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भारतीय मानक मसौदा

ओ-टोल्यूडीन – विशिष्टि  
(IS 5649 का तीसरा पुनरीक्षण)

*Draft Indian Standard*

***o*-TOLUIDINE — SPECIFICATION**

*(Third Revision of IS 5649)*

(ICS 71.080.30)

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Dye Intermediates Sectional Committee,  
PCD 26

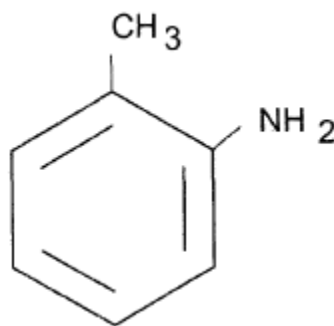
Last date for Comments  
1<sup>st</sup> July 2024

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FOREWORD

*(Formal clauses to be added later)*

*o*-Toluidine is also known as 2-aminotoluene and it is used as an intermediate in the manufacture of dyes. It is carcinogenic in nature. It has the following structural formula:



*o*-Toluidine / 2-Toluidine  
Molecular Mass 107.15  
CAS No. 95-53-4

This standard was first published in 1970 and subsequently revised in 1983 and 2003. In this (third) 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, determination of purity and impurities content of *o*-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 test for *o*-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:

| <i>IS No</i>   | <i>Title</i>  |
|----------------|---|
| 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 for sampling and tests for dye intermediates ( <i>first revision</i> )      |

## **3 REQUIREMENTS**

**3.1 Description** — The material shall be in the form of clear colourless to amber 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 col (4) and (5) of Table 1.

**TABLE 1 REQUIREMENTS FOR *o*-TOLUIDINE**

(Clauses 3.2, 5.3.1 and 6.1)

| Sl No. | Characteristic  | Requirement                       | Method of tests, Ref to |         |
|--------|---|-----------------------------------|-------------------------|---------|
|        |   |                                   | Annex (4)               | IS (5)  |
| (1)    | (2)   | (3)                               |                         |         |
| i)     | Assay by GC, percent area, <i>Min</i>                       | 99.80                             | A                       | —       |
| ii)    | Impurities by Gas chromatography                            | {<br>0.10<br>0.10<br>0.05<br>0.10 |                         | —       |
|        | <i>m</i> -Toluidine Content by GC, percent area, <i>Max</i> |                                   |                         | —       |
|        | <i>p</i> -Toluidine Content by GC, percent area, <i>Max</i> |                                   |                         | —       |
|        | Aniline content   |                                   |                         | —       |
|        | Sum of other impurities                                     | 0.10                              |                         |         |
|        | <i>Or</i>   |                                   |                         |         |
| iii)   | Impurities by thin layer chromatography                     |                                   | B                       | —       |
|        | <i>p</i> -Toluidine, percent by mass, <i>Max</i>            | 0.2                               |                         |         |
|        | <i>m</i> -Toluidine, present by mass, <i>Max</i>            | 0.2                               |                         |         |
| iv)    | Moisture Content by Karl Fischer, ppm, <i>Max</i>           | 2000 ppm                          | C                       | IS 2362 |

## **4 PACKING AND MARKING**

### **4.1 Packing**

The material shall be packed in galvanized iron drums (*see* IS 2552) or tanker 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) Gross, net and tare mass;
- d) Batch number,
- 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**

Tests for assay, impurities, 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 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, *m*-Toluidine, *p*-Toluidine and aniline content shall be carried 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 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-2.8.2 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** : 0.2 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<sub>2</sub>, N<sub>2</sub>) 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 PEAK TIME**

*m*-Toluidine 16.92 min

*p*-Toluidine 16.66 min

|                     |           |
|---------------------|-----------|
| <i>o</i> -Toluidine | 16.61min  |
| Aniline             | 15.63 min |

