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BUREAU OF INDIAN STANDARDS
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भारतीय मानक मसौदा

1- अमीनोएंथ्राक्विनोन — विशिष्टि

(IS 5042 का *दूसरा पुनरीक्षण*)

Draft Indian Standard

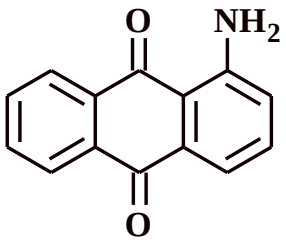
1-AMINOANTHRAQUINONE — SPECIFICATION

(*Second Revision* of IS 5042)
(ICS 71.080.99)

Dye Intermediate Sectional Committee, PCD 26	Last date for Comments 21st **February 2025**

FOREWORD
(*Formal clause to be added later*)

1-Aminoanthraquinone ($C_{14}H_9NO_2$) is a building block intermediate for various colorant and niche products. It has the following structural formula:



1-AMINOANTHRAQUINONE
(Molecular Mass 223.2)
(CAS No.: 82-45-1)

This standard was first published in 1969 and subsequently revised in 1976. The first revision was taken up to incorporate the requirements for melting point and benzene insolubles. The committee responsible for the preparation of this standard decided to update it in light of experience gained. In this (second) revision, High-performance liquid chromatography method for determination of assay and impurities such as 1-nitroanthraquinone and sum of Di-aminoanthraquinone have been incorporated.

The containers in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as given in 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-aminoanthraquinone.

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.

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 brownish-red crystalline 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-Aminoanthraquinone
(*Clause* 3.2, 5.3.1 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Matter insoluble in Chlorobenzene, percent by mass, *Max*	2.0	A	—
ii)	Purity by HPLC, percent by area, *Min*	98.0	B	—
iii)	Moisture content, percent by mass, *Max*	1.0	—	**10.3 of IS 5299**
iv)	Ash, percent by mass, *Max*	1.0	—	**12.1 of IS 5299**
v)	Impurities			
	a) 1-nitroanthraquinone, percent by area, *Max*	1.0	B	—
	b) Sum of Di-aminoanthraquinone, percent by area, *Max*	1.0		
vi)	Melting point (on dry basis)	Within 3 °C including the temperature	C	—

250 °C

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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. 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) Tare, net and gross weight
d) Batch number or lot number, in code or otherwise;
e) Month and year of manufacture;
f) Date of packing; and
g) 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 Tests for the determination of all characteristics 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 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 used 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 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.

A-3 PROCEDURE

A-3.1 Weigh accurately about 1.0 g of the sample in a 500 ml quick fit conical flask and add about 150 ml of chlorobenzene into it. Reflux for about 30 min 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°- 110°C for 4 h. Cool the crucible to room temperature in a desiccator and weigh. The increase in the mass of the crucible is the mass of insolubles.

A-4 CALCULATIONS

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

where

M_1 = Mass of insoluble in chlorobenzene, g; and

M_2 = Mass of material taken, g.

ANNEX B
[*Table* 1, *Sl No*. (ii) and (v)]
DETERMINATION OF 1-AMINOANTHRAQUINONE (PURITY), 1-NITRO ANTHRAQUINONE AND SUM OF DIAMINOANTHRAQUINONE (IMPURITIES) BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

B-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.

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-2.3 Digital Balance, 0.0001g

B-3 REAGENTS

B-3.1 2-Aminoanthraquinone — known assay

B-3.2 1-Aminoanthraquinone — known assay

B-3.3 1,2-Diaminoanthraquinone — known assay

B-3.4 Acetonitrile — known assay

B-3.5 Tetrahydrofuran (THF) — known assay

B-3.6 Water — HPLC grade

B-4 STANDARD SOLUTION PREPARATION

B-4.1 1-nitro anthraquinone

Weigh accurately 10 mg standard into 100 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

B-4.2 Diaminoanthraquinone

Weigh accurately 10 mg mix diamino anthraquinone into 100 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

B-5 1-aminoanthraquinone

Weigh accurately 20 mg sample into 100 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 — 1.0 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 — 25 min

B-11 WAVELENGTH — 242 nm

B-12 PEAK TIME

1-Amino anthraquinone	:10.4 min
1-Nitro anthraquinone	:10.8 min
1, 2-Diamino anthraquinone	:5.9 min
1, 4-Diamino anthraquinone	:6.1 min
1, 5-Diamino anthraquinone	:8.0 min

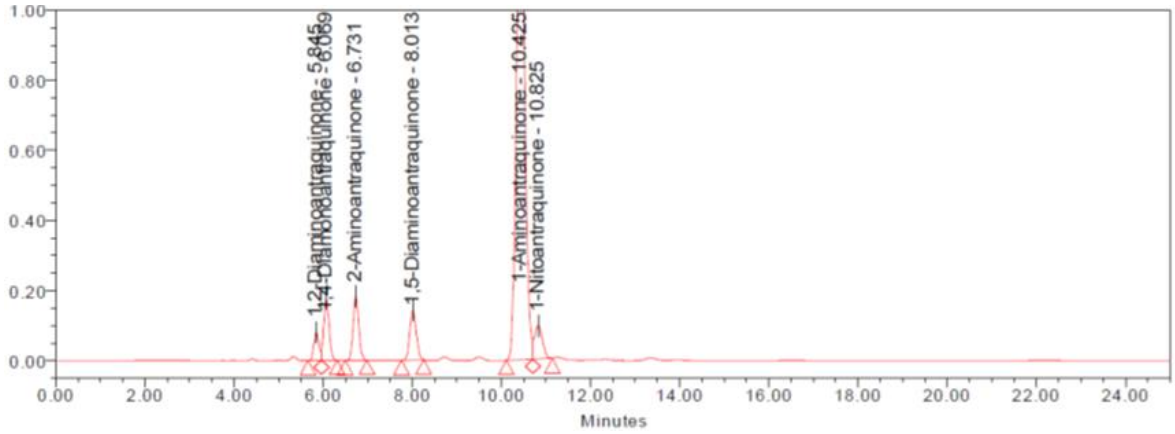

FIG 1 A TYPICAL CHROMATOGRAPH

B-13 CALCULATION

B-13.1 Calculate the peak area of individual constituent pertaining to 1-aminoanthraquinone 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-aminoanthraquinone.

1-Aminoanthraquinone, percent by area = $\frac{\text{1–Aminoanthraquinone peak area in the sample}}{\text{Sum Areas of all peaks in the chromatogram}}$ x 100

B-13.2 Similarly contents of 1-nitro anthraquinone and sum of diamino anthraquinone shall be calculated.

Annex C
DETERMINATION OF MELTING POINT
[*Table* 1, *Sl No.* (vi)]

D-1 SAMPLE PREPARATION

D-1.1 Dry the material at (105 ± 1) °C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests except for moisture determination.

D-1.2 Determine the melting point as prescribed in 9 of IS 5299.

Annex D
(*Forward*)

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.