

**BUREAU OF INDIAN STANDARDS**

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भारतीय मानक मसौदा

**2,5-डाइक्लोरोएनिलीन — विशिष्टि**

(IS 4526 का दूसरा पुनरीक्षण)

Draft Indian Standard

**2,5-DICHLOROANILINE — SPECIFICATION**

*(Second Revision of IS 4526)*

(ICS 71.080.30)

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Dye Intermediates Sectional Committee,  
PCD 26

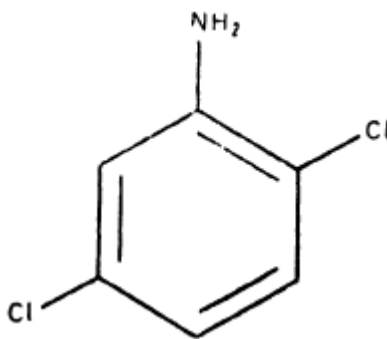
Last date for Comments  
**27<sup>th</sup> September 2025**

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**FOREWORD**

*(Formal clauses to be added later)*

2,5-Dichloroaniline (C<sub>6</sub>H<sub>3</sub>Cl<sub>2</sub>N) is used as a diazo component in the manufacture of dyestuffs of the azo series. It has the following structural formula:



**2,5-DICHLOROANILINE**

(Molecular Mass 162)

CAS No. 95-82-9

This standard was first published in 1968 and subsequently revised in 1976. In this (*second*) revision, determination of purity and impurities content namely aniline, 3,4- Dichloroaniline and *m*-Chloroaniline by gas chromatography have been incorporated. Requirement of matter insoluble in methanol have been deleted. An amendment has also been incorporated.

Requirement for marking of pictograms, signal word, hazard statement, and precautionary statement as given in Annex C, which are derived from GHS guidelines have been incorporated. At the time of publication, the latest edition of

GHS guidelines was 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 draft standard prescribes the requirements and the methods of sampling and testing for 2,5-Dichloroaniline.

## **2 REFERENCES**

The standards given below contain provisions, which through reference in this text, constitute provision of this standard. At the time of the publication, the editions indicated below were valid. All the standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

<i>IS No</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 2362 : 1993	Determination of water by karl fischer method — Test method ( <i>second 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 off white to white, free from lumps and extraneous substances flakes/ liquid.

**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 2,5-DICHLOROANILINE**  
(Clauses 3.2, 5.3 and 6.1)

Sl No.	Characteristic	Requirement	Method of tests, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, <i>Min</i>	98.0	—	<b>13.1</b> of IS 5299
	<i>Or</i>			
	Purity by GC <sup>1)</sup> , percent area, <i>Min</i>	99.50	A	—
ii)	Impurities by GC :			
	i) Aniline, percent area, <i>Max</i>	0.05		
	ii) 3,4- Dichloroaniline, percent area, <i>Max</i>	0.50		
	iii) <i>m</i> -Chloroaniline, percent area, <i>Max</i>	0.50	B	—
	iv) Other impurities, percent area, <i>Max</i>	0.50		
iii)	Moisture content by Karl Fischer, percent by mass, <i>Max</i>	0.30		
iv)	Crystallization point <sup>2)</sup> , °C	47.8	—	<b>8</b> of IS 5299

1) In case of disputes, the sample shall satisfy the requirement of purity by GC, percent by area

2) 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/ HDPE bags lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

### 4.2 Marking

**4.2.1** Each container/ bag 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, month and year of manufacturing;

- e) Shelf life of the material;
- f) Pictograms, signal word, hazard statement and precautionary statement as mentioned in Annex C; and
- h) 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**

Test for the determination of all characteristics, shall be conducted on the composite sample.

#### **5.3 Criteria for Conformity**

For declaring the conformity of the requirements of all characteristics tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

### **6 TESTS**

**6.1** Tests shall be carried out as prescribed in col (4) and (5) of Table 1.

#### **6.2 Quality of Reagents**

Unless specified otherwise, 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. (ii) and (iii)]

#### **DETERMINATION OF PURITY AND IMPURITIES CONTENT BY GAS CHROMATOGRAPHY**

##### **A-1 GENERAL**

Determination of purity 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** — (14% cyanopropyl-phenyl)-methylpolysiloxane column with length 30 m, inner diameter 0.25 mm and film thickness 1.00 µm or equivalent.

