भारतीय मानक Indian Standard IS 11751 : 2024 ISO 418 : 2001

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(पहला पुनरीक्षण)

Sodium Sulphite, Anhydrous, Photographic Grade — Specification

(First Revision)

ICS 37.040.30

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Price Group 6

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical to ISO 418 : 2001 'Photography - Processing chemicals — Specifications for anhydrous sodium sulfite' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Electroplating Chemicals and Photographic Materials Sectional Committee and approval of the Chemical Division Council.

The specification for sodium sulphite anhydrous, photographic grade was earlier covered under IS 247: 1972 along with other grades of the material. The Committee responsible for formulation of this standard felt that a separate standard for the photographic grade of the material should be formulated. Consequently, this standard covering only the photographic grade of the material was formulated in 1986.

Now this standard is being revised by adopting ISO 418 : 2001 on dual number bas



In this adopted standard, the reference appears to certain International Standards for which Indian Standards do not exist. So, the Committee has reviewed the provisions of the following International Standards/documents referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard:

International Standards

Title

ISO 10349-1 : <mark>2002</mark>	Photography — Photographic-grade chemicals — Test methods — Part 1: General
ISO 10349-3 : 1992	Photography — Photographic grade chemicals — Test methods — Part 3: Determination of matter insoluble in ammonium hydroxide solution
ISO 10349-5 : 1992	Photography — Photographic grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content
ISO 10349-7 : 1992	Photography — Photographic-grade chemicals — Test methods — Part 7: Determination of alkalinity or acidity
ISO 10349-9 : 1992	Photography — Photographic-grade chemicals — Test methods — Part 9: Reaction to ammoniacal silver nitrate

In this adopted standard, reference appears to certain international Standard where the standard atmospheric conditions to be observed are stipulated which are not applicable to tropical/subtropical countries. The applicable standard atmospheric conditions for Indian conditions are (27 ± 2) °C and (65 ± 5) percent relative humidity and shall be observed while using this standard.

The standard also makes a reference to the BIS Certification Marking of the product, details of which are given in National Annex A.

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

SODIUM SULPHITE, ANHYDROUS, PHOTOGRAPHIC GRADE — SPECIFICATION

(First Revision)

1 Scope

This International Standard establishes criteria for the purity of photographic-grade anhydrous sodium sulfite and specifies the tests to be used to determine the purity.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10349-1:1992, Photography — Photographic-grade chemicals — Test methods — Part 1: General.

ISO 10349-3:1992, Photography — Photographic-grade chemicals — Test methods — Part 3: Determination of matter insoluble in ammonium hydroxide solution.

ISO 10349-5:1992, Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.

ISO 10349-7:1992, Photography — Photographic-grade chemicals — Test methods — Part 7: Determination of alkalinity or acidity.

ISO 10349-9:1992, Photography — Photographic-grade chemicals — Test methods — Part 9: Reaction to ammoniacal silver nitrate.

3 General

3.1 Physical properties

Anhydrous sodium sulfite (Na₂SO₃) is a white granular powder. It has a relative molecular mass of 126,04.

3.2 Hazardous properties

Anhydrous sodium sulfite is not hazardous when handled with normal precautions. Avoid contact with acids.

3.3 Storage

Anhydrous sodium sulfite shall be stored in a closed container at room temperature.

4 Requirements

A summary of the requirements is shown in Table 1.

Test	Limit	Subclause	International Standard in which test method is given
Assay	Minimum: 97,0 %	7.1	ISO 418
Insoluble matter (as precipitate of calcium, magnesium and ammonium hydroxides)	Maximum: 0,5 %	7.2	ISO 10349-3
Mass fraction of heavy metals (as Pb)	Maximum: 0,002 %	7.3	ISO 10349-5
Mass fraction of iron (Fe)	Maximum: 0,005 %	7.4	ISO 10349-5
Alkalinity (as Na ₂ CO ₃)	Maximum: 0,15 %	7.5	ISO 10349-7
Reaction to ammoniacal silver nitrate	To pass test	7.6	ISO 10349-9
Mass fraction of thiosulfate (as $Na_2S_2O_3$)	Maximum: 0,03 %	7.7	ISO 418
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.8	ISO 418

Table 1 — Summary of requirements

5 Reagents and glassware

All reagents, materials and glassware shall conform to the requirements specified in ISO 10349-1 unless otherwise noted. The hazard warning symbols, used as a reminder in those steps detailing handling operations, are defined in ISO 10349-1. These symbols are used to provide information to the user and are not meant to provide conformance with hazardous labelling requirements, as these vary from country to country.

6 Sampling

See ISO 10349-1.

7 Test methods

7.1 Assay

7.1.1 Specification

The minimum mass fraction of anhydrous sodium sulfite shall be 97,0 %.

