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Draft Indian Standard

GAMMA ACID — SPECIFICATION
(Second Revision of IS 8627)

(ICS 71.080.99)

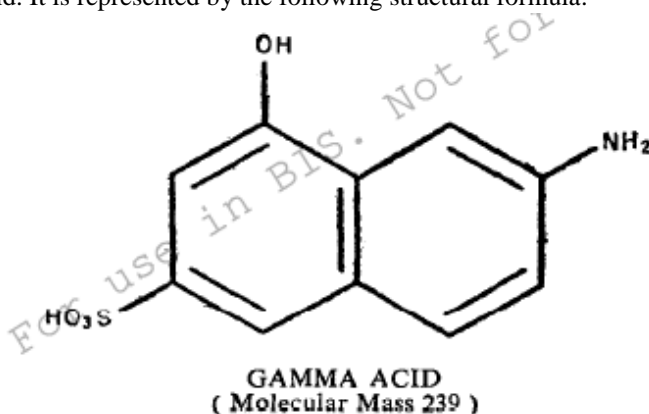
Dye Intermediates Sectional Committee,
PCD 26

Last date for comments
17 October 2023

FOREWORD

(Formal clauses to be added later)

Gamma acid (C₁₀H₉NO₄S) is an important intermediate used for making azo dyes. It is described as 2-amino-8-naphthol-6-sulphonic acid. It is represented by the following structural formula:



CAS No. 90-51-7

This standard was originally published in 1977 and revised in 1986. The *first* revision was taken up to modify the requirement of matter insoluble in sodium carbonate solution and also to incorporate the necessary details for assay. However, the Committee envisage to incorporate the chromatographic test method in near future after identification of various impurities.

Considering to recent development in analytical techniques in last one decade, Committee decided to revise the standard. In this revision (*second*), specification for additional impurity of Bronner's Acid, High-performance liquid chromatography method for assay determination are incorporated.

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 E, 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, the methods of sampling and test for gamma acid.

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 agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
IS 1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods Part 1 Dangerous goods (<i>first revision</i>)
IS 2552: 1989	Steel drums (galvanized and ungalvanized) — Specification (<i>third revision</i>)
IS 5299: 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless to pale brown powder or scales. It shall be free from dust and other 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 of Table 1.

Table 1 Requirements for *Gamma Acid*
(*Clause 3.2, 5.3.1, 5.3.2 and 6.2*)

SI No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Matter insoluble in sodium carbonate solution, percent by mass, <i>Max</i>	0.2	A
ii)	Assay, percent by mass, <i>Min</i>	85	B
iii)	Assay by HPLC ¹⁾ , percent Area, <i>Min</i>	98	C
iv)	Bronner's acid content, percent by mass, <i>Max</i>	0.2	D

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 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) Lot or batch number;
- e) Month and year of manufacturing;
- 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 Tests for description, assay and matter insoluble in sodium carbonate solution shall be conducted on each of the individual samples.

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 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 Preparation of sample

Dry the material at $105 \pm 1^\circ\text{C}$ 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 Tests shall be carried out according to the methods prescribed in col 4 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 MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

A-1 REAGENTS

A-1.1 Sodium Carbonate Solution — 20 percent (*w/v*).

A-1.2 Brilliant Yellow Indicator Paper

A-2 PROCEDURE

A-2.1 Weigh accurately about 12 g of the prepared sample in **6.2** and transfer to a 500 ml beaker. Paste well with about 200 ml of water. Add sodium carbonate solution till alkaline to brilliant yellow indicator paper. Heat it to dissolve the material completely. Filter hot solution through counterpoised filter paper and wash the residue with hot water till the filtrate is free from alkali. Dry the residue at $(100 \pm 5)^\circ\text{C}$ to constant mass.

A-2.2 Transfer quantitatively the filtrate and washings together into 500 ml volumetric flask and dilute with water up to the mark at room temperature. Mix it well. Use the solution for test in **B**.