#### **A-2.5.2 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** : 95.8 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<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 REAGENT**

#### **A-3.1 Methanol** — Solvent

### **A-4 PROCEDURE**

Take 1.0 g of 2,5-Dichloroaniline (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. Purity determination of 2,5-Dichloroaniline shall be carried by Gas chromatography instrument through area percent calculation.

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

2,5-Dichloroaniline	14.09 min
Aniline content	8.44 min
<i>m</i> -Chloroaniline	12.33 min
3,4- Dichloroaniline	16.22 min

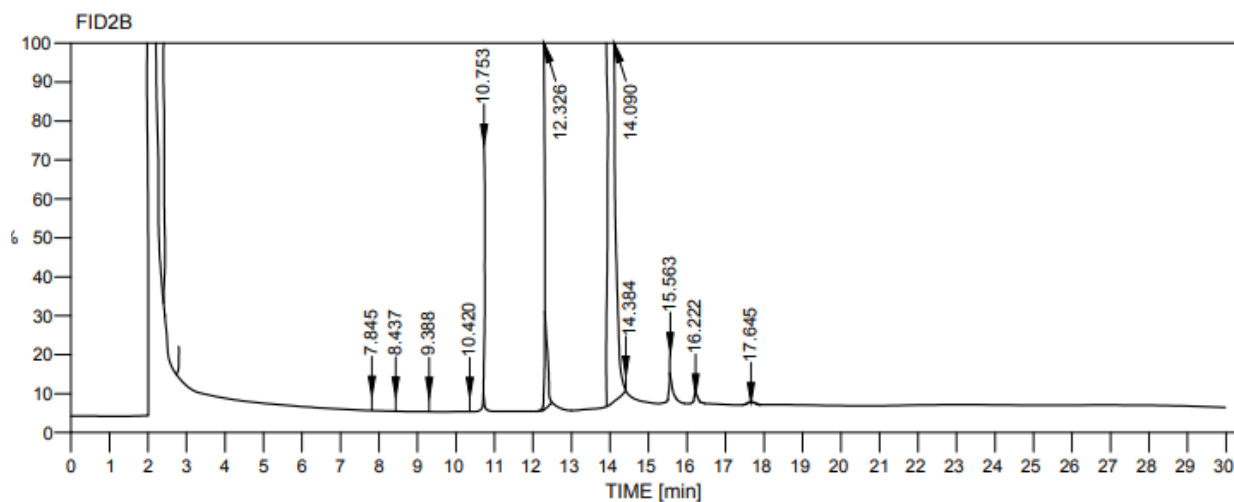


FIG. 1 A TYPICAL CHROMATOGRAM

#### A-6 CALCULATION

**A-6.1** Calculate the peak area of individual constituent pertaining to 2,5-Dichloroaniline 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 2,5-Dichloroaniline.

$$\text{Purity, percent by area} = \frac{\text{2,5-Dichloroaniline peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

**A-6.2** Similarly, aniline, 3,4-dichloroaniline and *m*-chloroaniline content shall be calculated.

### ANNEX B

[Table 1, Sl. No. (iii)]

#### DETERMINATION OF 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 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.

## **B-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, in ml, of karl fischer reagent consumed;




$F$  = karl fischer reagent factor, in mg/ml; and

$W$  = weight, in g, of sample taken.

## **ANNEX C**

*(Foreword)*

### **Pictograms, signal word, hazard statement and precautionary statement**

<b>Pictogram(s)</b> :			
<b>Signal Word</b> :	Warning	Health Hazard	Environmental Hazard
<b>Hazard statement(s)</b> :	<b>H301+H311+H331</b> - Toxic if swallowed, in contact with skin or if inhaled. <b>H373</b> - May cause damage to organs through prolonged or repeated exposure. <b>H410</b> - Very toxic to aquatic life with long lasting effects.		
<b>Precautionary Statements</b>	<b>P260</b> - Do not breathe dust. <b>P264</b> - Wash hands thoroughly after handling. <b>P270</b> - Do not eat, drink or smoke when using this product. <b>P271</b> - Use only outdoors or in a well-ventilated area. <b>P273</b> - Avoid release to the environment. <b>P280</b> - 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

**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.

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