7.1.2 Reagents

- **7.1.2.1** Hydrochloric acid, HCI, $\rho \approx 1,18$ g/ml (DANGER: $\langle B \rangle \langle C \rangle$)¹⁾.
- 7.1.2.2 Potassium iodide, Kl.
- **7.1.2.3** lodine, $c(I_2) = 0.05 \text{ mol/l} (12.7 \text{ g/l})^{2/3)}$.

Weigh, to the nearest 0,001 g, 12,7 g of freshly sublimed iodine (DANGER: $\langle C \rangle \langle O \rangle$) into a tared weighing flask. Add 36 g of potassium iodide (7.1.2.2) and 100 ml of water. After solution is complete, add three drops of hydrochloric acid (7.1.2.1) (DANGER: $\langle B \rangle \langle C \rangle$), and dilute to 1 litre at 20 °C in a volumetric flask. From the mass of iodine, *m*, calculate the concentration, *c*, in moles per litre, from

$$c(\mathbf{I}_2) = \frac{m}{254}$$

- **7.1.2.4** Sodium thiosulfate, $c(Na_2S_2O_3) = 0,100 \text{ mol/l} (15,8 \text{ g/l})^2)$.
- NOTE This solution is not required for the direct-titration method (7.1.4.2).
- **7.1.2.5** Salicylic acid, $c(HOC_6H_4COOH) = 1 \% (10 g/l)$.
- **7.1.2.6** Starch indicator, 5 g/l solution.

Stir 5 g of soluble starch into 100 ml of 1 % salicylic acid solution (7.1.2.5). Add 300 ml to 400 ml of boiling water. Boil until the starch dissolves and dilute to 1 litre with water.

7.1.3 Apparatus

- 7.1.3.1 Burette, of capacity 50 ml.
- 7.1.3.2 Pipette, of capacity 50 ml.
- 7.1.3.3 Magnetic stirrer and bar, for the direct-titration method (7.1.4.2).

7.1.4 Procedure

Use either the back-titration method (7.1.4.1) or the direct-titration method (7.1.4.2).

7.1.4.1 Back-titration method

Using a pipette (7.1.3.2), transfer 50,00 ml of the iodine solution (7.1.2.3) to a glass-stoppered flask. Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g and wash this into the flask. Add 5 ml of the hydrochloric acid (7.1.2.1) (DANGER: $\langle B \rangle \langle C \rangle$) and using a burette (7.1.3.1) titrate with the sodium thiosulfate solution (7.1.2.4), adding 2 ml of the starch indicator (7.1.2.6) just before the endpoint.

¹⁾ Hazard warning codes are defined in ISO 10349-1.

²⁾ Commercially available analysed reagent solution is recommended. If the solution is to be prepared, see any quantitative analytical chemistry test.

³⁾ It is recommended that self-prepared iodine solutions be standardized before use.

7.1.4.2 Direct-titration method

Weigh, to the nearest 0,000 1 g, a test portion of about 0,16 g. Using a pipette (7.1.3.2), transfer 50,00 ml of the iodine solution (7.1.2.3) to a completely dry 250 ml beaker that contains a magnetic stirring bar (7.1.3.3). While stirring the iodine solution in the beaker, add the test portion to the centre of the beaker using a camel-hair brush. Avoid contact of the sample with the sides of the beaker.

If the iodine is not decolourized after addition of the sample, discard the trial and restart the procedure. If necessary, increase the test portion by 0,01 g.

Wash down the side walls of the beaker using about 2 ml of the starch indicator (7.1.2.6). Using a burette (7.1.3.1), immediately titrate with the iodine solution to the first permanent light-purple colour. Wash any iodine solution remaining on the burette tip into the solution with deionized water.

If the titration exceeds 10 ml, repeat the test as this can result in test results lower than the actual assay. Adjust the sample appropriately.

7.1.5 Expression of results

7.1.5.1 Back-titration method

The assay, expressed as a percentage by mass of sodium sulfite (Na₂SO₃), is given by

$$\frac{6,302\left[\left(100c_{1}\right)-\left(c_{2}\cdot V\right)\right]}{m}$$

where

- c_1 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.3);
- c_2 is the actual concentration, expressed in moles per litre, of the sodium thiosulfate solution (7.1.2.4);
- *V* is the volume, expressed in millilitres, of the sodium thiosulfate solution used for the titration in 7.1.4.1;
- *m* is the mass, expressed in grams, of the test portion;
- 6,302 is the conversion factor obtained from the equivalent mass of sodium sulfite (i.e. 126,04/2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);
- 100 is the conversion factor obtained from the volume, expressed in millilitres, of the iodine solution added in 7.1.4.1 (i.e. 50) × the number of equivalents of the iodine solution (i.e. 2).

7.1.5.2 Direct-titration method

The assay, expressed as a percentage by mass of sodium sulfite (Na₂SO₃), is given by

$$\frac{12,604 \cdot c_1(50+V)}{m}$$

where

- c_1 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.3);
- *V* is the volume, expressed in millilitres, of the iodine solution used for the titration in 7.1.4.2;
- *m* is the mass, expressed in grams, of the test portion;

- 12,604 is the conversion factor obtained from the mass of sodium sulfite equivalent to 1 mole of iodine (i.e. 126,04) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);
- 50 is the volume, in millilitres, of the iodine solution added in 7.1.4.2.