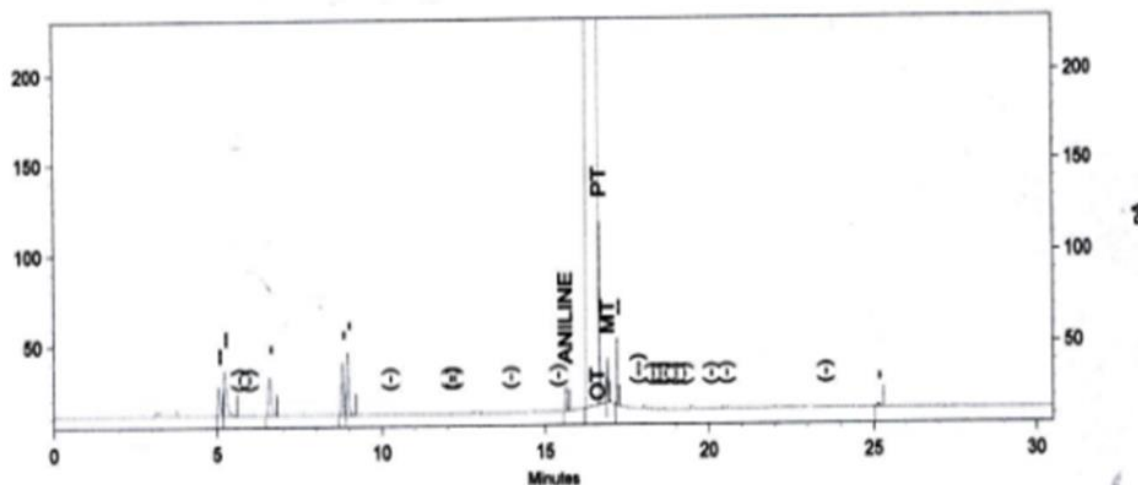


FIG. 1 A TYPICAL CHROMATOGRAM

#### A-4 CALCULATION

**A-4.1** Calculate the peak area of individual constituent pertaining to *o*-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 *o*-Toluidine.

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

**A-4.2** Similarly, *m*-Toluidine, *p*-Toluidine and aniline 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  | <i>o</i> -Toluidine (2 sets)  |
| Sample solution (on 100 percent basis)                  | 2 percent in acetone  |
| Application / sample volume for spotting                | 10 $\mu$ l (for sample)<br>2 $\mu$ l and 4 $\mu$ l (for impurities)   |
| Standard  | Reference standard  |
| Test substance for impurities                           | Set I                      Set II<br>(1) <i>p</i> - Toluidine        (2) <i>m</i> - Toluidine<br><br>(0.05 percent solution in acetone) |
| Plate type  | i) Silica gel G    ii) Silica gel G HF 254  |
| Eluent  | i) Toluene : Ethanol (98:2)<br>ii) Petroleum ether (40:60)<br>(Acetic acid atmosphere)  |
| Elution time  | i) 1.5 h    ii) 30 min  |
| Temperature   | 25 $\pm$ 5 $^{\circ}$ C   |
| Detection spray   | i) <sup>1)</sup> SnCl <sub>2</sub> solution + PDAB solution<br>ii) UV – 254 light   |
| Evaluation  | Semi quantitative   |
| Approximate R <sub>f</sub> value — Main band Impurities | <i>o</i> - Toluidine        : R <sub>f</sub> 0.7  |
|   | <i>m</i> - Toluidine        : R <sub>f</sub> 0.6  |
|   | <i>p</i> - Toluidine        : R <sub>f</sub> 0.4  |

<sup>1)</sup>SnCl<sub>2</sub> solution : 10 percent solution in (1:1) water + 5N HCl

PDAB solution : *p*-Dimethylamino benzaldehyde

1 percent solution in (1:0.5 : 0.5) Methanol : water : 5N HCl.

## ANNEX C

[Table 1, sl. no.(iii)]

### DETERMINATION OF *o*-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**

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

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

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**

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**Pictogram(s) :**



**Signal Word :**

**WARNING**

**HEALTH  
HAZARD**

**ENVIRONMENTAL  
HAZARD**

**Hazard  
statement(s) :**

H301+H331 — Toxic if swallowed or if inhaled.  
 H319 — Causes serious eye irritation.  
 H350 — May cause 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.  
 P273 — Avoid release to the environment.  
 P280 — Wear protective gloves, protective clothing, eye protection, face protection.  
 P301+P310 — **IF SWALLOWED:** Immediately call a doctor, a POISON CENTER.  
 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 doctor, a POISON CENTER.  
 P321 — Specific treatment (see supplemental first aid instruction on this label).  
 P330 — Rinse mouth.

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P337+P313 — If eye irritation persists: Get medical advice/attention.

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.

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