 percent cyanopropyl-phenyl)-methylpolysiloxane with length 30 m, inner diameter 0.25 mm and film thickness 1.0 µm or equivalent.

A-2.6.2 *Gas Chromatography Parameters*:

Carrier gas : Nitrogen

Injector Temperature : 275 °C

Column oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	100	2.0
10	230	15

Pressure : 100 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 Methanol — Solvent

A-4 PROCEDURE

Take 0.5 g of (sample) and make up to 10 ml with methanol. 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

m-Nitrotoluene 11.78 min

p-Nitrotoluene 12.26 min

o-Nitrotoluene 11.04 min

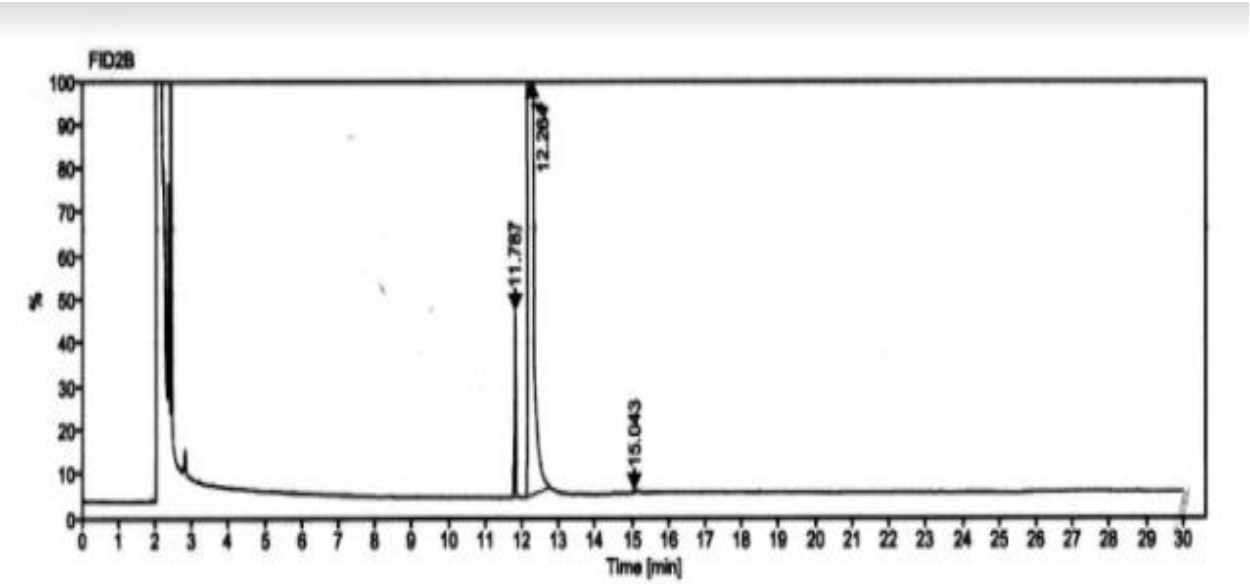


FIG. 1 A TYPICAL CHROMATOGRAM

A-6 CALCULATION

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

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

A-6.2 Similarly**,** *m*-Nitrotoulene and *o*-Nitrotoulene content shall be calculated.

ANNEX B

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

DETERMINATION OF *p*-NITROTOULENE 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 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.

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, *s1*. *no*. (iii)]

DETERMINATION OF *p*-NITROTOULENE CRYSTALLIZATION POINT

C-1 Take about 20 g of sample into a clean and dry test tube. Heat it up to 50 °C to 60 °C until material will melt. Remove the test tube from the water bath. Now take a calibrated thermometer and dip it into the test tube by stirring with thermometer. Whenever the material starts to crystallize, then the temperature of solidification will be constant at temperature. That temperature is the crystallization point of the material.

ANNEX D

(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s)			
Signal Word	**WARNING**	**HEALTH HAZARD**	**ENVIRONMENTAL HAZARD**
Hazard statement(s)	H301+H311+H331— Toxic if swallowed, in contact with skin or if inhaled. H341 — Suspected of causing genetic defects. H351 — Suspected of causing cancer. H373 — May cause damage to organs through prolonged or repeated exposure. H411 — Toxic to aquatic life with long lasting effects.		
Precautionary Statement(s)	P260 — Do not breathe dust. 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