**IS 6265: 2024**

***भारतीय मानक***

***Indian Standard***

**क्विनिज़रिन, तकनीकी — विशिष्टि**

(*पहला पुनरीक्षण*)

**Quinizarin, Technical — Specification**

 (*First 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 (First 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.

Quinizarin or 1, 4-dihydroxyanthraquinone (C14H8O4) is a very important dye intermediate used extensively in the manufacture of vat dyes, disperse dyes, etc. It also finds use as an antioxidant in synthetic lubricants. When heated it emits acrid fumes. It is represented by the following structural formula:



QUINIZARIN

(Molecular Mass: 240.21)

CAS No.: 81-64-1

**This standard was originally published in 1971. The committee responsible for the preparation of this standard decided to update it in light of experience gained. In this (*first*) revision, high-performance liquid chromatography method for determination of assay has been incorporated and requirements for characteristics such as matter insoluble in toluene, moisture content and ash content are updated.**

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 C, 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.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

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*

QUINIZARIN, TECHNICAL — SPECIFICATION

(*First Revision*)

**1 SCOPE**

This standard prescribes the requirements, the methods of sampling and test for quinizarin, technical.

**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 agreements based on this standard are encouraged to investigate the possibilities of applying the most recent editions of the standards indicated below.

|  |  |
| --- | --- |
| *IS No.* | *Title* |
| 1070 : 2023 | Reagent grade water — Specification *(fourth revision)* |
| 5299 : 2001 | Methods of sampling and tests for dye intermediate (*first revision*) |

**3 REQUIREMENTS**

**3.1 Description**

The material shall be in the form of red or orange-red crystalline powder, and shall be free from 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 and 5 of Table 1.

**Table 1 Requirements for Quinizarin, Technical**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl** **No.** | **Characteristic** | **Requirement** | **Method of Test, Ref to** |
| **Annex** | **Clause of IS 5299** |
| (1) | (2) | (3) | (4) | (5) |
| i) | Purity by HPLC, percent by area, *Min*  | 93.0 | A | — |
| ii) | Assay, percent by mass, *Min*  | 89 | B | — |
| iii) | Moisture content, percent by mass, *Max* | 1.0 | — | 10.3  |
| iv) | Ash, percent by mass, *Max* | 0.5 | — | 12.2  |
| v) | Melting point, oC  | Shall melt within the range of 3 °C including191 °C | — |  9 |
| vi) | Matter insoluble in toluene, percent by mass, *Max* |  1.0 | — | 11.3  |
|  |

**4 PACKING AND MARKING**

**4.1 Packing**

The material shall be suitably packed in polythene bags or as agreed to between the purchaser and the supplier.

**4.2 Marking**

**4.2.1** Each bag shall be securely closed and shall bear legibly and indelibly the following information:

1. Name of the material;
2. Name of the manufacturer and his recognized trade-mark, if any;
3. Batch number;
4. Gross, net and tare mass;
5. Month and year of manufacture;
6. Shelf life of the material; and
7. 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, using an appropriate sampling implement.

**5.2 Number of Tests**

**5.2.1** Tests for the determination of all characteristics, namely purity (by HPLC), assay, moisture content, ash, melting point and matter insoluble 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 requirement given in Table 1.

**6 TEST METHODS**

**6.1** Tests shall be carried out as prescribed in col 4 and col 5 of Table 1.

**6.2 Quality of Reagents**

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

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

**ANNEX A**

[*Table* 1*, Sl No.* (i)]

**DETERMINATION OF QUINIZARIN CONTENT (PURITY) BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY**

**A-1 GENERAL**

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 APPARATUS**

**A-2.1 High-performance Liquid Chromatography** **—** quaternary gradient liquid chromatography system with UV-visible detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

**A-2.1.1** *Column* **—** C18 column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 μm or equivalent.

**A-2.2 Volumetric Flask — c**lassA grade.

**A-3 REAGENTS**

**A-3.1 Quinizarin** — known purity.

**A-3.2 Acetonitrile** — HPLC grade.

**A-3.3 Tetrahydrofuran (THF)** — HPLC grade.

**A-3.4 Water** — HPLC grade.

**B-4 SAMPLE PREPARATION**

Weigh accurately 20 mg sample into 50 ml volumetric flask. Add 2 ml tetrahydrofuran (THF) to it and dissolve. Make the volume up to the mark with acetonitrile.

