**IS 8626 : 2024**

***भारतीय मानक***

***Indian Standard***

 **आर साल्ट, तकनीकी — विशिष्टि**

 (*दूसरा पुनरीक्षण*)

**R Salt, Technical — Specification**

 (***Second*** *Revision*)

ICS 71.080.99

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भारतीय मानक ब्यूरो

BUREAU OF INDIAN STANDARDS

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**December 2024 Price Group X**

Dye Intermediates Sectional Committee, PCD 26

**FOREWORD**

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

R Salt (C10H6O7S2Na2) 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 first published in 1977 and subsequently revised in 1987. The revision has been undertaken to update the standard by considering the development in analytical techniques in last few decades and use of more sophisticated instrument to determine purity and impurity profile. In this (second) revision, a new characteristic which is determination of purity by High Performance Liquid Chromatography and its requirement has been incorporated and existing method of test for determination of β- Napthol, G salt and Schaeffer’s salt content has been modified to High performance liquid chromatography method.

The containers 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 D, 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 E.

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.

*Indian Standard*

R SALT, TECHNICAL — SPECIFICATION

(*Second Revision*)

**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:

|  |  |
| --- | --- |
| *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)* |
| 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.**(1) | **Characteristic**(2) | **Requirement**(3) | **Method of Test,** **Ref to** **Annex**(4) |
| i)  | Assay, percent by mass (on dry basis), *Min* | 65 | A |
| ii) | Matter insoluble in sodium hydroxide solution, percent by mass, *Max* | 0.2 | B |
| iii) | Purity (by HPLC), percent by area, *Min* | 98 | C |
| iv)  | β- Napthol content by HPLC, percent by area, *Max* | 0.5 | C |
| v)  | G salt content by HPLC, percent by area, *Max* | 1.0 |
| vi)  | Schaeffer’s salt content by HPLC, percent by area, *Max* | 1.0 |

**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.

**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** Tests for the determination of all characteristics, namely, assay, purity (by HPLC), β-napthol content, matter insoluble in sodium hydroxide solution, G salt and Schaeffer’s salt content 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 according to the methods prescribed in co1 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 distilled 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 = $\frac{V\_{1}×N\_{1}× 304 }{M x 10}$

Where,

*V1 =* Volume of standard sodium nitrite solution used in the titration, ml;

*N1*= 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 (m*/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 oC until the sample is dissolved and filter it through sintered crucible of porosity G4, wash residue well with hot water, dry at (100 ± 5) oC, cool and weigh.

**B-3 CALCULATION**

Matter insoluble in sodium hydroxide solution, percent by mass = $\frac{M\_{1}×100}{M\_{2}}$

where

*M1* = Mass in g of the residue; and

*M2* = Mass in g of sample taken for the test.

**ANNEX C**

[*Table* 1 and *SI No. (iii), (iv), (v) and (vi)*]

**DETERMINATION OF PURITY AND IMPURITIES 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 and Schaeffer’s Acid, G Salt and β-Naphthol in 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

**D-5.5** **Schaeffer’s Acid,** Reference standard

**D-5.6** **G Salt,** Reference standard

**D-5.7** **β-Naphthol,** Reference standard

**C-6 REFERENCE STANDARD SOLUTION PREPARATION**

Weigh accurately 0.0500 gm (50 mg) reference standard in 100 ml volumetric flask and 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 100 ml volumetric flask and 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** | 2µl |
| **C.13** | **Run Time**  | 20 minutes |
| **C.14** | **Wave Length**  | 230nm |
| **C.15** | **Peak Time**  | R SaltSchaeffer’s AcidG Saltβ-Naphthol | 7.44minutes3.93 minutes6.58 minutes9.68 minutes |



FIG 1 A Typical Chromatogram

**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.

Purity of R Salt, percent by area = $\frac{A}{Total Area}×100$

 where,

 A = Area of R salt peak

**C-16.1** Similarly, Schaeffer’s acid, G salt and β-Naphthol content shall be calculated.

**ANNEX D**

*(Foreword)*

**Pictograms, signal word, hazard statement and precautionary statement**

|  |  |
| --- | --- |
| **Pictogram(s) :**  | GHS-pictogram-exclam.svg  |
| **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 clothingP351 Rinse cautiously with water for several minutesP353 Rinse skin with water/showerP281 Use personal protective equipment as required.P313 Get medical advice/attention |

