

**BUREAU OF INDIAN STANDARDS**  
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*Draft Indian Standard*  
**1, 4-DICHLORO-2-NITROBENZENE — SPECIFICATION**  
(First Revision of IS 8861)  
(ICS 71.080.99)

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Dye Intermediates Sectional Committee,  
PCD 26

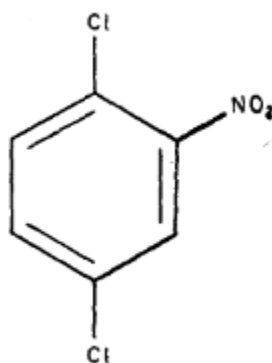
Last date for Comments  
2 March 2024

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**FOREWORD**

(Formal clauses to be added later)

1, 4-Dichloro-2-nitrobenzene ( $C_6H_3O_2NCl_2$ ) is an important dye intermediate used in the manufacture of dyes. It is represented by the following structural formula:



1, 4-DICHLORO-2-NITROBENZENE  
Molecular Mass 192  
CAS-No. 89-61-2

This standard was originally published in 1978. In this (*first*) revision, determination of 1, 4-dichloro-2-nitrobenzene content (assay) and 3, 4-Dichloronitrobenzene content (impurity) by gas chromatography, as an alternative method has been incorporated. Three new characteristics that are 2, 3-Dichloronitrobenzene, para-Dichlorobenzene and moisture content have been incorporated along with their requirements and methods of determination.

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 F, 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 the methods of sampling and test for 1, 4-dichloro-2-nitrobenzene.

## 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:

<i>IS No.</i>	<i>Title</i>
1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
2552 : 1989	Steel drums (galvanized and ungalvanized) ( <i>third revision</i> )
5299 : 2001	Methods for sampling and tests for dye intermediates ( <i>first revision</i> )

## 3 GRADES

**3.1** There shall be two grades of the material, namely:

- a) *Grade 1* — Liquid, and
- b) *Grade 2* — Flakes.

## 4 REQUIREMENTS

### 4.1 Description

a) Grade-1 (Liquid) — The material shall be in the form of clear light yellow to reddish liquid and free from extraneous matter.

b) Grade-2 (Flakes) — The material shall be in the form of clear light yellow to reddish flakes and free from extraneous matter.

**4.2** The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 5 and 6 of Table 1.

**TABLE 1 REQUIREMENTS FOR 1, 4-DICHLORO-2-NITROBENZENE**  
(*Clauses 4.2, 6.3 and 7.1*)

SI No.	Characteristic	Requirement		Method of tests Ref to	
		Grade 1 (Liquid)	Grade 2 (Flakes)	Annex	IS
(1)	(2)	(3)	(4)	(5)	(6)
i)	Assay by GC <sup>1</sup> ), percent area, <i>Min</i>	99.7	99.7	A	—
	<i>Or</i>				
ii)	Assay (based on Coupling Value), percent by mass, <i>Min</i>	99.5	99.5	B	—

iii)	3,4-Dichloronitrobenzen Content by TLC	—	0.1	C	—
	<i>Or</i>				
iv)	3,4-Dichloronitrobenzen Content by GC, <i>Max</i>	—	0.10	A	—
v)	2,3-Dichloronitrobenzene Content by GC, percent area, <i>Max</i>	—	0.10		—
vi)	para-Dichlorobenzene Content by GC, percent area, <i>Max</i>	—	0.10		—
vii)	Moisture Content by Karl Fischer, ppm, <i>Max</i>	3000	3000	D	IS 2362
viii)	Crystallization point <sup>2)</sup> , °C, <i>Min</i>	52.5	52.5	E	—

<sup>1)</sup>In case of disputes, determination of assay by GC shall be the referee method.

<sup>2)</sup>Crystallization point is optional requirement to be tested.

## 5 PACKING AND MARKING

### 5.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or HDPE bag, or in tanker or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

### 5.2 Marking

**5.2.1** 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 requirement.

**5.2.2** For supplies of material in bulk, a test certificate containing the details mentioned at **5.2.1** shall be provided for each consignment.

