

BUREAU OF INDIAN STANDARDS
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Draft Indian Standard
R SALT, TECHNICAL — SPECIFICATION
(Second Revision of IS 8626)
(ICS 71.080.99)

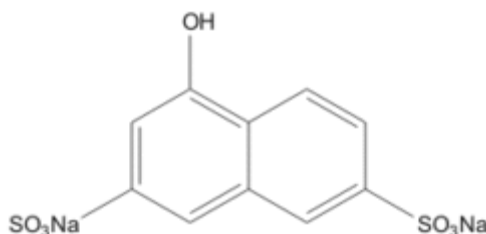
Dye Intermediates Sectional Committee,
PCD 26

Last date for Comments
20 October 2023

FOREWORD

(Formal clauses to be added later)

R Salt ($C_{10}H_6O_7S_2Na_2$) is a dye intermediate. It is disodium salt of 2-naphthol-3, 6-disulphonic acid. It is represented by the following structural formula:



R SALT
Molecular Mass, Free Acid, 304
CAS No. 148-75-4

This standard was originally published in 1977 and subsequently revised in 1987. This (*second*) revision has been undertaken to update the standard by considering the development in analytical techniques in last one decade and use of more sophisticated instrument to determine assay and impurity profile, Committee decided to revise specification of R Salt Test method for Purity and impurities like β Naphthol, G salt and Schaffer's acid content using High- performance liquid chromatography (HPLC) is 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 and methods of sampling and test for R salt, technical.

2 REFERENCES

The following 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:

<i>IS No.</i>	<i>Title</i>
1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
2552: 1989	Steel drums (galvanized and ungalvanized) — Specification (<i>third revision</i>)
5299: 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)
5762: 1970	Methods for determination of melting point and melting range

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off white powder or off white moist cake.

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

Table 1 Requirements for R Salt, Technical

(Clause 3.2, 5.2.2, 5.3.1, 5.3.2 and 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), <i>Min</i>	65	A
ii)	Matter insoluble in sodium hydroxide solution , percent by mass , <i>Max</i>	0.2	B
iii)	Assay (by HPCL), percent by mass, <i>Min</i>	98	C
iii)	β - Naphthol content by HPLC ¹⁾ , percent w/w by mass, <i>Max</i>	0.5	D
iv)	G salt content by HPLC , percent w/w by mass, <i>Max</i>	1.0	
v)	Schaeffer's salt content by HPLC , percent w/w by mass, <i>Max</i>	1.0	

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 drum (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

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

5.2.2 Tests for the determination of all other characteristics, namely, β -naphthol content, solubility in sodium hydroxide solution, G salt and Schaeffer's salt content given under Table 1, shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Sample*

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, 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 according to the methods prescribed in col 4 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, SI No.(i)]

DETERMINATION OF ASSAY BY COUPLING VALUE

A-1 OBJECTIVE

To determine Assay of R Salt by Coupling Value titration.

A-2 APPARATUS AND REAGENTS

A-2.1 Watch Glass or Paper Boat [Butter Paper/Glossy Paper]

A-2.2 Wash Bottle

A-2.3 Ice

A-2.4 500 ml or 1000 ml Glass Beaker

A-2.5 Glass Rod

A-2.6 10 ml Graduated Pipette

A-2.7 Magnate and Magnetic Stirrer

A-2.8 0.1N p-Nitro Aniline diazo solution [PNA DIAZO] [Standardized]

A-2.9 Sodium Acetate [Hydrous]

A-3.0 Spot Paper [Whatman No.1]

A-3.1 0.1 percent H Acid Solution

A-3.2 Starch Iodide Paper (SI Paper)

A-3.3 Sodium Carbonate solution (10 percent)

A-3 PROCEDURE

Weight accurately about 1 g of sample and transfer to a 500 ml beaker with the help of water. Add 100 ml of Distilled water to dissolve it then add 25 to 30 gm Sodium acetate (Hydrous). Stirrer on magnetic stirrer till the solution is clear. Then add ice to cool it. Add 0.1N p-Nitro Aniline diazo solution to it using 10 ml graduated pipette. Check the spot on the Spot paper, it should be positive to Coupler Side (1 percent H-Acid Solution) and negative to diazo side (i.e. for p-Nitro Aniline diazo). It should be remain positive to Coupler Side (1 percent H-Acid Solution) for 10 min. (If not then add more p-Nitro Aniline solution till the Spot of reaction show positive to Coupler Side for 10 min). Note the reading

A-4 CALCULATION

Purity (by nitrite value), percent by mass = $\frac{V_1 \times N_1 \times 304}{M \times 10}$

Where,

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

N_I = Normality of sodium nitrite solution; and

M = Mass of the material taken for the test, gm

ANNEX B

[Table 1, SI No. (ii)]

DETERMINATION OF MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

B-1 REAGENT

Sodium Hydroxide solution — Approximately 5 percent (w/v) filtered free from suspended impurities.

