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Draft Indian Standard

5-SULPHOANTHRANILIC ACID - SPECIFICATION
(First Revision of IS 13841)
(ICS 71.080.99)

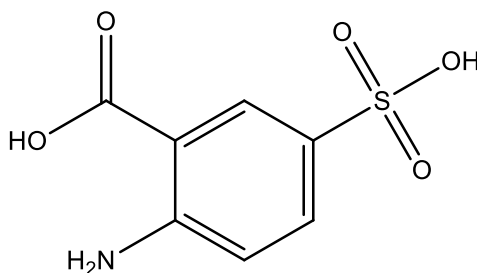
Dye Intermediates Sectional Committee,
PCD 26

Last date for comments
20 October 2023

FOREWORD

(Formal clauses to be added later)

5-sulphoanthranilic acid (C₇H₇NO₅S), is used as an intermediate for reactive dyes. It is also known as 2-amino-5-sulphobenzoic acid. It is represented by the following structural formula:



5-sulphoanthranilic acid
Molecular Mass: 217.20
CAS Number: 3577-63-7

The bags 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 and methods of sampling and test for 5-sulphoanthranilic acid.

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 editions 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
1070: 2023	Reagent Grade Water Specification (<i>fourth revision</i>)
5299: 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)
14887: 2014	Textiles – High Density Polyethylene (HDPE) / Polypropylene (PP) Woven Sacks for Packaging of 50 kg food grains — Specification (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off-White Powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirement for 5-sulphoanthranilic acid
(Clauses 3.2,5.2.2,5.3.1,5.3.2 and 6.2)

SI No.	Characteristic	Requirement	Method of Test, Ref to	
(1)	(2)	(3)	Annex (4)	IS (5)
i)	Assay by nitrite value, percent by mass (on dry basis), <i>Min</i>	90.0	A	-
	Or			
	Assay by HPLC ¹⁾ , percent area (on dry basis), <i>Min</i>	95.0	B	
ii)	Anthranilic acid content, percent area (on dry basis), <i>Max.</i>	5.0	B	-
iii)	Matter Insoluble in dilute sodium carbonate, percent by mass, <i>Max</i>	0.5	-	11.2 of IS 5299: 2001
iv)	Free Acid content (as HCl), percent by mass, <i>max.</i>	30	C	-

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 HDPE/PP Woven Sacks (*see* IS 14887). Each container shall be securely closed.

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 purity by nitrite value shall be conducted on each of the individual samples.

5.2.2 Tests for determination of all other characteristics 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 nitrite value if each of the individual test results satisfies the relevant requirements 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 in Table 1.

6 TEST METHODS

6.1 Dry the material at $105 \pm 5^\circ\text{C}$ 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.

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

6.3 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, and SI No. (i)]

DETERMINATION OF 5-SULPHOANTHRANILIC ACID CONTENT (ASSAY) BY NITRITE VALUE

A-1 Reagents

A-1.1 Concentrated Hydrochloric Acid

A-1.2 Potassium Bromide

A-1.3 Standard Sodium Nitrite Solution – 0.1N

A-1.4 Potassium Starch Iodide Indicator Paper

A-1.5 Ice

A-2 Procedure — Weight 10-14 gm dry powder in 250 ml glass beaker. Add distilled water approx. 150 cc and stir with glass rod to make a smooth slurry. Add 20 % soda ash solution (approx. 7-10 cc) to dissolve the powder to make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 cc of the solution by using pipette in to 1000 ml beaker. Add 200-250 ml distilled water. Add ice cubes to make the temperature around 10°C. Weigh and add 1 gm Potassium bromide into the cold solution. Add Hydrochloric acid to make the pH acidic (pH around 2-2.5 on pH paper) approx. 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the endpoint to put the spot-on starch iodide paper, the end- point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

A-2.3 Calculation

Assay (by nitrite value), percent by mass (on dry basis) = $\frac{V \times N \times 217.20}{M}$

where

V = Volume of standard sodium nitrite solution used in the titration, ml

N = Normality of sodium nitrite solution, and

M = Mass of the dry material taken for the test, g

ANNEX B

[Table 1, and SI No. (ii)]

DETERMINATION OF 5-SULPHOANTHRANILIC ACID CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

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.

B-2 Objective

To determine Purity of 5-sulphoanthranilic acid by high performance liquid Chromatography

B-3 Apparatus

B-3.1 Analytical balance

B-3.2 Isocratic Liquid chromatography system with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-3.2.1 Column, C18, 250 × 4.6mm, 5µm or equivalent

B-4 Reagent

B-4.1 Tetrabutyl ammonium hydrogen sulphate, Analytical grade (AR)

B-4.2 Dipotassium hydrogen phosphate, HPLC grade

B-4.3 Acetonitrile, HPLC grade

B-4.4 Water, HPLC grade

B-4.5 5-sulphoanthranilic acid, Reference standard

B-4.6 Anthranilic acid, Reference standard

B-5 Standard Preparation

Weigh accurately 0.010gm (10mg) Anthranilic acid in 100ml volumetric flask dissolve it in Water and make up to 100 ml with water.

B-6 Sample Preparation

Weigh accurately 0.010gm (10mg) dry 5-sulphoanthranilic acid in 100ml volumetric flask dissolve it in Water and make up to 100 ml with water.

B-7 Buffer Preparation

Take 12 g Tetrabutyl ammonium hydrogen sulphate (TBAHS) and 29 g Dipotassium hydrogen phosphate (K_2HPO_4) in 190 ml Water and dissolve (stock solution).