**5.2.3** All bags/ containers/ tankers for bulk transport in which the material is stored or transported shall be prominently and clearly marked with minimum cautionary notice, worded as:

**‘TOXIC! HANDLE WITH CARE!’**

### 5.2.4 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.

## 6. SAMPLING

6.1 The method of drawing representative samples of the material shall be as prescribed in 4 of IS 5299.

### 6.2 Number of Tests

6.2.1 Tests for assay and impurities shall be conducted on each of the individual samples.

6.2.2 Test for the remaining characteristics shall be conducted on the composite sample.

### 6.3 Criteria for Conformity

#### 6.3.1 For Individual Samples

The lot shall be declared as conforming to the requirements of all tests mentioned in 6.2.1, if each of the individual test results satisfies the relevant requirements given in Table 1.

#### 6.3.2 For Composite Sample

For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (*see* 6.2.2), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

## 7 TEST

7.1 Tests shall be conducted according to the methods prescribed in col 5 and 6 of Table 1.

### 7.2 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. (i)]

### DETERMINATION OF ASSAY OF 1, 4-DICHLORO-2-NITROBENZENE BY GAS CHROMATOGRAPHY

#### A-1 GENERAL

Determination of 1, 4-Dichloro-2-nitrobenzene content (assay) and impurities that is 3,4-Dichloronitrobenzene, 2,3-Dichloronitrobenzene and para-Dichlorobenzene 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 Pipette

##### A-2.4 Micro syringe

**A-2.5 Gas Chromatograph** — any 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 : 40 ml/min  
Zero air flow : 400 ml/min  
Purge Flow : 3.0 ml/min  
Make up gas (N<sub>2</sub>) flow : 30 ml/min  
Split Ratio : 1 : 40  
Detector Type : Flame Ionization Detector (FID)  
Detector Temperature : 275°C  
Injection Volume : 1.0 µl  
Total run time : 25 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 1, 4-dichloro-2-nitrobenzene 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.

### A-5 PEAK TIME

1, 4-Dichloro-2-nitrobenzene : 13.40 min.  
3, 4-Dichloronitrobenzene : 13.45 min.  
2,3-Dichloronitrobenzene : 13.80 min.  
para-Dichlorobenzene : 7.25 min.

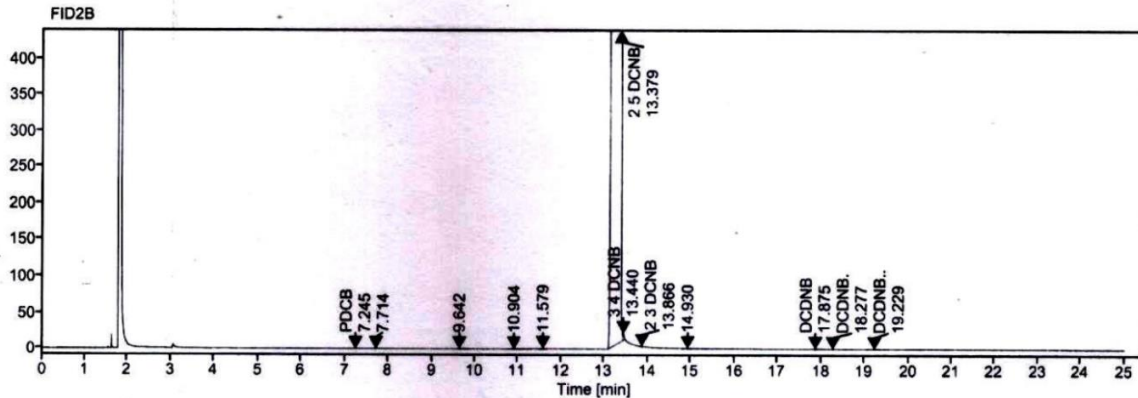


FIG 1 A TYPICAL CHROMATOGRAPH

### A-6 CALCULATION

**A-6.1** Calculate the peak area of individual constituent pertaining to 1,4-Dichloro-2-nitrobenzene 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 1, 4-Dichloro-2-nitrobenzene.

$$\text{Assay, percent by area} = \frac{\text{1,4-Dichloro-2-nitrobenzene peak area in the sample}}{\text{Sum area of all the peaks in the chromatogram}} \times 100$$

**A-6.2** Similarly, 3, 4-Dichloronitrobenzene, 2,3-Dichloronitrobenzene and para-Dichlorobenzene content shall be calculated.

