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भारतीय मानक मसौदा

2,6-डाइक्लोरो-4-नाइट्रोएनिलिन — विशिष्टि

(IS 15133 का पहला पुनरीक्षण)

Draft Indian Standard

2,6-Dichloro-4-Nitroaniline — Specification

(First Revision of IS 15133)

(ICS 71.080.20)

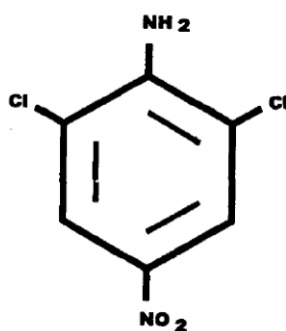
Dye Intermediates Sectional Committee,
PCD 26

Last date for Comments:
9th February 2025

FOREWORD

(Formal clauses to be added later)

2, 6-Dichloro-4-nitroaniline is an important dyestuff intermediate used in the manufacture of azo disperse dyes. It has the following structural formula:



2, 6-Dichloro-4-Nitroaniline
(Molecular Mass: 207)
CAS No. 99-30-9

This standard was original published in 2002. In this (first) revision, a new characteristic that is purity by high performance liquid chromatography and its requirement has been incorporated. Other existed characteristics such as 2-chloro-4-nitroaniline content, 4-nitroaniline content and matter insoluble in methanol have been deleted.

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 testing for 2, 6-Dichloro-4-Nitroaniline.

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 given below:

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

3 REQUIREMENTS

3.1 Description

The material shall be in the form of yellow color, free from lumps and extraneous substances.

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, 6-DICHLORO-4-NITROANILINE
(Clauses 3.2, 5.3.1 and 6.1)

Sl No.	Characteristic	Requirement	Method of tests, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Purity by HPLC ¹⁾ , percent by area, <i>Min</i>	97.0	A	—
	<i>Or</i> Purity by GC, percent by area, <i>Min</i>	97.0	B	—
ii)	Moisture Content by moisture balance, <i>Max</i>	1.0	C	10 of IS 5299

¹⁾In case of disputes, HPLC shall be the referee method in the determination of purity.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in galvanized iron drums (*see* IS 2552) or tanker or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container 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) Batch number;
- d) Gross, net and tare mass;
- e) Month and year of manufacturing;
- f) Shelf life of the material; and
- g) 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 the determination of all characteristics, namely purity by HPLC, purity by GC and moisture content, shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 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 requirements given in Table 1.

6 TESTS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col (4) and (5) of Table 1.

6.2 Quality of Reagents

Unless otherwise specified, pure chemicals 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 PURITY BY HIGH-PERFORMANCE LIQUID
CHROMATOGRAPHY**

A-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

A-2 OBJECTIVE

To determine purity of 2, 6-dichloro-4-nitroaniline by high performance liquid chromatography.

A-3 APPARATUS

A-3.1 Volumetric flask, 100 ml

A-3.2 Pipette

A-3.3 Beaker

A-3.4 Digital Balance, 0.0001g

A-3.5 High-Performance Liquid Chromatography – quaternary gradient liquid chromatography system with UV- visible detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

A-3.5.1 Column – C18 100 Å with length 250 mm, Internal Diameter 4.6 mm and particle size 5 µm or equivalent

A-4 REAGENTS

A-4.1 Methanol, HPLC grade

A-4.2 Acetonitrile, HPLC grade

A-4.3 Water, HPLC grade

A-5 REFERENCE STANDARD PREPARATION

Weigh accurately 0.1 g to 0.11 g reference standard in 100 ml volumetric flask and make up to 100 ml with Methanol and mix well. Use this standard solution within 72 hr.

A-6 SAMPLE PREPARATION

Weigh accurately 0.1 g to 0.11 g sample in 100 ml volumetric flask and make up to 100 ml with Methanol and mix well. Use this standard solution within 72 hr.

A-8 FLOW RATE, 1.0 ml / min

A-9 MOBILE PHASE

Acetonitrile	Water
45	55

A-10 INJECTION VOLUME, 10 μ l

A-11 RUN TIME, 40 min

A-12 WAVE LENGTH, 220nm (UV)

