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Draft Indian Standard
3-NITRO-CHLOROBENZENE — SPECIFICATION
(First Revision of IS 8194)
(ICS 71.080.99)

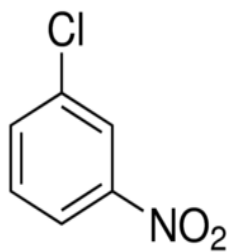
Dye Intermediates Sectional Committee,
PCD 26

Last date for Comments
04 February 2023

FOREWORD

(Formal clauses to be added later)

3-Nitro-chlorobenzene (C₆H₄O₂NCl) is an important dye intermediate used in the manufacture of dyestuffs. It is represented by the following structural formula:



3-Nitro chlorobenzene
Molecular mass 157.5
CAS No. 121-73-3

This standard was originally published in 1976. In this (*first*) revision, determination of 3-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 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 (second revision). The number of significant places retained in the rounded of 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 3-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:

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

3 REQUIREMENTS

3.1 Description

The material shall be in the form of yellow/brown crystalline mass or molten mass.

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

TABLE 1 REQUIREMENTS FOR 3-NITRO-CHLOROBENZENE
(Clauses 3.2, 5.3.1 and 6.1)

SI No.	Characteristics	Requirement	Method of Test Ref to Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Assay by GC, percent area, <i>Min</i>	99.40	A	—
ii)	Moisture Content by Karl Fischer, <i>Max</i>	2000 ppm	B	IS 2362
iii)	Crystallization point ¹⁾ , <i>Min</i>	43.5 °C ²⁾	C	7 of IS 5299
iv)	Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	—	11.3 of IS 5299

¹⁾Optional test

²⁾3-Nitro-chlorobenzene exists in two polymorphic forms The stable form has a crystallizing point of 43.5°C and the unstable form has a crystallizing point of 23.5°C If the crystallizing point observed is that of the unstable form, the sample should be heated and the test is to be repeated.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or in tanker 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 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 requirement of assay 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 conducted 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)]
**DETERMINATION ASSAY OF 3-NITRO-CHLOROBENZENE BY GAS
CHROMATOGRAPHY**

A-1 GENERAL

Assay determination of 3-nitro-chlorobenzene shall be carried out 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-2.3 Micro Syringe

A-2.4 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID) , a split /splitless injector and a suitable electronic integrator/ software may be used with following accessories and operating condition:

A-2.4.1 Column, DB-1701 with length 30 m, inner diameter 0.25 mm and film thickness 1.0 µm or equivalent.

A-2.4.2 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/pressure : 40 ml/min or 50 kpa

Air flow/pressure : 400 ml/min or 50 kpa

Purge flow : 3.0 ml/min

Make up gas : Nitrogen

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

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

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 a micro syringe. Confirm if there are no air bubbles in the syringe and then inject the sample and allow the run to complete run time.

A-5 PEAK TIME

3-Nitrochlorobenzene : 13 min

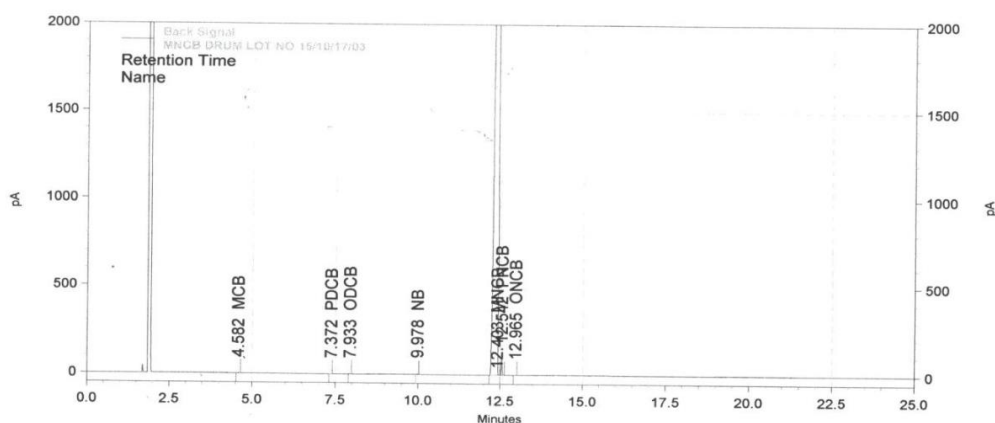


FIG 1 A TYPICAL CHROMATOGRAM

A-6 CALCULATION

Calculate the peak area of individual constituent pertaining to 3-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 3- nitro-chlorobenzene.

$$\text{Assay, percent by area} = \frac{\text{3-Nitrochlorobenzene peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

ANNEX B

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

DETERMINATION OF 3-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 then add Karl Fischer reagent to complete neutralization of methanol. After that, weigh 10 g of sample in titration vessel and dissolve it in methanol. If they are not soluble in methanol then heat a solution. Now, instrument automatically starts addition of Karl Fischer reagent in the titration vessel to titrate moisture content present in sample. Instrument will stop adding Karl Fischer 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 3-NITRO CHLOROBENZENE SET POINT

C-1 APPARATUS

C-1.1 Thermometer

NOTE — The thermometer shall bear a 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-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:

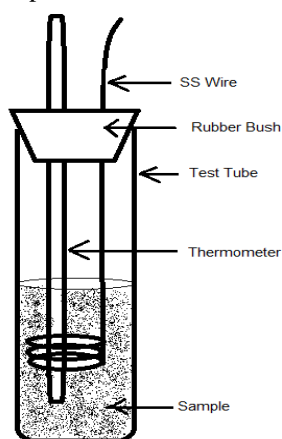


FIG. 2 APPARATUS

Now, stir the 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. Use water/ice bath while determination of crystallization point for products having lower crystallization point than room temperature.

ANNEX D
(Foreword)

Pictograms, signal word, hazard statement and precautionary statement:

Pictogram(s)



Signal Word

Warning

Hazard Statement

H302 - Harmful if swallowed
H319 - Causes serious eye irritation

Precautionary Statement

P264 - Wash hands, forearms and face thoroughly after handling.
P270 - Do not eat, drink or smoke when using this product.
P280 - Wear protective gloves/protective clothing/eye protection/face protection.
P301+P312 - If swallowed: Call a poison center or doctor if you feel unwell.
P305+P351+P338 - IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P330 - Rinse mouth.
P337+P313 - If eye irritation persists: Get medical advice/attention.
P501 - Dispose of contents/container to hazardous or special waste collection point, in accordance with local, regional, national and/or international regulation.