**ANNEX E**

(*Foreword*)

**COMMITTEE COMPOSITION**

Dye Intermediates Sectional Committee, PCD 26

|  |  |
| --- | --- |
| *Organization* | *Representative(s)* |
| Institute of Chemical Technology, Mumbai | PROF. GANAPATI SUBRAY SHANKARLING **(*Chairperson*)** |
| Aarti Industries Limited, Mumbai | DR. VAISHALI BHANDARY SHRI JAYESH VASHI (*Alternate)* |
| Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar | SHRI MANSUKH H. VEKARIA |
| Archroma India Private Limited, Thane | DR. RAJESH RAMAMURTHY SHRI HEMANT MHADESHWAR *(Alternate)* |
| Atul Limited, Gujarat | SHRI MAYUR DESAI DR. RAIS KHAN *(Alternate)* |
| BASF India Limited, Mumbai | SHRI AMARISH SAMEL |
| Central Revenue Control Laboratory, New Delhi | SHRI V. SURESH SHRI SHIVRAJ SINGH *(Alternate)*SHRI MRITUNJOY MAITY *(Alternate)* |
| Colourtex Industries Limited, Mumbai | DR PANKAJ DESAI SHRI R. K. JAISWAL *(Alternate)* |
| Deepak Nitrite Limited, Vadodara | SHRI SAILASH RAVAL SHRI RAJENDRA SHINDE *(Alternate)* |
| Defence Research Development Organization, Ministry of Defence, New Delhi | DR. PRADEEP K. GUPTA |
| Dystar, Mumbai | DR. MONIKA SINGH |
| Gujarat Dyestuffs Manufacturers Association, Ahmedabad | SHRI NILESH DAMANI SHRI ANKIT PATEL *(Alternate)* SHRI SHIRIN PARIKH *(Alternate)* |
| Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad | SHRI R. M. PATEL SHRI C. S. PATEL *(Alternate)* |
| Gujarat Pollution Control Board, Gandhinagar, Ahmedabad | SHRI D.M. THAKER |
| Heubach Colour Private Limited, Mumbai | SHRI J. I. SEVAK |
| Indian Chemical Council, Mumbai | SHRI  DHRUMIL SONI MS. SHRADDHA RANE  |
| Jay Chemicals Industries Private Limited, Ahmedabad | SHRI VILPESH YADAV  SMT MAITRI VYAS *(Alternate)* |
| Kiri Industries Limited, Ahmedabad | DR GIRISH H TANDEL MS. SHRADDHA RANE *(Alternate)* |
| Meghmani Dyes and Intermediates Limited, Ahmedabad | SHRI MANOHAR MAHESHWARI SHRI RAMESH SHINGARE *(Alternate)* |
| Ministry of Environment Forest and Climate Change, New Delhi | SHRI N. SUBRAHMANYAM |
| NimkarTek Technical Services Private Limited, Mumbai | SHRI ULLHAS NIMKAR MS. ANAGHA NIMKAR *(Alternate)* |
| Sudarshan Chemical Industries Limited, Pune | DR. R. SRIDHARAN |
| The Bombay Textile Research Association, Mumbai | MS. SHITAL PALASKAR |
| The Dyestuff Manufactures Association of India Office, Mumbai | SHRI MANOJ SAPTARSHI |
| BIS Director General | SHRI CHINMAY DWIVEDI, SCIENTIST ‘E’/ DIRECTOR AND HEAD (PCD) [REPRESENTING DIRECTOR GENERAL (*Ex-Officio*)] |
|  *Member Secretary* MS ANMOL AGARWALSCIENTIST B/ASSISTANT DIRECTOR (PETROLUEM, COAL AND RELATED PRODUCTS), BIS |