B-2 PROCEDURE

Weigh accurately 10 to 15 g thoroughly mixed sample into a 1000 ml beaker, add 300 ml water and sufficient 5 percent sodium hydroxide solution to make the solution alkaline to brilliant yellow paper. Heat the solution to 60 °C until the sample is dissolved and filter it through sintered crucible of porosity G4, wash residue well with hot water, dry at (100 ± 5) °C, cool and weigh.

B-3 CALCULATION

Matter insoluble in sodium hydroxide solution percent by mass = $\frac{M_1 \times 100}{M_2}$

where

M_1 = Mass in g of the residue; and

M_2 = Mass in g of sample taken for the test.

ANNEX C

[Table 1 and SI No. (iii)]

TO DETERMINE PURITY OF R SALT 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 Purity of R Salt by high performance liquid Chromatography.

C-3 APPARATUS — 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-4 COLUMN — C18 100Å, 250 × 4.6mm, 5µm or equivalent

C-5 REAGENTS

C-5.1 Acetonitrile, HPLC grade

C-5.2 Water, HPLC grade

C-5.3 Tetrabutylammonium hydrogen sulphate, HPLC Grade

C-5.4 R Salt, Reference standard

C-6 REFERENCE STANDARD SOLUTION PREPARATION

Weigh accurately 0.0500 gm reference standard R salt in 100 ml volumetric flask dissolve it in Water : Acetonitrile (1:1) and make up to the mark with Water : Acetonitrile (1:1).

C-7 SAMPLE PREPARATION

Weigh accurately 0.0500 gm (50mg) of Sample in 100ml volumetric flask dissolve it in Water : Acetonitrile (1:1) & make up to the mark with Water : Acetonitrile (1:1).

C-8 BUFFER PREPARATION

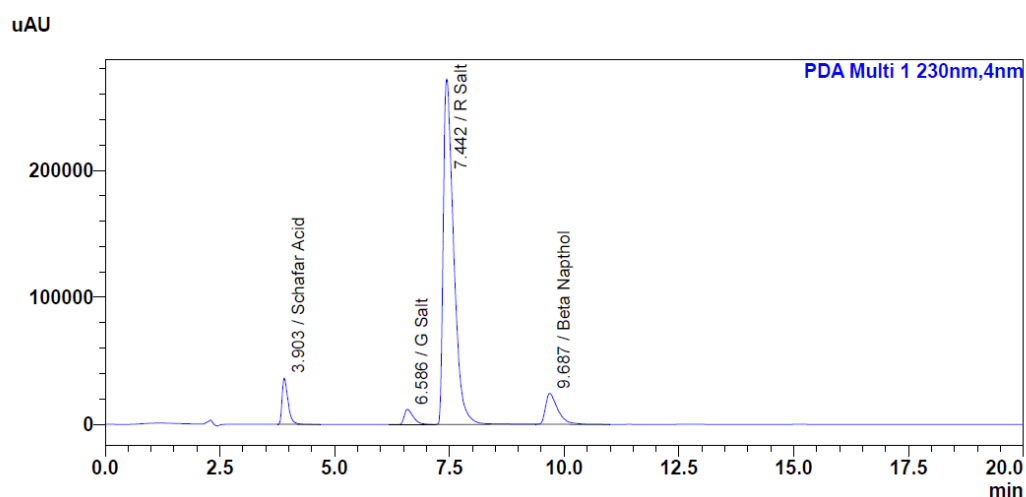
Take 2 gm Tetrabutylammonium hydrogen sulphate in 1litre volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water. Then filter the solution through 0.2 μ membrane.

C-9 FLOW RATE 1.00ml/min

C-10 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	35	65

C.11 Column Oven Temperature	Ambient	
C.12 Injection Volume	2 μ l	
C.13 Run Time	20 minutes	
C.14 Wave Length	230nm	
C.15 Peak Time	R Salt	7.44minutes



C-16 CALCULATION

Calculate the peak area of individual constituent pertaining to R Salt 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 purity R Salt.

$$\% \text{ Area of R Salt} = \frac{A}{\text{Total Area}} \times 100$$

where,

A = Area of R salt peak

ANNEX D

[Table 1 and SI No. (iii)]

TO DETERMINE SCHAEFFER'S ACID, G SALT and β -NAPHTHOL IN R SALT 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 Schaeffer's Acid, G Salt and β -Naphthol in R Salt by High-performance liquid Chromatography

D-3 APPARATUS

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-4 COLUMN — C18 100Å, 250 × 4.6mm, 5µm or equivalent