### ANNEX B

[Table 1, sl no. (ii)]

#### DETERMINATION OF ASSAY OF 1, 4- DICHLORO-2-NITROBENZENE (COUPLING VALUE)

### B-1 PREPARED SAMPLE

**B-1.1** Dry the material at  $(45 \pm 1)^\circ\text{C}$  under vacuum. Grind and mix well. Transfer the material to a wide mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for the determination of assay by coupling value.

**B-1.2** Assay of 1, 4-dichloro-2-nitrobenzene shall be determined according to the method prescribed in 14 of IS 5299.

### ANNEX C

[Table 1, sl no. (iii)]

#### DETERMINATION OF 3, 4-DICHLORONITROBENZENE CONTENT BY THIN LAYER CHROMATOGRAPHY

### C-1 GENERAL

The isomer 3, 4- dichloronitrobenzene in 1, 4-dichloro-2-nitrobenzene is determined by thin layer chromatography after reducing the nitro compound to the amino compound.

## C-2 APPARATUS

**C-2.1 Thin layer glass plate** — of size 10 × 20 cm coated uniformly with silica gel-G or an equivalent powder in a thickness of 250 micron and activated at 110°C for 1 hour.

**C-2.2 Micropipette**

**C-2.3 Developing Chamber**

**C-2.4 Chromatographic sprayer**

## C-3 REAGENTS

**C-3.1 1, 4-Dichloro-2-nitrobenzene** — pure, free from isomers.

**C-3.2 3, 4-Dichloronitrobenzene** — pure

**C-3.3 Glacial Acetic Acid**

**C-3.4 Zinc Dust**

**C-3.5 Benzene**

**C-3.6 Sodium Nitrite**

**C-3.7 Hydrochloric acid**

**C-3.8 Spray Reagent** — Dissolve 1 g of 1 N naphthylethylene diamine dihydrochloride in a mixture of 50 ml of methanol and 50 ml of 10 percent hydrochloric acid (v/v).

**C-3.9 Sodium hydroxide Solution** — 20 percent (m/v).

## C-4 PROCEDURE

**C-4.1** Weigh 1 g each of the pure 1, 4- dichloro-2-nitrobenzene into 4 beakers of 500 ml capacity each. Weigh 0.1 g of 3, 4-dichloronitrobenzene into a dry 100 ml volumetric flask, dissolve and make up to volume with glacial acetic acid. Pipette out (using a safety pipette) 0.5 ml , 1 ml , 2 ml, and 3 ml of this acetic acid solution into the 4 beakers containing pure 1,4-dichloro-2- nitrobenzene. Thus we have standards containing 0.05 percent, 0.1 percent, 0.2 percent of 3, 4- dichloronitrobenzene. Now add 20 ml of glacial acetic acid to each beaker to dissolve the contents, warm, if necessary. Add 5 g of zinc dust to each beaker, followed by 10 ml of Hydrochloric acid. Keep for 30 minutes. Again add 5 g more of Zinc dust and 10 ml of hydrochloric acid and keep for 30 minutes. Repeat the operation for a third time. Now make the contents of all the beakers alkaline using sodium hydroxide solution. Add 25 ml of benzene and stir well to extract the amine formed. Transfer the benzene layer to a separating funnel by decantation. Repeat the extraction with two more instalments of 25 ml each of benzene and collect all the benzene extracts in the same separating funnel. Separate the benzene layer and transfer it into a 100 ml volumetric flask.

