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भारतीय मानक मसौदा

पी-टोल्यूडीन – विशिष्टि

(IS 5647 का दूसरा पुनरीक्षण)

Draft Indian Standard

p-TOLUIDINE — SPECIFICATION

(*Second Revision* of IS 5647)

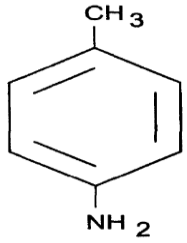
(ICS 71.080.30)

Dye Intermediates Sectional Committee, PCD 26	Last date for Comments **07th June 2024**

FOREWORD

(*Formal clauses to be added later*)

p-Toluidine is also known as 4-amino toluene. It is used as an intermediate in the manufacture of dyes. It is carcinogenic in nature. It has the following structural formula:



p-Toluidine /4- Toluidine
Molecular mass 107.15
CAS number 106-49-0

This standard was first published in 1970 and subsequently revised in 2003. In this (second) revision, determination of assay and impurities content by gas chromatography has been incorporated. A new characteristic that is moisture content and its requirement has been added. Requirement of matter

insoluble in hydrochloric acid and impurities content of *p*-nitrotoluene by thin layer chromatography have been deleted.

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

This standard prescribes the requirements and methods of sampling and tests for *p*-Toluidine.

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*
1070 : 2023	Reagent grade water — Specification *(fourth revision)*
2552 : 1989	Steel Drums (Galvanized and Ungalvanized) — Specification (*third revision*)
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 a clear colourless to light yellow color liquid, free from extraneous substances.

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 *p*-TOLUIDINE

(*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*	99.50		—
ii)	Impurities		A	
	m-Toluidine Content by GC, percent area, *Max*	0.15		—
	o-Toluidine Content by GC, percent area, *Max*	0.20		—
	Or			
iii)	Impurities			
	o-Toluidine, percent by mass, *Max*	0.2	B	—
	m-Toluidine, present by mass, *Max*	0.2		—
iv)	Moisture Content by Karl Fischer, percent by mass, *Max*	0.30	C	IS 2362
v)	Crystallization Point[1)], °C	43	—	**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) 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 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 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 content 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 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* — Polyethylene glycol phase with length 50 m, inner diameter 0.32 mm and film thickness 0.25 µm or equivalent.

A-2.5.2 *Gas Chromatography Parameters*:

Carrier gas	: Hydrogen
Injector temperature	: 250 °C

Column oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	100	8.0
10	225	10

Pressure : 42.19 kPa

Hydrogen flow : 30 ml/min

Air flow : 400 ml/min

Column flow : 0.6 ml/min

Split ratio : 1:50

Detector type : FID

Detector temperature : 250 °C

Injection volume : 1.0 µl

Run time : 30.5 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 1.0 g of *p*-Toluidine (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