7.2 Mass fraction of insoluble matter (as a precipitate of calcium, magnesium and ammonium hydroxides)

7.2.1 Specification

The maximum mass fraction of insoluble matter shall be 0,5 %.

7.2.2 Procedure

Determine the percentage of insoluble matter in accordance with ISO 10349-3.

7.3 Mass fraction of heavy metals

7.3.1 Specification

The maximum mass fraction of heavy metals shall be 0,002 %.

7.3.2 Procedure

NOTE The standard for the iron test (7.4) is prepared in the same way as the heavy metals standard.

Determine the percentage of heavy metals in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g prepared in accordance with ISO 10349-5:1992, 7.3. Use 4 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.4 Mass fraction of iron

7.4.1 Specification

The maximum mass fraction of iron shall be 0,005 %.

7.4.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g prepared in accordance with ISO 10349-5:1992, 7.3. Use 10 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.5 Alkalinity (as Na₂CO₃)

7.5.1 Specification

The maximum mass fraction of free alkali shall be 0,15 %.

7.5.2 Reagents

7.5.2.1 Hydrogen peroxide, neutral solution, approximately 33 g/l.

Dilute 30 % hydrogen peroxide (DANGER: $\langle\langle C \rangle\rangle$) (1 + 9) and neutralize to the methyl red indicator (7.5.2.2).

7.5.2.2 Methyl red indicator, methanol solution, 0,1 g/l.

7.5.3 Procedure

Weigh, to the nearest 0,05 g, a test portion of about 5 g and dissolve it in 50 ml of freshly boiled and cooled water. Add 100 ml of the neutral hydrogen peroxide solution (7.5.2.1) and 2 drops of methyl red indicator (7.5.2.2). Proceed as specified in ISO 10349-7:1992, 7.1 and determine the percentage alkalinity as sodium carbonate using a factor K equal to 5,3 in the calculation.

7.6 Reaction to ammoniacal silver nitrate

7.6.1 Specification

To pass test.

7.6.2 Procedure

Determine the reaction to ammoniacal silver nitrate in accordance with ISO 10349-9.

7.7 Mass fraction of thiosulfate (as Na₂S₂O₃)

7.7.1 Specification

The maximum mass fraction of sodium thiosulfate shall be 0,03 %.

7.7.2 Reagents

- 7.7.2.1 Potassium bromide, KBr.
- 7.7.2.2 Mercury(II) chloride, HgCl₂ (DANGER: $\langle\langle S \rangle\rangle$).

7.7.2.3 Mercury(II) chloride reagent.

Dissolve 25 g of potassium bromide (7.7.2.1) and 25 g of mercury(II) chloride (7.7.2.2) (DANGER: $\langle\langle S \rangle\rangle$) in 900 ml of water at 50 °C. Cool, dilute to 1 litre and allow to stand overnight. Filter the solution if it is not perfectly clear.

7.7.2.4 Thiosulfate standard solution, $c(S_2O_3^{2-}) = 0,056 \text{ mg/ml}.$

Dilute 5 ml of the thiosulfate solution (7.1.2.4) to 1 litre.

7.7.3 Apparatus

- 7.7.3.1 Graduated pipettes, of capacity 1 ml.
- 7.7.3.2 Two matched Nessler colour-comparison cylinders, of capacity 50 ml.

7.7.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 13 g. Dissolve it in water, dilute to 100 ml and mix well. Slowly transfer, using a pipette (7.7.3.1), 0,5 ml of this solution to 10 ml of the mercury(II) chloride reagent (7.7.2.3) contained in one of the Nessler colour-comparison cylinders (7.7.3.2). To 10 ml of the mercury(II) chloride reagent contained in the second Nessler cylinder, slowly add 0,25 ml of the thiosulfate standard solution (7.7.2.4) using a second pipette. Swirl to mix and allow both to stand for 10 min. At the end of this time, swirl again to distribute the opalescence. Without proper mixing, a repeatable turbidity may not be obtained.

Immediately examine, in the Nessler colour-comparison cylinders, the opalescence produced in the test and control solutions. The opalescence in the test solution shall not exceed that of the control solution.

If the solutions are allowed to stand for more than 15 min, secondary reactions occur which will affect the results.

7.8 Appearance of solution

7.8.1 Specification

The solution shall be clear and free from insoluble matter except for a slight flocculence.

7.8.2 Procedure

Dissolve a test portion of 20,0 g in 100 ml of water. Observe the solution for colour and clarity.

National Annex A

(National Foreword)

A-1 BIS CERTIFICATION MARKING

The product may also be marked with the Standard Mark.

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made thereunder. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

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