D-5 REAGENT

D-5.1 Acetonitrile, HPLC grade

D-5.2 Water, HPLC grade

D-5.3 Tetrabutylammonium hydrogen sulphate, HPLC Grade

D-5.4 Schaeffer's Acid, Reference standard

D-5.5 G Salt, Reference standard

D-5.6 β -Naphthol, Reference standard

D-6 STANDARD SOLUTION PREPARATION

D-6.1 Schaeffer's acid standard solution

Weigh accurately 0.0050 gm (5mg) of standard Schaeffer's Acid in 100 ml volumetric flask dissolve it in Water : Acetonitrile (1:1) and make up to the mark with Water : Acetonitrile (1:1)

D-6.2 G salt standard solution

Weigh accurately 0.0050 gm (5mg) of standard G Salt in 100 ml volumetric flask dissolve it in Water : Acetonitrile (1:1) and make up to the mark with Water : Acetonitrile (1:1).

D-6.3 β -Naphthol standard solution

Weigh accurately 0.0050gm (5mg) of standard β -Naphthol in 100 ml volumetric flask dissolve it in Acetonitrile and make up to the mark with Acetonitrile.

D-6.4 Take 10 ml of above solution of Schaeffer's Acid Solution, G Salt Standard Solution and β -Naphthol standard solution in 100 ml volumetric flask and make up to the mark with Acetonitrile – Mixture of standard solution. If require dilute the standard solution.

D-7 SAMPLE PREPARATION

Weigh accurately 0.0500 gm (50mg) of Sample in 100 ml volumetric flask dissolve it in Water: Acetonitrile (1:1) and make up to the mark with Water: Acetonitrile (1:1).

D-8 BUFFER PREPARATION

D-8.1 Take 2 gm Tetrabutylammonium hydrogen sulphate in 1litre volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water. Then filter the solution through 0.2 μ m membrane.

D-9 Flow Rate 1.00ml/min

D-10 Mobile Phase

Time	Acetonitrile	Buffer
0.01	35	65

D-11 Column Oven Temperature

Ambient

D-12 Injection Volume

2 μ l

D-13 Run Time

20 minutes

D-14 Wave Length

230 nm

D-15 Peak Time

Schaeffer's Acid

3.93 minutes

G Salt

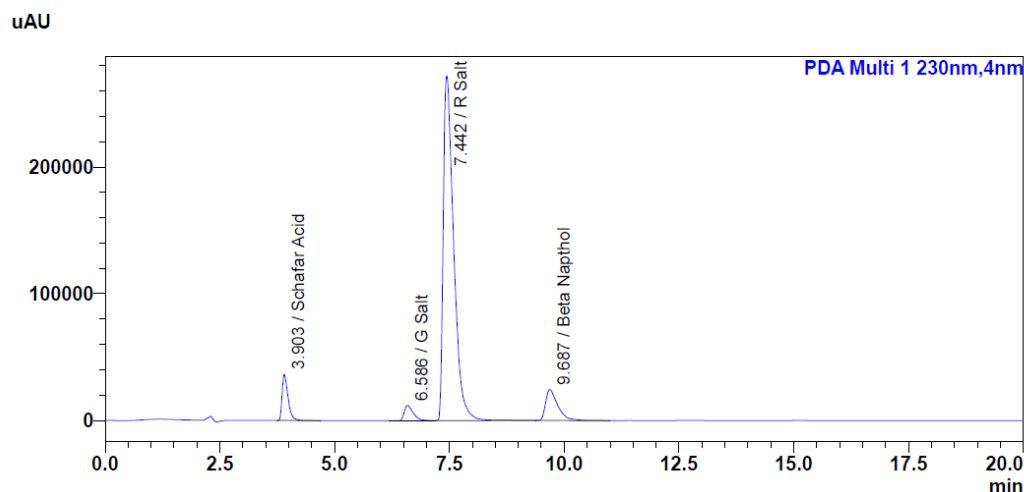
6.58 minutes

R Salt

7.44 minutes

β -Naphthol

9.68 minutes



D-16 CALCULATION

D-16.1 Calculate the peak area of individual constituent pertaining to G Salt 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 purity G Salt.

$$\% \text{ Area of G Salt} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where,

- A_1 = Area of Standard Galt
- V_1 = Injection Volume of Standard G Salt
- W_1 = Weight of Standard G Salt
- B_1 = Total Volume of Standard G Salt
- A_2 = Area of G salt peak in Sample
- V_2 = Injection Volume of Sample
- W_2 = Weight of Sample
- B_2 = Total Volume of Sample

D-16.2 Similarly, Schaeffer's acid & β Naphthol content shall be calculated.

ANNEX E

(Foreword)

Pictograms, signal word, hazard statement and precautionary statement

Pictogram(s) :



Signal Word : **WARNING**

Hazard Statement : H317 May cause an allergic skin reaction.
H319 Causes serious eye irritation

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