p-Toluidine: 16.60 min

m-Toluidine: 16.86 min

o-Toluidine: 16.45 min

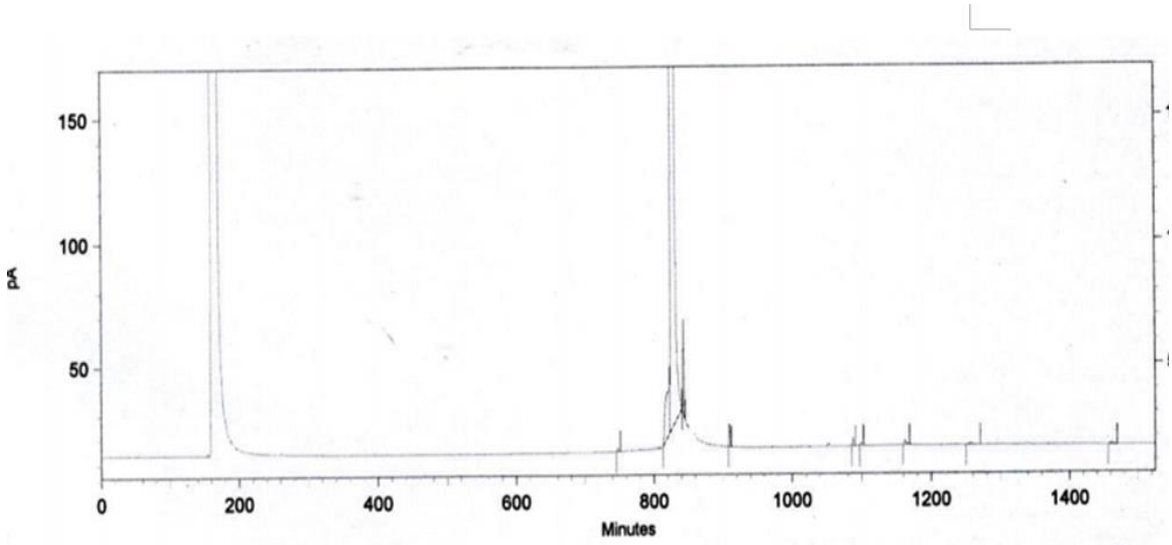


FIG. 1 A TYPICAL CHROMATOGRAM

A-6 CALCULATION

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

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

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

ANNEX B

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

THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

B-1 GENERAL

Impurities are determined by thin layer chromatography. Reference may be made to IS 5299 for details of TLC test method to be followed. However, necessary details of test conditions are given below for guidance only:

Product name	*p*-Toluidine (2 sets)
Sample solution (on 100 percent basis)	2 percent in acetone

Application/volume for spotting	10 µl (for sample) 2 µl and 4 µl (for impurities)
Standard	Reference standard
Test substance for impurities	Set I Set II (1) *m*- Toluidine (2) *o*- Toluidine (0.05 percent solution in acetone)
Plate type	i) Silica gel G ii) Silica gel G HF 254
Eluent	i) Toluene : Ethanol ii) Petroleum ether (40:60) (100 :2) (Acetic acid atmosphere)
Elution time	i) 1.5 h ii) 230 min.
Temperature	25±5 °C
Detection spray	i) [1)] $SnCl_2$ solution + PDAB solution ii) UV – 254 light
Evaluation	Semi quantitative
Approximate R_f value — Main band	*p*- Toluidine : R_f 0.4
— Impurities	*o*- Toluidine : R_f 0.7
	m- Toluidine : R_f 0.6

[1)] $SnCl_2$ solution : 10 percent solution in (1:1) water + 5N HCl

PDAB solution : *p*-Dimethylamino benzaldehyde 1 percent solution in Methanol : water : 5N HCl. (1 :0.5:0.5)

ANNEX C

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

DETERMINATION OF *p*-TOLUIDINE MOISTURE CONTENT BY KARL FISCHER

C-1 REAGENTS

C-1.1 Karl Fischer reagent

C-1.2 Methanol Dried

C-2 APPARATUS

C-2.1 Karl Fischer Moisture Analyzer

C-2.2 Dry Heating Block

C-2.3 Analytical Balance

C-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.

C-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 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.
H317 - May cause an allergic skin reaction.
H319 - Causes serious eye irritation.
H351 - Suspected of causing cancer.
H400 - Very toxic to aquatic life.

Precautionary Statements	P201 - Obtain special instructions before use. P202 - Do not handle until all safety precautions have been read and understood. P261 - Avoid breathing dust, fume, gas, mist, spray, 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. P272 - Contaminated work clothing should not be allowed out of the workplace. P273 - Avoid release to the environment. P280 - Wear protective gloves, protective clothing, eye protection, face protection. P301+P310 - IF SWALLOWED: Immediately call a POISON CENTER, a doctor. 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. P305+P351+P338 - IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. P308+P313 - IF exposed or concerned: Get medical advice/attention. P311 - Call a POISON CENTER, doctor. P321 - Specific treatment (see supplemental first aid instruction on this label). P330 - Rinse mouth. P333+P313 - If skin irritation or rash occurs: Get medical advice/attention. P337+P313 - If eye irritation persists: Get medical advice/attention. P361+P364 - Take off immediately all contaminated clothing and wash it before reuse. P362+P364 - Take off 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.