A-3 CALCULATION

Matter insoluble in sodium carbonate solution, percent by mass = $\frac{m \times 100}{M}$

where

m = mass of the residue, g, and

M = mass of the material taken for the test, g.

ANNEX B

[Table 1, SI No.(ii)]

DETERMINATION OF ASSAY BY COUPLING VALUE

B-1 REAGENTS

B-1.1 Acetic Acid Solution, 10 percent (*v/v*).

B-1.2 Sodium Acetate Solution, 20 percent (*w/v*).

B-1.3 Standard p-Nitroaniline Diazo Solution, 0.1 N.

B-1.4 H-Acid, Neutral

Dissolve 0.5 g of purified H-Acid in 10 ml of 20 percent (*w/v*) sodium acetate solution. Dilute to 20 ml with 20 percent (*w/v*) sodium acetate solution and mix well.

B-1.5 Common Salt (*see* IS 797).

B-2 Procedure

B-2.1 Pipette out 50 ml aliquot from volumetric solution prepared for the determination of insoluble in sodium carbonate solution (*see* A-2.2) into a 1 liter breaker. Add about 300 ml of water and render faintly acidic to litmus paper with acetic acid. Add about 175 ml of sodium acetate solution and washed ice to cool to 0 to 5 °C. Titrate while stirring mechanically at 0 to 5 °C with *p*-nitroaniline diazo using neutral H-acid as indicator. Towards the end, add 100 g of common salt to precipitate the dye. The end point is taken when a slight excess of diazo is shown for 5 min period by the following test.

B-2.2 Spot a drop of coupling slurry alongside a drop of H-acid indicator (solution neutral) on filter paper (Whatman No. 1 or equivalent) so that outspreads merge. The formation of a faint red purple line at the inter junction indicates excess diazo.

B-3 Calculation

$$\text{Assay (coupling value), percent by mass} = \frac{V_1 \times N_1 \times 239}{M}$$

where

V_1 = volume of the *p*-nitroaniline diazo solution used in the test, ml

N_1 = normality of *p*-nitroaniline diazo solution, and

M = mass of the material taken for the test (*see* A-2.1), in g.

ANNEX C

[Table 1 and Sl No.(iii)]

DETERMINATION OF ASSAY OF GAMMA ACID BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

C-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.

C-2 OBJECTIVE

To determine assay of gamma acid by high performance liquid chromatography.

C-3 APPARATUS

C-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.

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

C-4 Reagent

C-4.1 Acetonitrile, HPLC grade

C-4.2 Water, HPLC grade

C-4.3 Tetrabutylammonium hydrogen sulphate, HPLC grade

C-4.4 Gamma acid, known purity

C-5 SAMPLE PREPARATION

Weigh accurately 0.0500 g (50 mg) Sample in 100 ml volumetric flask dissolve it in water: acetonitrile (2:8) and make up to the mark with water: acetonitrile (2:8).

C-6 BUFFER PREPARATION

Take 2g Tetrabutylammonium hydrogen sulphate in 1 liter volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water.

