भारतीय मानक Indian Standard

IS 500 : 2024 ISO 3629 : 2000

पोटेशियम मेटाबाइसल्फाइट, फोटोग्राफिक ग्रेड — विशिष्टि

(पाँचवां पुनरीक्षण)

Potassium Metabisulphite, Photographic Grade — Specification

(Fifth Revision)

ICS 37.040.30

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NATIONAL FOREWORD

This Indian Standard (Fifth Revision) which is identical to ISO 3629: 2000 'Photography — Processing chemicals — Specifications for potassium metabisulfite' 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.

This standard was first published in 1953 and subsequently revised in 1963, 1972, 1980 and 1999. Now this standard is being revised by adopting ISO 3629: 2000 on dual number basis.

The text of the ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and
- b) Comma (,) has been used as a decimal marker, while in Indian Standards the current practice is to use a point (.) as the decimal marker.

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 : 1992 ¹	Photography — Photographic-grade chemicals — Test methods — Part 1: General
ISO 10349-5 : 1992	Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content
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 Standards 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 $(27 \pm 2)^{\circ}$ 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 <u>National Annex A</u>.

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 ISO 10349-1: 1992 has been revised as ISO 10349-1: 2002.

Indian Standard

POTASSIUM METABISULPHITE, PHOTOGRAPHIC GRADE — SPECIFICATION

(Fifth Revision)

1 Scope

This International Standard establishes criteria for the purity of photographic-grade potassium metabisulfite 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 10349-5:1992, Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.

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

3 General

3.1 Physical properties

Potassium metabisulfite $(K_2S_2O_5)$ exists in the form of white, glassy crystals. It has a relative molecular mass of 222.33.

3.2 Hazardous properties

Potassium metabisulfite is not hazardous when handled with normal precautions, but it is toxic if ingested. Avoid contact with acids.

3.3 Storage

Potassium metabisulfite shall be stored in a closed container at room temperature.

4 Requirements

A summary of the requirements is shown in Table 1.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	Minimum: 95,0 %	7.1	ISO 3629
Mass fraction of heavy metals (as Pb)	Maximum: 0,005 %	7.2	ISO 10349-5
Mass fraction of iron (Fe)	Maximum: 0,005 %	7.3	ISO 10349-5
Reaction to ammoniacal silver nitrate	To pass test	7.4	ISO 10349-9
pH value	3,7 to 4,6	7.5	ISO 3629
Mass fraction of thiosulfate (as S ₂ O ₃ ²⁻)	Maximum: 0,04 %	7.6	ISO 3629
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.7	ISO 3629

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 potassium metabisulfite shall be 95,0 %.

7.1.2 Reagents

7.1.2.1 Acetic acid, glacial, CH_3COOH (DANGER: $\langle B \rangle \langle C \rangle$)¹⁾.

7.1.2.2 Acetic acid, $c(CH_3COOH) \approx 2 \text{ mol/l.}$

Dilute 120 g of glacial acetic acid (7.1.2.1) (DANGER: $\langle B \rangle \langle C \rangle$) to 1 litre.

7.1.2.3 Hydrochloric acid (HCl), $\rho \approx 1,18$ g/ml (DANGER: $\langle B \rangle \langle C \rangle$).

7.1.2.4 Potassium iodide (KI).

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

7.1.2.5 lodine, $c(l_2) = 0.05 \text{ mol/l } (12.7 \text{ g/l})^{2) 3}$.

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 (7.1.2.4) and 100 ml of water. After solution is complete, add three drops of hydrochloric acid (7.1.2.3) (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

$$c = \frac{m}{254}$$

- **7.1.2.6** Salicylic acid, $c(HOC_6H_4COOH) = 1 \% (10 g/l)$.
- **7.1.2.7 Starch indicator**, 5 g/l solution.

Stir 5 g of soluble starch into 100 ml of 1 % salicylic acid solution (7.1.2.6). Add 300 ml of this solution to 400 ml of boiling water. Boil until the starch dissolves and dilute to 1 litre with water.

- **7.1.2.8** Sodium thiosulfate, $c(Na_2S_2O_3) = 0,100 \text{ mol/l } (15,8 \text{ g/l})^2)$.
- **7.1.2.9** Sulfuric acid, $c(H_2SO_4) = 0.05 \text{ mol/l } (4.9 \text{ g/l})^{2) \text{ 4}}$.
- **7.1.2.10** Ethanol (C₂H₅OH), 95 % (denatured).
- **7.1.2.11** Phenolphthalein indicator, 5 g/l solution.

Dissolve 1 g of phenolphthalein in 100 ml of ethanol (7.1.2.10) and add 100 ml of water with constant stirring. Filter if necessary.

7.1.2.12 Neutral formaldehyde, $c(HCHO) \approx 37\%$ (360 g/l) (DANGER: $\langle B \rangle \langle C \rangle \langle S \rangle$).

Adjust the pH of the formaldehyde solution so that it is neutral to phenolphthalein indicator (7.1.2.11).

- 7.1.3 Apparatus
- **7.1.3.1** Burette, of capacity 50 ml.
- **7.1.3.2 Pipette**, of capacity 50 ml.