A-13 PEAK TIME

2,6-Dichloro-4-nitroaniline 9.30 min

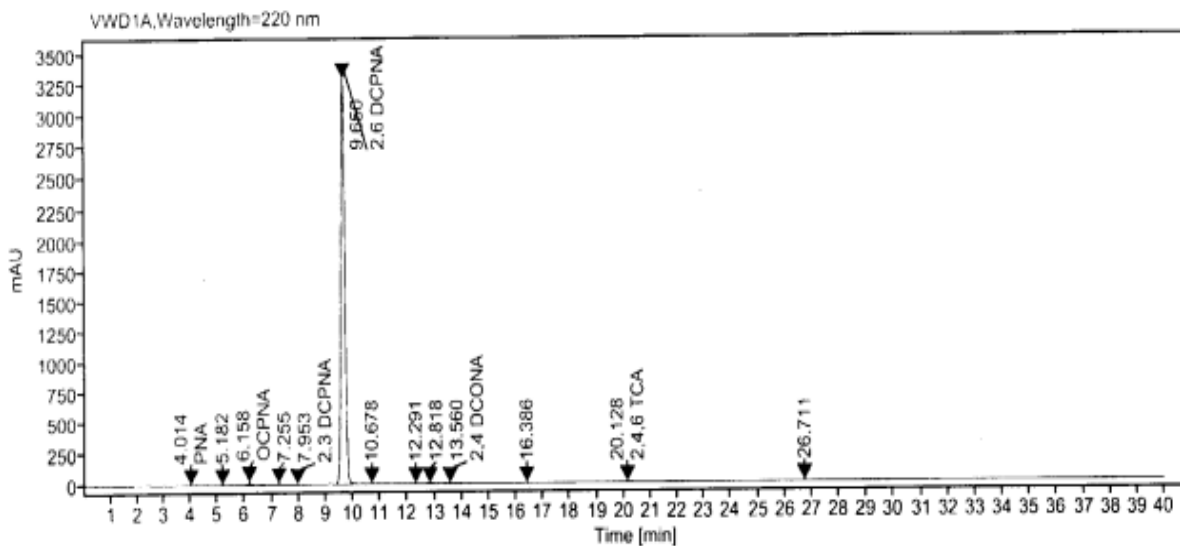


FIG. 1 A TYPICAL CHROMATOGRAM

A-14 CALCULATION

Calculate the peak area of individual constituents pertaining to 2,6-Dichloro-4-nitroaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of purity 2,6-Dichloro-4-nitroaniline as given below:

$$\text{Purity, percent by area} = \frac{\text{2,6-Dichloro-4-nitroaniline peak area in sample}}{\text{Sum area of all peaks in the chromatogram}} \times 100$$

ANNEX B

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

DETERMINATION OF PURITY BY GAS CHROMATOGRAPHY

B-1 OUTLINE OF THE METHOD

The purity of 2, 6-dichloro-4-nitroaniline is determined by the gas chromatographic method. For detailed test procedure, reference may be made to IS 5299. However, the conditions for a typical chromatographic analysis are given below as well.

B-2 APPARATUS /Instruments details

B-2.1 Volumetric Flask, 10ml

B-2.2 Pipette

B-2.3 Glass Beaker

B-2.4 N, N-Dimethylformamide

B-2.5 Digital Balance, 0.0001g

B-2.6 Gas chromatography – Any gas chromatography equipped with a flame ionization detector (FID).

B-2.6.1 Column, 100% dimethylpolysiloxane with length 30 m, inner diameter 0.53 mm and film thickness 0.5 µm or equivalent.

A-2.8 Gas Chromatography Parameters:

Carrier Gas	Nitrogen
Carrier Pressure	24.9 kPa
Split ratio	1:20
Purge Flow	3.0 ml/min
Make up Flow	30 ml/min
Hydrogen Flow	40 ml/min
Zero Air Flow	400 ml/min

Oven temp.	Rate (°C/min)	Temperature (°C)	Hold time (min)
	--	100	2
	10	250	13

Injector temp	250°C
Detector temp	250°C
Type of Detector	Flame Ionization detector (FID)
Run time	30 min
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.

B-3 SAMPLE PREPARATION

Take 0.2 g of the sample and put it into a 10 ml volumetric flask. Add N, N-Dimethylformamide to the flask until it reaches the 10 mL mark and shake it well to mix. Using a micro syringe, take

1.0 µl of the sample and check that there are no air bubbles in the syringe. Inject the sample using an auto-Sampler or manually, and let the analysis run to complete run time.