C-7 FLOW RATE 0.80ml/min

C-8 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	15	85

C-9 COLUMN OVEN TEMPERATURE Ambient

C-10 INJECTION VOLUME 2µl

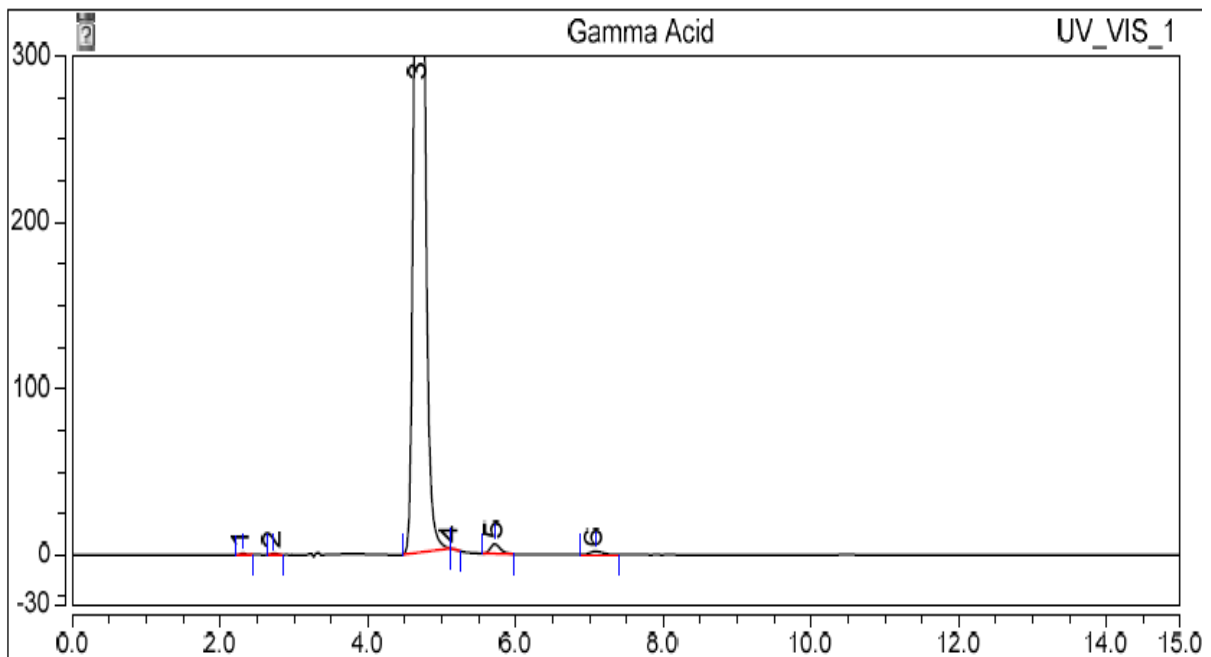
C-11 RUN TIME 15 min

C-12 WAVE LENGTH 240 nm

C-13 PEAK TIME

Gamma acid 4.69 min

Bronner's Acid 5.73 min



C-14 CALCULATION

Calculate the peak area of individual constituent pertaining to Gamma Acid the chromatogram of the material. The concentration of the constituent may be obtained as per below calculation.

$$\% \text{ of Gamma Acid} = \frac{A}{\text{Total Area}} \times 100$$

where,

A = Area of Gamma acid peak

ANNEX D

[Table 1 and SI No. (iv)]

DETERMINATION OF BRONNER'S ACID CONTENT IN GAMMA ACID BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

D-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.

D-2 OBJECTIVE

To determine Bronner's acid in Gamma acid by high performance liquid Chromatography

D-3 APPARATUS

D-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.

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

D-4 REAGENT

D-4.1 Acetonitrile, HPLC grade

D-4.2 Water, HPLC grade

D-4.3 Tetrabutylammonium hydrogen sulphate, HPLC grade

D-4.4 Bronner's acid, Reference Standard

D-5 STANDARD PREPARATION

Weigh accurately 0.0050 g (5 mg) in 100 ml volumetric flask dissolve it in Water: Acetonitrile (2:8) & make up to the mark with Water: Acetonitrile (2:8).

D-6 SAMPLE PREPARATION

Weigh accurately 0.0500g (50 mg) Sample in 100 ml volumetric flask dissolve it in Water: Acetonitrile (2:8) & make up to the mark with Water: Acetonitrile (2:8).

D-7 BUFFER PREPARATION

Take 2 g Tetrabutylammonium hydrogen sulphate in 1 liter volumetric flask. Add 200 ml HPLC grade water & complete dissolve it. Make total volume with HPLC grade water. Then filter the solution through 0.2 µ membrane.

