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Draft Indian Standard
1,5-DICHLOROANTHRAQUINONE, TECHNICAL – SPECIFICATION
(Second Revision of IS 7360)
(ICS 71.080.99)

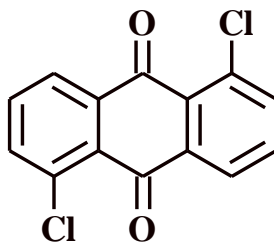
Dye Intermediates Sectional Committee,
PCD 26

Last date for Comments
29th December 2023

FOREWORD

(Formal Clause to be added later)

1, 5-Dichloroanthraquinone (C₁₄H₆O₂Cl₂) is an important intermediate used in the manufacture of vat dyes. It has the following structural formula:



1, 5-DICHLOROANTHRAQUINONE
(Molecular Mass: 277.0)
(CAS No.: 82-46-2)

This standard was first published in 1974 and subsequently revised in 1992. The *first* revision was taken up to stipulate the requirement of assay and impurities such as 1-chloroanthraquinone and 1,8-dichloroanthraquinone. The requirement of melting point was modified. Further, oxygen flask method was incorporated for determination of organically bound chlorine. In this (*second*) revision, HPLC method for determination of assay and impurities such as 1-chloroanthraquinone and 1,8-dichloroanthraquinone have been updated. A new characteristic named mercury content and its requirement and method of test are incorporated and requirement for characteristics such as moisture content and matter insoluble in chlorobenzene are updated.

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, the latest edition of GHS guidelines was 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, the methods of sampling and test for 1, 5-dichloroanthraquinone, technical.

2 REFERENCES

The following 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 possibilities of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water – Specification (<i>third revision</i>)
2552 : 1989	Steel drums (galvanized and un-galvanized) (<i>third revision</i>)
5299 : 2001	Methods of sampling and tests for dye intermediates (first revision)
5591 : 2003	Chlorobenzene – Specification (first revision)
1448 (Part 172):2020	Methods of Test for Petroleum and its Products Part 172 ICP-AES Method for Determination of Trace Elements in Petroleum Products

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light-yellow powder and shall be free from visible impurities.

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

Table 1 Requirements for 1, 5-Dichloroanthraquinone, Technical
(Clauses 3.2, 5.3.1, 5.3.2 and 6.1)

Sl. No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by mass, <i>Max</i>	1.0	—	10.3 of IS 5299
ii)	Sulphated ash, percent by mass, <i>Max</i>	1.0	—	12.2 of IS 5299
iii)	Matter insoluble in chlorobenzene, percent by mass, <i>Max</i>	0.5	A	—

iv)	Assay by HPLC ¹⁾ , percent by area, <i>Min</i> Impurities by HPLC : a) 1-Chloroanthraquinone, percent by area, <i>Max</i> b) 1,8-Dichloroanthraquinone, percent by area, <i>Max</i> Or	97 1.5 0.5	} B	—
v)	Assay by spectrophotometer, percent by mass Impurities: a) 1,5-dichloroanthraquinone, percent by mass, <i>Max</i> b) 1,8-dichloroanthraquinone, percent by mass, <i>Max</i>	95 4	} C	—
vi)	Melting range	Shall melt within the range of 246 °C to 248 °C		9 of IS 5299
viii)	Mercury content, <i>Max</i>	4 ppm		IS 1448 Part 172

¹⁾ In case of disputes, determination of assay by HPLC shall be the referee method.

4. PACKING AND MARKING

4.1 PACKING

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film 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 manufacture;
- f) Shelf life of the material; and
- g) Any other statutory requirements

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 Test for assay and its impurities shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of 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 a lot to the requirements of all other characteristics tested on the composite sample (*see 5.2.2*) the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in col 4 and col 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. (iii)]

DETERMINATION OF MATTER INSOLUBLES IN CHLOROBENZENE

A-1 APPARATUS

A-1.1 Quick Fit Conical Flask, 500 ml.

A-1.2 Sintered Glass Crucible, G4

A-1.3 Reflux Condenser

A-2 REAGENT

A-2.1 Chlorobenzene, reagent grade (*see IS 5591*).

A-3 PROCEDURE

A-3.1 Weigh accurately about 1.0 g of the sample in a 500 ml conical flask and add about 150 ml of chlorobenzene into it. Reflux for about 30 minutes using a water condenser. Filter on a tarred sintered glass crucible. Wash the conical flask, two to three times with hot chlorobenzene. Dry the sintered glass crucible in an oven at 100 °C to 110 °C for four hours. Cool the crucible to room temperature in a desiccator and weigh. The increase in the mass of the crucible is the mass of insoluble.

A-4 CALCULATIONS

Matter insoluble in chlorobenzene, percent by mass = $\frac{M_1}{M_2} \times 100$

where

M_1 = mass of insoluble in chlorobenzene, in g; and

M_2 = mass of material taken, in g.

