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Draft Indian Standard
4-NITRO-CHLOROBENZENE — SPECIFICATION
(First Revision of IS 8195)
(ICS 71.080.99)

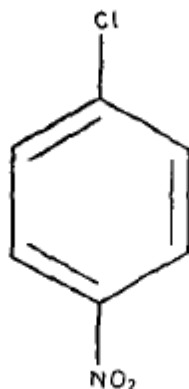
Dye Intermediates Sectional Committee,
PCD 26

Last date for comment
11 February 2023

FOREWORD

(Formal clauses to be added later)

4-Nitro-chlorobenzene ($C_6H_4O_2NCl$), also known as *p*-nitro chlorobenzene is an important dye intermediate used in the manufacture of dyestuffs. It is represented by the following structural formula:



4- NITRO-CHLOROBENZENE
Molecular Mass 157.5
CAS-No. 100-00-5

This standard was originally published in 1976. In this (*first*) revision, determination of 4-nitro-chlorobenzene content by Gas chromatography has been updated and a new characteristic that is moisture content has been incorporated. The requirement of dinitro compounds contents has been deleted. The amendment has also been incorporated in the standard.

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 the methods of sampling and test for 4-nitro-chlorobenzene.

2 REFERENCES

The following standards contain provisions which through reference in the 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 agreement, based on the 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 2552 : 1989	Steel Drums (Galvanized and Ungalvanized) — Specification (third revision)
IS 5299 : 2001	Methods of Sampling and Tests for Dye Intermediates (first revision)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light yellow crystalline mass.

3.2 The material shall also comply with the requirements as given in Table 1, when tested according to the methods prescribed col 4 and 5 of Table 1.

TABLE 1 REQUIREMENTS FOR 4-NITRO CHLOROBENZENE
(Clauses 3.2, 5.3.1 and 6.1)

Sl No.	Characteristics	Requirement	Method of Test Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay by GC, percent area, <i>Min</i>	99.50	A	—
ii)	2-Nitro-chlorobenzene, percent area, <i>Max</i>	0.35	A	—
iii)	Moisture Content by karl fischer, ppm, <i>Max</i>	1000	B	IS 2362
iv)	Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	—	11.3 of IS 5299
v)	Crystallization Point ¹⁾ , °C	82.2	C	—

¹⁾ Crystallization point is optional requirement to be tested.

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. The containers shall be securely closed.

4.2 Marking

4.2.1 Each 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) Gross, net and tare mass;
- d) Batch number, month and year of manufacturing;
- e) Shelf life of the material; and
- f) 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 shall be conducted on each of the individual samples.

5.2.2 Tests for the remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

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

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (*see* 5.2.2), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TESTS

6.1 Tests shall be carried out according to the methods 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.(i)]
ASSAY OF 4-NITRO-CHLOROBENZENE BY GAS CHROMATOGRAPHY

A-1 GENERAL

Determination of 4-nitro-chlorobenzene (assay) and 2-nitro-chlorobenzene 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 Pipette

A-2.4 Micro Syringe

A-2.5 Gas Chromatograph — Any gas chromatograph equipped with a flame ionization detector (FID).

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

Column oven programme

Rate (°C/min)	Temperature (°C)	Hold time (min)
--	100	2
10	230	10

Nitrogen pressure : 120 kPa

Hydrogen flow : 40 ml/min

Zero Air flow : 400 ml/min

Make up gas (N₂) flow : 30 ml/min

Split ratio : 1:40

Detector type : FID

Detector temperature : 275 °C

Injection volume : 1.0 µl

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, H₂, N₂) 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 Acetone — solvent

A-4 PROCEDURE

Take 1.0 g of 4-nitro-chlorobenzene and make up to 10 ml with acetone. Now, dissolve properly and take 1.0 µl of sample as prepared in micro syringe. Confirm there are no air bubbles in the syringe and inject the sample and allow the run to complete run time.

A-5 PEAK TIME

4-Nitro-chlorobenzene : 12.6 min

2-Nitro-chlorobenzene : 13 min

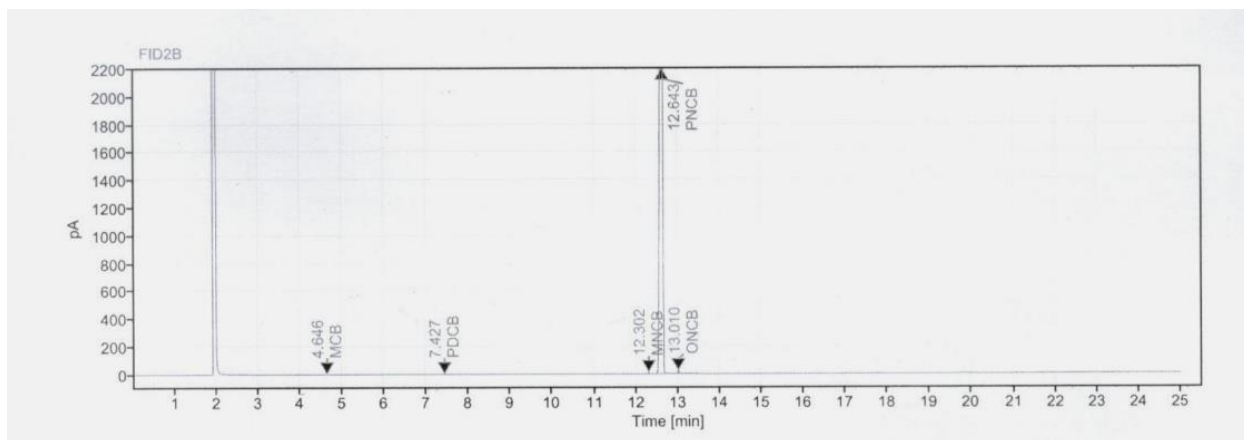


FIG. 1 A TYPICAL CHROMATOGRAM

A-6 CALCULATION

A-6.1 Calculate the peak area of individual constituent pertaining to 4-nitro-chlorobenzene 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 4- nitro-chlorobenzene.

$$\text{Assay, percent by area} = \frac{\text{4-Nitro-chlorobenzene peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

A-6.2 Similarly, 2-nitro-chlorobenzene content shall be calculated.

ANNEX B

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

DETERMINATION OF 4-NITRO CHLOROBENZENE 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 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 sample and melt the 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 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 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, sl. no.(iii)]

DETERMINATION OF 4- NITRO CHLOROBENZENE CRYSTALLIZATION POINT

C-1 APPARATUS

C-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.

C-1.2 Dry Heating Block

C-1.3 Stainless Steel Wire

C-1.4 Analytical Balance

C-2 PROCEDURE

Take approximately 15 g to 20 g of sample 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:

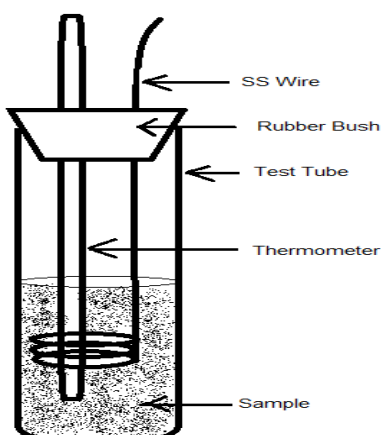


FIG 2 APPARATUS

Now, stir the material 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 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.