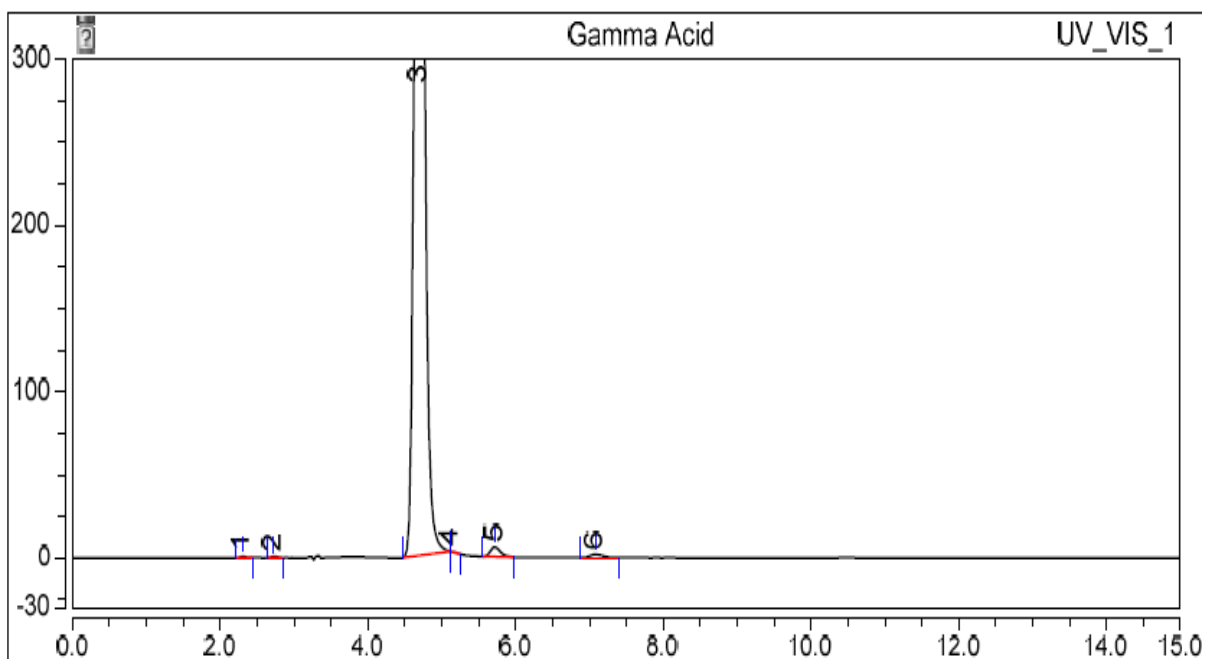
D-8 FLOW RATE

0.80ml/min

D-9 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	15	85

D-10 COLUMN OVEN TEMPERATURE	Ambient	
D-11 INJECTION VOLUME	2µl	
D-12 RUN TIME	15 min	
D-13 WAVE LENGTH	240 nm	
D-14 PEAK TIME	Gamma acid	4.69 min
	Bronner's Acid	5.73 min



D-15 CALCULATION

Calculate the peak area of individual constituent pertaining to Bronner's Acid 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 pure Bronner's Acid.

$$\text{percent of Bronner's Acid} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where,

- A_1 = Area of Standard Bronner's Acid
- V_1 = Injection Volume of Standard Bronner's Acid
- W_1 = Weight of Standard Bronner's Acid
- B_1 = Total Volume of Standard Bronner's Acid
- A_2 = Area of Bronner's Acid peak in Sample
- V_2 = Injection Volume of Sample
- W_2 = Weight of Sample
- B_2 = Total Volume of Sample

ANNEX E
(Forward)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :



Signal Word : **Danger**

Hazard Statement : H314 Causes severe skin burns and eye damage.

Precautionary Statement : 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
P281 Use personal protective equipment as required.
P313 Get medical advice/attention
