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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

2-Nitro Chlorobenzene — Specification

(*First Revision of* IS 8193)

(ICS 71.080.99)

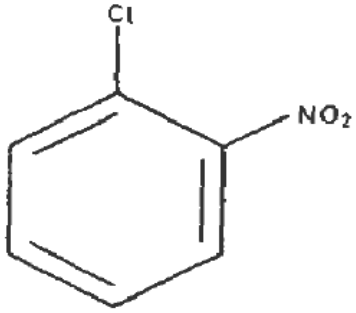
Dye Intermediates Sectional Committee, PCD 26

Last date for comments **30 January 2024**

FOREWORD

(*Formal clauses to be added later*)

2-Nitro chlorobenzene ($C_6H_4O_2NC1$), is an important dye intermediate used in the manufacture of dyestuffs and pigments. It is represented by the following structural formula:



2-Nitro chlorobenzene
Molecular mass 157.5
CAS NO. 88-73-3

This Indian standard was originally published in 1976. In this first revision, determination of 2-nitro-chlorobenzene content by Gas chromatography and a new characteristic i.e. moisture content has been incorporated. The existed characteristics that are determination of Dinitro compounds contents and 4-nitro chlorobenzene contents has been deleted.

The bags 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, 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 2-nitro chlorobenzene.

2 REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisions of this Standard. At the time of publication the additions 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*
1070 : 2023	Reagent grade water — Specification (*fourth revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) – Specification (*third revision*)
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 greenish yellow crystalline mass of disagreeable odour.

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

TABLE 1 REQUIREMENTS FOR 2-NITRO CIILOROBENZENE
(*Clauses* 3.2,5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test. Ref to
(1)	(2)	(3)	(4)
i)	Assay by GC, percent area, *Min*	99.80	A
ii)	Moisture Content by Karl Fischer, *Max*	1000 ppm	B/ IS 2362
iii)	Crystallization point, *Min*	31.7 °C	C /8 of IS 5299
iv)	Matter insoluble in methanol, percent by mass, *Max*	0.2	clause 11.3 of IS 5299

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in GP drums (*see* IS 2552) or tanker /ISO tank or 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 *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 All Tests shall be conducted on each of the individual samples.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

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

6 TESTS

6.1 Tests shall be conducted according to the methods prescribed in col 4 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)]
DETERMINATION ASSAY OF 2-NITROCHLORO BENZENE BY GAS CHROMATOGRAPHY

A-1 GENERAL

2-Nitrochlorobenzene assay is determined using gas chromatography (GC).

A-2 APPARATUS

A-2.1 Analytical Balance

A-2.2 Glassware

A-2.2.1 Volumetric Flask — 10 ml and pipette

A-2.3 Micro Syringe

A-2.4 Gas Chromatograph — GC 7890B equipped with a flame ionization detector (FID) or Equivalent

A-2.4.1 Gas Chromatography Parameters

Column	:DB-1701 (30 m,0.25 mm ID,1.0 µm) or Equivalent, Temperature limit: -20 °C to 280 °C
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 pressure /flow	: 50 kpa / 40 ml/min
Air pressure /flow	: 50 kpa / 400 ml/min
Purge flow	: 3.0 ml/min
Make up flow/ pressure	: 30 ml/min or 50 kpa
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, 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 Acetone

A-4 PROCEDURE

Take 1.0 g of sample and make up to 10 ml with acetone. 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. Assay determination of 2-nitrochlorobenzene shall be carried by Gas Chromatography instrument through area percent calculation.

A-4 PEAK TIME

2-Nitrochlorobenzene : 13 min

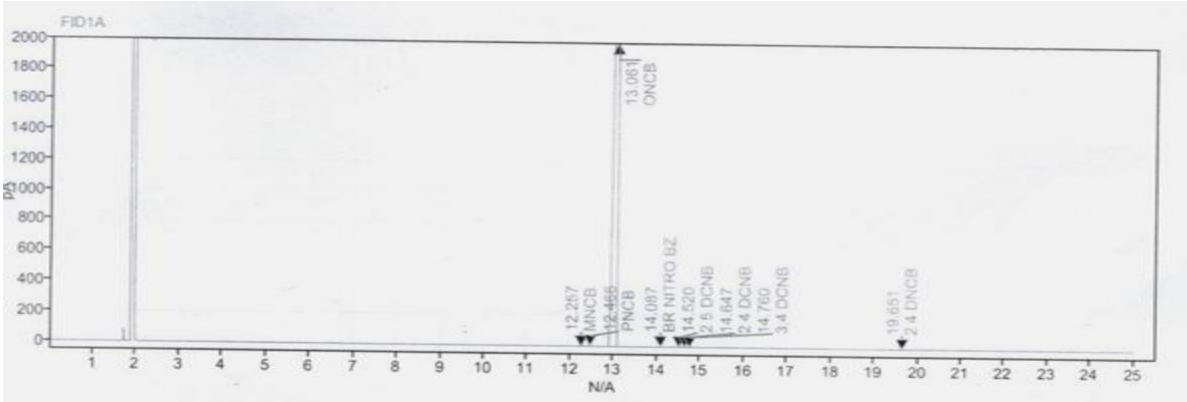


FIG 1 A TYPICAL CHROMATOGRAM

A-5 CALCULATION

Calculate the peak area of individual constituent pertaining to 2-Nitrochlorobenzene 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-Nitrochlorobenzene.

$$\text{Assay, percent by area} = \frac{\text{2−Nitrochlorobenzene peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

ANNEX B

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

DETERMINATION OF 2-NITRO CHLOROBENZENE MOISTURE CONTENT BY KARL FISCHER

B-1 REAGENTS

B-1.2 Karl Fischer Reagent

B-1.3 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 and melt the sample, if required.

After that, weigh 10 g of solid sample (10 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

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

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 SET POINT OF 2-NITRO CHLOROBENZENE

C-1 APPARATUS

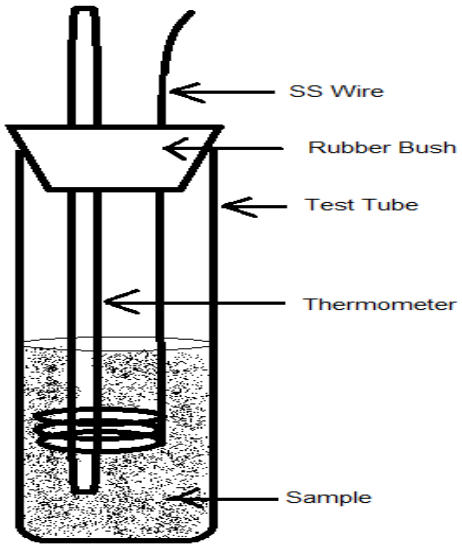
C-1.1 Calibrated Thermometer

C-1.2 Dry Heating Block

C-1.3 Stainless Steel Wire

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 calibrated 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:



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 Set Point. Use water/ice batch while determination of set point for products having lower set point then room temperature.

ANNEX D
(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement:

Pictogram(s)	
Signal Word	**Danger**
Hazard Statement	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.
Precautionary Statement	P201 - Obtain special instructions before use. P202 - Do not handle until all safety precautions have been read and understood. P260 - Do not breathe dust. 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.