**C-4.2** Prepare the amine extract of the material under test in the same way as in the case of the four standards (*see C-4.1*).

**C-4.2** Spot 10 microlitre each of the sample solution and the four standards on the thin layer plate. Allow to dry and keep the plate in the developing chamber containing benzene solvent. Allow to run for about 30 min. Take out the plate and allow to dry. Keep the plate for 2 min in a chamber containing nitrous acid fumes (prepared by mixing sodium nitrite and hydrochloric acid). Blow out the excess nitrous fumes from the plate and spray with the spraying reagent. The sample and the impurities are seen as violet spots. The material has  $R_f$  value of 0.95 and the 3, 4-dichloronitrobenzene 0.69. Compare the intensity of the impurity in the sample to the nearest in the standards.

## ANNEX D

[Table 1, sl no. (vii)]

### DETERMINATION OF 1, 4-DICHLORO-2-NITROBENZENE 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 25 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. Weigh 10 g of 1, 4-dichloro-2-nitrobenzene (10 ml, if liquid) and melt it, if required. Now, add the weighed sample 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 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

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

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

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 E

[Table 1, sl no. (viii)]

### DETERMINATION OF 1, 4-DICHLORO-2-NITROBENZENE CRYSTALLIZATION POINT

#### E-1 APPARATUS



**E-1.1 Thermometer**

NOTE — The thermometer shall bear a calibration certificate from any institution authorized to issue certificate traceable to international or national measurement standards.

**E-1.2 Dry Heating Block**

**E-1.3 Stainless steel Wire**

**E-1.4 Analytical Balance**

**E-2 PROCEDURE**

Take approximately 15 g to 20 g of sample (1, 4-dichloro-2-nitrobenzene) in a test tube and heat the sample in a dry heater till it completely melts. Remove the test tube from the dry heating block and place thermometer in the test tube in such a way that the thermometer bulb is fully covered by sample material as shown in the figure below:

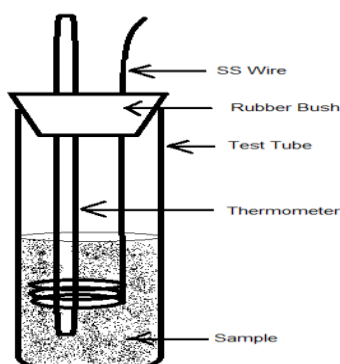





FIG. 2 APPARATUS

Now, stir the sample with stainless steel wire with vertical movement until solidification takes place. The constant temperature observed during solidification of the sample material shall be considered as crystallization point.

**ANNEX F**  
(Foreword)

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

Pictogram(s)	DANGER	WARNING	ENVIRONMENTAL HAZARD
  			
<b>Signal Word</b>	<b>DANGER</b>	<b>WARNING</b>	<b>ENVIRONMENTAL HAZARD</b>
<b>Hazard Statement</b>	<b>H302</b> Harmful if swallowed. <b>H315</b> Causes skin irritation. <b>H319</b> Causes serious eye irritation. <b>H411</b> Toxic to aquatic life with long lasting effects. <b>H335</b> May cause respiratory irritation. <b>H317</b> May cause an allergic skin reaction.		

**Precautionary  
Statement**

**Precautionary statement(s) Prevention**

**P271** Use only outdoors or in a well-ventilated area.

**P280** Wear protective gloves/protective clothing/eye protection/face protection.

**P261** Avoid breathing dust/fumes.

**P270** Do not eat, drink or smoke when using this product.

**Precautionary statement(s) Response**

**P302+P352** IF ON SKIN: Wash with plenty of water and soap.

**P305+P351+P338**

IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

**P333+P313** If skin irritation or rash occurs: Get medical advice/attention.

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

**Precautionary statement(s) Storage**

**P405** Store locked up.

**P403+P233** Store in a well-ventilated place. Keep container tightly closed.

**Precautionary statement(s) Disposal**

**P501** Dispose of contents/container in accordance with local regulations.

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