B-4 PEAK TIME

2,6-Dichloro-4-nitroaniline 12.60 min

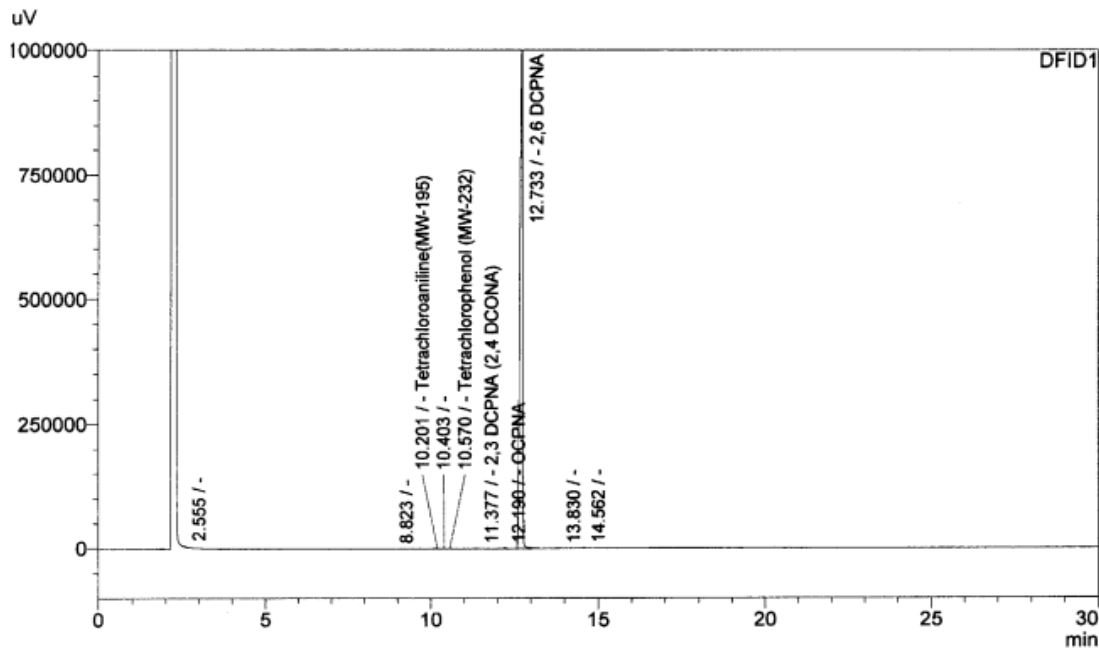


FIG.2 A TYPICAL CHROMATOGRAPH

B-5 CALCULATION

Calculate the peak area of individual constituent pertaining to 2,6-dichloro -4-nitroaniline on the chromatogram. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained as given below:

$$\text{Purity, percent by area} = \frac{\text{2,6-dichloro -4-nitroaniline peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

ANNEX C
[Table 1, sl. no.(ii)]
MOISTURE CONTENT BY MOISTURE BALANCE

C-1 APPARATUS

C-1.1 Aluminium petri plate

C-1.2 Tongue

C-1.3 Moisture Balance

C-2 PRINCIPLE

This instrument performs measurement based on the thermogravimetric principle. Moisture is determined from the weight loss of a sample dried by heating. Check the spirit level of the instrument, it should be in the center.

C-3 PROCEDURE

Switch on the instrument by pressing the on/off button. Check the set temperature for 2,6- dichloro-4- nitroaniline, it should be 100 °C and the time mode should be in auto. Press the tare button, this sets the moisture Balance to zero, then open the heating module. Place the empty sample pan in the pan handler. Place the pan handler in the sample chamber. Ensure that the tongue of the pan handler fits exactly in the slot of the draft shield element. The sample pan must lie flat in the pan handler. Place the specimen sample in the sample pan. Sample weight should be 3.0 g to 5.0 g. Close the heating module. Press the start button, then the Moisture Balance starts drying and moisture analysis measurement process. The first display comes up when the test is started. The display indicates the temperature, time and moisture results and one graph of process indicator. When mean weight loss is less than 1 mg/30 second, then auto switch-off is reached. Then the final results and END is displayed. Display will show the result in percent. Note down the results.

ANNEX D
(Foreword)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :



Signal Word :

DANGER

Hazard statement(s) :

Fatal if swallowed, In contact with skin or if inhaled.
May cause damage to organs through prolonged or repeated exposure

Precautionary Statements

Do not breathe dust/ fume/gas/ mist/ vapors/ spray.
Do not get in eyes, on skin or on clothing.
Wash hands, forearms and face thoroughly after handling.
Do not eat or smoke when using this product
Use only outdoors or in a well ventilated area
Wear protective gloves/ protective clothing/ eye protection/ face protection.
[In case of inadequate ventilation] where respiratory protection.
If swallowed: Immediately call a poison center or doctor.
If on skin: Wash with plenty of water.
If inhaled, remove the person to fresh air and keep comfortable for breathing.
Immediately call a poison center or doctor.
Get medical advice/ attention if you feel unwell.
Specific treatment is urgent (see supplemental first aid instruction on this label)
Specific treatment (see supplemental first aid instruction on this label).
Rinse mouth
Take off immediately all contaminated clothing and wash it before reuse.
Store in a well ventilated place. Keep the container tightly closed.
Store locked up.
Dispose of contents/container to hazardous or special waste collection point in accordance with local, regional, national and/or international regulations.