7.1.4 Procedure

Using a pipette (7.1.3.2), transfer 50,00 ml of the iodine solution (7.1.2.5) to a flask. Weigh, to the nearest 0,000 1 g, a test portion of about 0,23 g and wash this into the flask. Add 5 ml of the acetic acid (7.1.2.2) and mix to ensure complete dissolution of the sample. Using a burette (7.1.3.1), titrate with the sodium thiosulfate solution (7.1.2.8), adding 2 ml of the starch indicator (7.1.2.7) just before the endpoint.

Weigh, to the nearest 0,001 g, another test portion of about 5 g. Dissolve it in 50 ml of water and add 50 ml of the neutral formaldehyde (7.1.2.12). Add a few drops of the phenolphthalein indicator (7.1.2.11) and, using a burette (7.1.3.1), titrate with the sulfuric acid (7.1.2.9) to the colour change.

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

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

⁴⁾ This can be prepared from concentrated sulfuric acid, $\rho \approx 1,84$ g/ml (DANGER: $\langle\langle C \rangle\rangle$).

7.1.5 Expression of results

The assay, expressed as a percentage by mass of potassium metabisulfite $(K_2S_2O_5)$, is given by

$$\frac{5,558(100 \cdot c_1 - c_2 \cdot V_2)}{m_1} - \frac{11,12 \cdot c_3 \cdot V_3}{m_2}$$

where

- c_1 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.5);
- c_2 is the actual concentration, expressed in moles per litre, of the sodium thiosulfate solution (7.1.2.8);
- c_3 is the actual concentration, expressed in moles per litre, of the sulfuric acid (7.1.2.9);
- *V*₂ is the volume, expressed in millilitres, of the sodium thiosulfate solution used for the first titration in 7.1.4;
- V_3 is the volume, expressed in millilitres, of the sulfuric acid used for the second titration in 7.1.4;
- m_1 is the mass, expressed in grams, of the test portion used for the first titration;
- m_2 is the mass, expressed in grams, of the test portion used for the second titration;
- 5,558 is the conversion factor obtained from the equivalent mass of potassium metabisulfite (i.e. 222,33/4) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);
- is the conversion factor obtained from the volume, expressed in millilitres, of the iodine solution added in 7.1.4 (i.e. 50) × the number of equivalents of the iodine solution (i.e. 2);
- 11,12 is the conversion factor obtained from the equivalent mass of potassium metabisulfite (i.e. 222,33/4) × the number of equivalents of potassium metabisulfite for potassium sulfite (i.e. 2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

NOTE When an assay based on the mass fraction of sulfite but expressed as potassium metabisulfite ($K_2S_2O_5$) is desired, the second titration in 7.1.4 is not required and the assay is given by

$$\frac{5,558 \left(100 \cdot c_1 - c_2 \cdot V_2\right)}{m_1}$$

7.2 Mass fraction of heavy metals

7.2.1 Specification

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

7.2.2 Procedure

NOTE The standard for the iron test (7.3) 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 10 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.3 Mass fraction of iron

7.3.1 Specification

The maximum mass fraction of iron shall be 0.005 %.

7.3.2 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.

7.4 Reaction to ammoniacal silver nitrate

7.4.1 Specification

To pass test.

7.4.2 Procedure

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

7.5 pH value

7.5.1 Specification

The pH shall be between 3,7 and 4,6.

7.5.2 Apparatus

7.5.2.1 Electronic pH-meter, equipped with a glass electrode and standard reference electrode.

7.5.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g. Dissolve it in about 80 ml of boiled and cooled water and dilute to 100 ml. Measure the pH of the solution at 20 °C, using the pH-meter (7.5.2.1) in accordance with the manufacturer's instructions.

7.6 Mass fraction of thiosulfate (as $S_2O_3^{2-}$)

7.6.1 Specification

The maximum mass fraction of thiosulfate (as $\mathrm{S}_2\mathrm{O}_3^{2-}$) shall be 0,04 %.

7.6.2 Reagents

- **7.6.2.1 Potassium bromide** (KBr).
- **7.6.2.2** Mercury(II) chloride, $HgCl_2$ (Danger: $\langle\langle S \rangle\rangle$).

7.6.2.3 Mercury(II) chloride reagent.

Dissolve 25 g of potassium bromide (7.6.2.1) and 25 g of mercury(II) chloride (7.6.2.2) (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.

7.6.2.4 Thiosulfate standard solution, $\rho(S_2O_3^{2-}) = 0.056$ mg/ml.

Dilute 5 ml of sodium thiosulfate solution (7.1.2.8) to 1 litre.

7.6.3 Apparatus

- **7.6.3.1** Graduated pipettes, of capacity 1 ml.
- **7.6.3.2** Two matched Nessler colour-comparison cylinders, of capacity 50 ml.

7.6.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 6 g. Dissolve it in water, dilute to 100 ml and mix well. Using a pipette (7.6.3.1), slowly transfer 0,5 ml of this solution into 10 ml of the mercury(II) chloride reagent (7.6.2.3) contained in one of the Nessler colour-comparison cylinders (7.6.3.2). To 10 ml of the mercury(II) chloride reagent contained in the second Nessler colour-comparison cylinder, slowly add 0,25 ml of the thiosulfate standard solution (7.6.2.4) using a second pipette. 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.

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

7.7 Appearance of solution

7.7.1 Specification

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

7.7.2 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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Review of Indian Standards

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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