**A-5 BUFFER PREPARATION**

Take 1ml of orthophosphoric acid in l liter volumetric flask and make it up to the mark with HPLC grade water.

**A-6 FLOW RATE —** 1.0 ml/min.

**A-7 MOBILE PHASE —** Acetonitrile: Buffer80: 20 (*v/v*)

**A-8 COLUMN OVEN TEMPERATURE —** Ambient Temperature.

**A-9 INJECTION VOLUME —** 20 μl

**A-10 RUN TIME —** 20 min

**A-11 WAVELENGTH —** 254 nm

**A-12 PEAK TIME —** Quinizarin: 7 min



FIG. 1 TYPICAL CHROMATOGRAPH

**TABLE 2 TYPICAL PEAK TABLE**

|  |  |  |  |
| --- | --- | --- | --- |
| **Components** | **Retention time** | **Area** | **Area percent** |
|  | 2.63 | 61249 | 0.15 |
|  | 4.17 | 835846 | 2.08 |
|  | 4.73 | 15833 | 0.04 |
| Quinizarin | 7.01 | 38468773 | 95.52 |
|  | 8.95 | 889347 | 2.21 |
|  |  | 40271048 | 100.00 |

**A-13 CALCULATION**

Calculate the peak area of individual constituent pertaining to quinizarin 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 quinizarin.

Purity of Quinizarin, percent by area $=\frac{Quinizarin Peak area in the sample }{Sum Areas of all peaks in the chromatogram}$ × 100

**ANNEX B**

[*Table* 1*, Sl No.* (ii)]

**DETERMINATION OF QUINIZARIN CONTENT (ASSAY)**

**B-1 REAGENTS**

**B-1.1 Polycaprolactum —** Polycaprolactum for column chromatography.

NOTE — Any other equally effective column material may also be used.

**B-1.2 Toluene** *—* Dried for 24 h over fused calcium chloride, filtered and distilled. The portion distilling between 109 oC and 111°C is collected for use.

**B-1.3 Quinizarin (Chromatographically pure)** *—* Five grams of commercial sample of quinizarin is treatedwith 200 ml of hot boiling water, filtered on sintered-glass crucible (G4) washed with hot water and dried. This treatment removes water-soluble impurities present in the sample. Two grams of the dried sample is dissolved in about 300 ml of toluene by refluxing for about 30 minutes. The solution of quinizarin is poured in the chromatographic column prepared as in **B-2.1** and allowed to get chromatographed. When all the solution has passed into the adsorbent column, first wash the sides of the tube with small portions of toluene. When the cotton plug at the top is free of colour of quinizarin, fill the whole tube with toluene. The chromatogram will develop into two distinct bands (a) lower yellow one is quinizarin, and (b) upper pink one is an impurity. Collect the quinizarin band in a suitable conical flask. Filter the collected solution through a filter paper to remove any extraneous matter. Concentrate the solution, cool and allow to crystallize. Filter off the crystallized quinizarin and wash with a small portion of toluene. Dry at 100 °C to 110 °C. Finally, keep under vacuum at 100 °C to 110 °C for 1h to 2 hours to drive off any solvent. In order to check the purity of chromatographed sample, dissolve about 10 mg, accurately weighed, in 10 ml of toluene and pass the cooled solution through a chromatographic column set up as described in **B-2.2** eluting with toluene as necessary. Collect the band carefully (only one band shall appear) and determine its optical density at 475 nm. Make up a solution of purified crystals using the same proportion of crystals to solvent. Determine its optical density in the same way. A difference in the two figures for optical density amounting to more than 0.003 indicates the presence of impurity. The chromatographic purification shall then be repeated until this check test is satisfied.

**B-2 APPARATUS**

**B-2.1 Polycaprolactum Column** *—* A glass tube of one meter length and having internal diameter 2.5 cm joined with a stop-cock at the lower end, is setup vertically so that the percolation passing through the tube can be collected conveniently in a conical flask. Place a cotton-wool plug in the tube and press it to the bottom of the tube by means of a glass rod flattened at the end. Place a disc of filter paper, cut to the approximate internal diameter of the chromatographic tube, on the top of the cotton-wool.