ANNEX B

[Table 1, Sl. No. (iv)]

DETERMINATION OF 1,5-DICHLOROANTHRAQUINONE CONTENT (ASSAY), 1-CHLOROANTHRAQUINONE AND 1,8-DICHLOROANTHRAQUINONE CONTENT (IMPURITIES) BY HPLC

B-1 OUTLINE OF METHOD

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. 1,5-Dichloroanthraquinone and impurities like 1-chloroanthraquinone and 1,8-dichloroanthraquinone are determined using High Performance Liquid Chromatography (HPLC) method.

B-2 APPARATUS

B-2.1 HPLC — 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.

B-2.1.1 Column — C18 column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-2.2 Volumetric Flask — class A grade

B-3 REAGENTS

B-3.1 1-Chloroanthraquinone — known purity.

B-3.2 1, 5-Dichloroanthraquinone — known purity.

B-3.3 1, 8-Dichloroanthraquinone — known purity.

B-3.4 Acetonitrile — HPLC grade.

B-3.5 Tetrahydrofuran (THF) — HPLC grade.

B-3.6 Water — HPLC grade.

B-4 STANDARD SOLUTION PREPARATION

B-4.1 1-Chloroanthraquinone

Weigh accurately 20 mg of 1-Chloroanthraquinone into 50 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

B-4.2 1,8-Dichloroanthraquinone

Weigh accurately 20 mg of 1,8-Dichloroanthraquinone into 50 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

B-5 SAMPLE SOLUTION PREPARATION

Weigh accurately 20 mg sample into 50 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

B-6 FLOW RATE — 1.4 ml/min.

B-7 MOBILE PHASE — Acetonitrile: Water, 55: 45 (v/v)

B-8 COLUMN OVEN TEMPERATURE — Ambient temperature.

B-9 INJECTION VOLUME — 20 µl.

B-10 RUN TIME — 30 min.

B-11 WAVELENGTH — 254 nm.

B-12 PEAK TIME

1-Chloroanthraquinone: 13.5 min

1,8-Dichloroanthraquinone: 15.0 min

1,5-Dichloroanthraquinone: 16.6 min

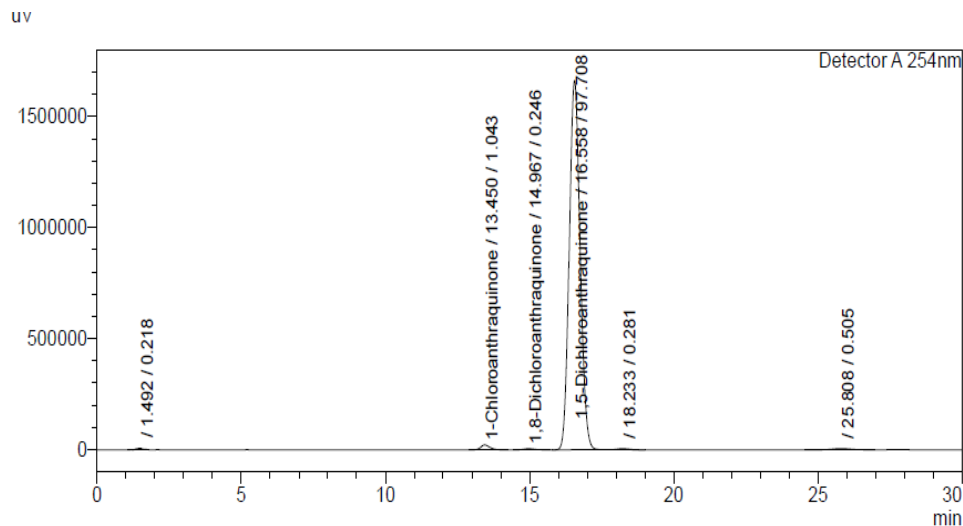


FIG 1 TYPICAL CHROMATOGRAPH

TABLE 2 TYPICAL PEAK TABLE

Components	Peak time	Area	Area percent
	1.49	96759	0.22
1-Chloroanthraquinone	13.45	462749	1.04
1,8-Dichloroanthraquinone	14.97	108932	0.25
1,5-Dichloroanthraquinone	16.56	43351150	97.71
	18.23	124529	0.28
	25.81	224109	0.51
Total		44368228	100.00

B-13 CALCULATION

B-13.1 Calculate the peak area of individual constituent pertaining to 1, 5-dichloroanthraquinone on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure 1, 5-dichloroanthraquinone.

$$1, 5\text{-Dichloroanthraquinone, percent by area} = \frac{1,5\text{-Dichloroanthraquinone Peak area in sample}}{\text{Sum Areas of all peaks in the chromatogram}} \times 100$$

B-13.2 Similarly, contents of 1-chloroanthraquinone and 1,8-dichloroanthraquinone shall be calculated.