B-8 Flow Rate 1ml/min

B-9 Mobile Phase 100ml water + 4ml Buffer + 40ml Acetonitrile

B-10 Column Oven Temperature 26°C

B-11 Injection Volume 20 µl

B-12 Run Time 10 min *maximum*

B-13 Wavelength 254 nm

B-14 Peak Time 5-sulphoanthranilic acid – 4.3 min approx.
Anthranilic acid – 4.8 min approx.

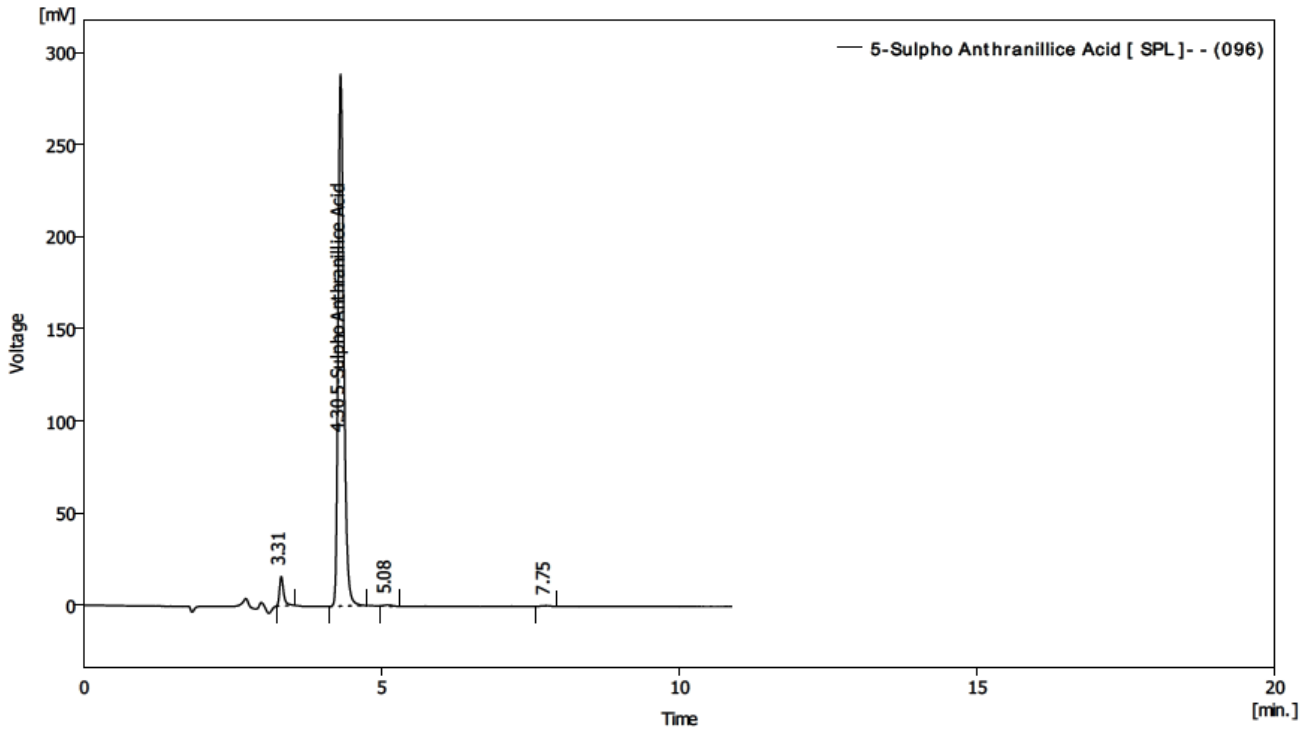


FIG 1 TYPICAL CHROMATOGRAM

B-15 Calculation

B-15.1 Calculate the peak area of individual constituent pertaining to 5-sulphoanthranilic acid 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 5-sulphoanthranilic acid.

$$\text{Percent of 5-sulphoanthranilic acid} = \frac{A}{\text{Total Area}} \times 100$$

where,

A = Area of 5-sulphoanthranilic acid peak in Sample

B-15.2 Similarly contents of Anthranilic acid shall be calculated.

ANNEX C

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

Determination of Free acid content (as HCl)

C-1 Reagents

C-1.1 Standard Sodium Hydroxide Solution, 1.0 N

C-1.2 Phenolphthalein Indicator

C-2 Procedure

Weigh 3 gm of sample in a beaker, add 150 to 160 ml water and dissolve it. Take 1.0 N sodium hydroxide solution in burette. Titrate the prepared solution against 1.0 N sodium hydroxide solution with constant stirring by using a magnetic stirrer. The end point shows faint pink colouration, check the sodium hydroxide consumed by burette reading.

C-3 CALCULATION

Free acid content as HCl (percent by mass): $\frac{V \times 217.20 \times N \times 100}{M}$

where,

V = Volume of standard sodium hydroxide solution used in the titration, ml

N = Normality of sodium hydroxide solution, and

M = Mass of the dry material taken for the test, g

ANNEX D (Forward)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :



Signal Word : **WARNING**

Hazard Statement : H315 Causes skin irritation.
H319 Causes serious eye irritation.

Precautionary Statement : **Prevention**

P264: Wash hands thoroughly after handling.

P280: Wear protective gloves/protective clothing/eye protection/face protection.

Response

P305 +P351 +P338: IF IN EYES: Rinse cautiously with water for several minutes. Remove Contact lenses, if present and easy to do. Continue rinsing.

P337+P313: IF eye irritation persists: Get medical advice/attention.

P362: Take off contaminated clothing and wash before reuse.

P302 +P352: IF ON SKIN: wash with plenty of soap and water

P332+P313: IF SKIN irritation occurs: Get medical advice/attention.
