BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

J-ACID, TECHNICAL – SPECIFICATION

(*Second Revision of* IS 6264)

(ICS 71.080.99)

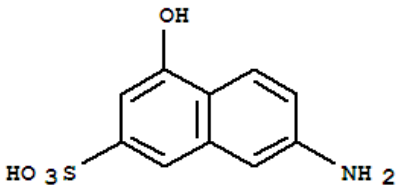
Dyes Intermediates Sectional Committee, PCD 26

Last date for comment
05 November 2023

FOREWORD

(*Formal clauses to be added later*)

J-acid is an important intermediate used for making azo dyes. Chemically, it is described as 2-amino-5-naphthol-7-sulphonic acid [formula ($C_{10}H_9NO_4S$) and molecular mass (239.3)].It has the following structural formula:



J-ACID
Molecular Mass : 239.3
CAS No. : 87-02-5

This standard was first published in 1971 and subsequently revised in 1994. In the *first* revision, the requirement of assay and matter insoluble in sodium carbonate was modified. Considering to recent development in analytical techniques in last one decade, Committee decided to revise the standard. In this (*second)* revision, requirement for determination of J-acid content by High-performance liquid chromatography method has been incorporated and requirement of matter insoluble in sodium carbonate solution have been modified.

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 B, 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.

The composition of the Committee responsible for formulation of this standard is given in Annex C (*to be added later*).

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 tests for J-acid, technical.

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*
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of dangerous goods: Part 1 Dangerous goods
IS 2552 : 1989	Steel Drums (galvanized and ungalvanized) - Specification (*third revision*)
IS 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 a paste or in the form of grey to pinkish-grey lumps or powder.

3.2 The material shall 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 J-Acid, Technical
(*Clauses* 3.2, 5.3 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Cl No. in IS 5299	Annex
(1)	(2)	(3)	(4)	(5)
i)	Assay[1)] (based on Coupling Value), percent by mass, *Min*	90	14	—
	Or			
ii)	Assay by HPLC[2)], percent by area, *Min*	98	—	A
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0.2	11.2	—

[1)]For determination of assay (based on coupling value), the 4-chloroaniline diazonium chloride solution may be used as it is more convenient.
Media Alkaline i.e. 100 ml (10 percent Na_2CO_3,).
Diazo 4-Chloroaniline (0.1 N).

[2)]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 suitable containers made of glass, wood or multi walled paper sacks or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each bag shall bear legibly and indelibly the following information:

a) Name of the Material;
b) Name of the manufacturer / supplier, complete address 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 Test for assay based on coupling method shall be conducted on each of the individual samples.

5.2.2 Tests for determination of all other characteristics namely, assay by HPLC and matter insoluble in sodium carbonate solution, given under Table 1, 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 based on coupling method if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements given under **3** and Table 1.

6 TESTS

6.1 Tests shall be carried out according to the methods prescribed in col 4 and 5 of Table 1.

6.2 Quality of Reagents

6.2.1 Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis

ANNEX A
[Table 1, *SI No.* (ii) and *Clause* 5.1]
ASSAY OF J-ACID BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

A-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.

A-2 OBJECTIVE

To determine assay of J-acid by high performance liquid Chromatography.

A-3 APPARATUS

A-3.1 Binary Gradient Liquid chromatography, system with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

A-3.1.1 *Column,* C18 column of 100 Å with length 250 mm, internal diameter 4 mm and particle size 5 µm or equivalent.

A-3.2 Analytical Balance, least count of 0.0001 g

A-4 REAGENTS

A-4.1Acetonitrile, HPLC grade

A-4.2 Water, HPLC grade

A-4.3 Tetra-*n*-Butyl Ammonium Bromide, HPLC grade

A-4.4 Sodium Di-hydrogen Orthophosphate

A-4.5 Liquor Ammonia solution

A-4.6 J-acid, reference standard

A-5 SAMPLE PREPARATION

Weigh accurately 0.0500 g Sample in 100 ml volumetric flask dissolve it in Water : Acetonitrile (2:8) and 2 drops of liquor ammonia and make up to the mark with water : acetonitrile (2:8).