ANNEX C

[Table 1, Sl. No. (v)]

ASSAY BY SPECTROPHOTOMETRIC METHOD AND IMPURITIES

C-1 General

Assay and impurities like 1-chloroanthraquinone and 1,8-dichloroanthraquinone are estimated by spectrophotometric method.

C-2 REAGENTS

C-2.1 1 – Chloroanthraquinone – pure (100 percent, on dry basis).

C-2.2 1,5-Dichloroanthraquinone – pure (100 percent, on dry basis).

C-2.3 1,8-Dichloroanthraquinone – pure (100 percent, on dry basis).

C-2.4 1,4-Dioxane – dry

C-3 APPARATUS

C-3.1 Infra-red Spectrophotometer – Any suitable spectrophotometer.

C-3.2 Cells - Two fixed cells for liquid with Sodium chloride window and thickness of 0.5 mm.

C-3.3 One Mark Volumetric Flask – See IS 915

C-4 PROCEDURE

C-4.1 Preparation of Sample Solution

Weigh accurately about 150 mg of dry, pulverized and sieved (100 mesh) sample in a 25 ml volumetric flask. Add about 10 ml to 15 ml dry dioxane (C-2.4) and heat to boil on a hot plate to dissolve the sample. Cool to room temperature. Dilute up to the mark and shake well.

C-4.2 Preparation of Standard Solution

Weigh accurately about 150 mg of pure 1,5-dichloroanthraquinone (C-2.2) in a 25 ml volumetric flask. Add accurately weighed about 3 mg each of pure 1-chloroanthraquinone (C-2.1) and 1,8-dichloroanthraquinone (C-2.3). Add about 10 ml to 15 ml of dry dioxane (C-2.4) and heat to boil on a hot plate to dissolve the sample. Cool to room temperature. Dilute to the mark and shake well.

C-4.3 Fill the reference cell and sample of 0.5 mm path length with dry dioxane and adjust the instrument for the 100 percent transmittance, fill the sample cell with standard solution and read the optical density at various wave lengths which correspond to different components as under:

Component	Wavelength
1-chloroanthraquinone	964 cm ⁻¹
1,8-dichloroanthraquinone	733.3 cm ⁻¹
1-chloroanthraquinone <i>plus</i> 1,5-dichloroanthraquinone	709.3 cm ⁻¹

Read the optical density for the standard solution at the above wave lengths. Similarly run the instrument with sample solution and note the optical densities at the above wave lengths.

C-5 CALCULATION

C-5.1 1-chloroanthraquinone, percent by mass = $\frac{A \times B \times C}{D \times M}$ Say, X_1

where

- A = optical density of sample at 964 cm⁻¹;
- B = percent content of 1-chloroanthraquinone in standard solution;
- C = mass of pure 1-chloroanthraquinone, in g;
- D = optical density of standard solution at 964 cm⁻¹ ; and
- M = mass of sample taken, in g.

C-5.2 1,8-dichloroanthraquinone, percent by mass = $\frac{A_1 \times B_1 \times C_1}{D_1 \times M_1}$

where

- A_1 = optical density of sample at 733.3 cm⁻¹;
- B_1 = percent content of 1,8-dichloroanthraquinone in standard solution;
- C_1 = mass of pure 1,8-dichloroanthraquinone, in g;
- D_1 = optical density of standard solution at 733.3 cm⁻¹; and
- M_1 = mass of sample taken, in g.

C-5.3

$$\left. \begin{array}{l}
 \text{1,5-dichloroanthraquinone} \\
 \text{plus} \\
 \text{1-chloroanthraquinone,} \\
 \text{percent by mass}
 \end{array} \right\} = \frac{A_2 \times B_2 \times C_2}{D_2 \times M_2} = \text{Say, } Y_1$$

where

A_2 = optical density of sample at 709.3 cm^{-1} ;

B_2 = percent content of 1-chloroanthraquinone plus 1,5-dichloroanthraquinone in standard solution;

C_2 = mass of pure isomers, in g;

D_2 = optical density of standard solution of 709.3 cm^{-1} ; and

M_2 = mass of sample taken, in g.

C-5.4 1,5-dichloroanthraquinone content, percent by mass = $Y_1 - X_1$


where

Y_1 = as calculated in **C-5.3**, and

X_1 = as calculated in **C 5.1**.

Annex D
(Foreword)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s)	:	
Signal Word	:	Warning
Hazard Statement	:	Causes skin irritation. Causes serious eye irritation. May cause respiratory irritation.
Precautionary Statement	:	Avoid breathing dust / fumes / gas / mist / vapour / spray. Wear protective gloves / eye protection / face protection. If inhaled: Remove person to fresh air and keep comfortable for breathing. If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Store locked up. Dispose of contents / container in accordance with local / regional / national / international regulations.