**B-2.1.1** Prepare a slurry of about 25 g of the polycaprolactum in toluene and pour it into the tube. Wash down the sides of the tube and allow the adsorbent to set for one hour. Place, first, a disc of filter paper and then a cotton-wool plug at the top of the column. Care should be taken to see that the polycaprolactum column always remains soaked with the solvent so that at least a 2 cm layer of the solvent remains on top of the polycaprolactum. On no account shall the column be left to dry. In the event of this happening, the adsorbent in the tube shall be re-slurried and re-packed.

**B-2.2** Set up another column taking a tube 40 cm long and 1.3 cm in diameter and packing 3 g of polycaprolactum in the same manner.

**B-3** **STANDARD CALIBRATION GRAPH**

Prepare a number of standard solutions of chromatographically pure quinizarin, as obtained in **B-1.3**, in toluene, varying in concentrations from 0.6 mg/100 ml to 2.0 mg/100 ml, with a difference of 0.2 mg/100 ml between successive concentration. Take readings for optical density or percentage transmittance for the above mentioned concentrations at a wavelength of 475 nm (*see* Notes) using the specially matched cells specified for the particular spectrophotometer used. The temperature of the solutions immediately before and after measurement shall be (27 ± 2) °C. Where the instrument is calibrated in percentage transmittance only, the optical density can be read out from standard tables. Plot the calibration curve of concentration (in mg/100 ml) against optical density. This is the standard calibration graph.

NOTES

**1** The readings for optical density or percentage transmittance are taken at the predetermined wavelength at which there is maximum absorption.

**2** The standard calibration curve and the spectrophotometer shall be checked for accuracy from time to time.

**B-4** **PROCEDURE**

**B-4.1** Weigh accurately 100 mg to 120 mg of the sample in a 100 ml clean, dry conical flask. Add about 75 ml of toluene and reflux for 30 min. Cool and transfer the solution to a 100 ml, volumetric flask. Wash the conical flask with toluene and then fully transfer the material to the volumetric flask. Dilute the solution to the 100 ml mark (I). Pipette out 10 ml from this solution (I) and transfer to another 100 ml volumetric flask and further dilute to the 100 ml mark with toluene (II). With the help of l0 ml burette, transfer 10 ml of this latter diluted solution (II) accurately to the chromatographic column (prepared as in **B-2.1**). The solution starts passing down the column. When it is just fully adsorbed at the top of the column, wash the sides carefully with small quantities of toluene.

Finally fill the upper part of the column with toluene and allow the chromatogram to develop, keeping a good ahead of solvent above the column throughout. Start collecting the main yellow band of quinizarin in a l00 ml dry volumetric flask. When the entire band of quinizarin is thus eluted, dilute to 100 ml exactly with toluene (III). Use this solution to take readings from the optical instrument.

**B-4.2** Adjust the wavelength of maximum absorption (λ max) at 475 nm and then adjust the instrument to 100 percent transmittance with toluene blank. Now replace the blank with the solution (III) of the sample and read the percentage transmittance. Refer to the standard tables for conversion of percent transmittance to optical density. Read out the concentration (C) against the optical density from the standard calibration graph (**B-3**) in terms of milligrams per 100 ml of the final diluted solution (III).

**B-5** **CALCULATION**

The amount of quinizarin in the sample is C **×** 100 mg.

Thus,

 Assay, percent by weight = $\frac{10 000C}{W}$

where

*C* = quantity of quinizarin in milligrams contained in 100 ml of the final dilute solution (III) as read from the calibration graph; and

*W* = weight, in milligrams of the sample taken for the test.

**ANNEX C**

 (*Foreword*)

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

|  |  |
| --- | --- |
| **Pictogram(s) :**  | Environment  |
| **Signal Word :**  | Warning  |
| **Hazard Statement :** | Very toxic to aquatic life with long lasting-effects.  |
| **Precautionary Statement :** | Avoid release to the environment. Collect spillage. Dispose of contents / container in accordance with local / regional / national / international regulations. |

**ANNEX D**

(*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 |