A-6 BUFFER PREPARATION

Take 5.0000 g Tetra n-butyl ammonium bromide and 1.6666 g Sodium di-hydrogen ortho phosphate in 1 litre volumetric flask. Add 200ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water. Sonicate and filter though the 0.20 µm membrane.

A-7 FLOW RATE, 0.90 ml/min

A-8 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	40	60

A-9 COLUMN OVEN TEMPERATURE, Ambient

A-10 INJECTION VOLUME, 2µl

A-11 RUN TIME, 20 min

A-12 WAVE LENGTH, 254nm

A-13 PEAK TIME

2-amino-5-naphthol-7-sulphonic acid (J-acid) — 3.91 min.

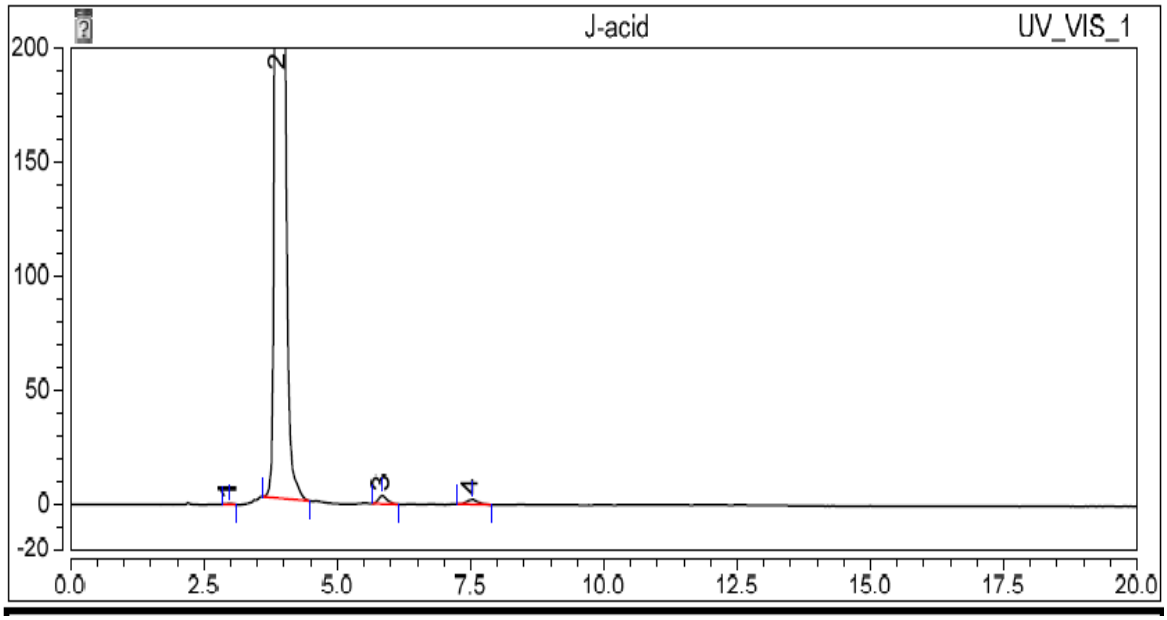


FIG 1 TYPICAL CHROMATOGRAM

A-14 CALCULATION

Calculate the peak area of individual constituent pertaining to J-acid the chromatogram of the material. The concentration of the constituent as per below calculation

$$\text{Percent of J-acid} = \frac{A}{Total\ Area} \times 100$$

where,

A = area of J acid peak in sample

ANNEX B
(*Foreword*)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s)		
Signal Word	Warning	
Hazard Statement	H314	Causes severe skin burns and eye damage
Precautionary Statement	P260	Do not breathe dust
	P262	Do not get in eyes, on skin, or on clothing
	P351	Rinse cautiously with water for several minutes
	P353	Rinse skin with water/shower
	P361	Remove/Take off immediately all contaminated clothing
	P281	Use personal protective equipment as required.
	P313	Get medical advice/attention