INTERNATIONAL STANDARD

ISO 3627

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Photography — Processing chemicals — Specifications for anhydrous sodium metabisulfite

Photographie — Produits chimiques de traitement — Spécifications pour le métadisulfite de sodium anhydre

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3627 was prepared by Technical Committee ISO/TC 42, *Photography*.

This third edition cancels and replaces the second edition (ISO 3627:1994), of which it constitutes a technical revision.

Introduction

This International Standard is one of a series that establishes criteria of purity for chemicals used in processing photographic materials. General test methods and procedures cited in this International Standard are compiled in ISO 10349-1.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques, which may not always be the case. Some of the procedures utilize caustic, toxic or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks or aprons. Normal precautions for the safe performance of any chemical procedure shall be exercised at all times, but specific details have been provided for hazardous materials. Hazard warnings designated by a letter enclosed in angle brackets "〈 〉" are used as a reminder in those steps detailing handling operations and are defined in ISO 10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

This International Standard provides chemical and physical requirements for the suitability of a photographic-grade chemical. The tests correlate with undesirable photographic effects. Purity requirements are set as low as possible, consistent with these photographic effects. These criteria are considered to be the minimum requirements necessary to ensure sufficient purity for use in photographic processing solutions, except that if the purity of a commonly available grade of chemical exceeds photographic processing requirements and if there is no economic penalty in its use, the purity requirements have been set to take advantage of the availability of the higher-quality material.

Every effort has been made to keep the number of requirements to a minimum. Inert impurities are limited to amounts that will not unduly reduce the assay. All tests are performed on samples "as received" to reflect the condition of materials furnished for use. Although the ultimate criterion for suitability of such a chemical is its successful performance in an appropriate use test, the shorter, more economical test methods described in this International Standard are generally adequate.

Assay procedures have been included in all cases where a satisfactory method is available. An effective assay requirement serves not only as a safeguard of chemical purity, but also as a valuable complement to the identity test. Identity tests have been included whenever a possibility exists that another chemical or mixture of chemicals could pass the other tests.

All requirements listed in clause 4 are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

Efforts have been made to employ tests that are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests that require highly specialized equipment or techniques. Instrumental methods have been specified only as alternative methods or alone in those cases where no other satisfactory method is available.

Over the past few years, great improvements have been made in instrumentation for various analyses. Where such techniques have equivalent or greater precision, they may be used in place of the tests described in this International Standard. Correlation of such alternative procedures with the given method is the responsibility of the user. In case of disagreement in results, the method called for in the specification shall prevail. Where a requirement states "to pass test", however, alternative methods shall not be used.

Photography — Processing chemicals — Specifications for anhydrous sodium metabisulfite

1 Scope

This International Standard establishes criteria for the purity of photographic-grade anhydrous sodium 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

Anhydrous sodium metabisulfite ($Na_2S_2O_5$) exists as a white or pale-cream granular powder. It has a relative molecular mass of 190,10.

3.2 Hazardous properties

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

3.3 Storage

Anhydrous sodium 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 3627
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 3627
Mass fraction of thiosulfate (as $S_2O_3^{2-}$)	Maximum: 0,03 %	7.6	ISO 3627
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.7	ISO 3627

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 metabisulfite shall be 95,0 %.

7.1.2 Reagents

7.1.2.1 Acetic acid, glacial, CH_3COOH (DANGER: ⟨B⟩⟨C⟩)[1)].

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

Dilute 120 g of glacial acetic acid (7.1.2.1) (DANGER: ⟨B⟩⟨C⟩) to 1 litre.

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

7.1.2.3 Hydrochloric acid, HCl, $\rho \approx 1{,}18$ g/ml (DANGER: ⟨B⟩⟨C⟩).

7.1.2.4 Potassium iodide, KI.

7.1.2.5 Iodine, $c(I_2) = 0{,}05$ mol/l (12,7 g/l)[2)3)].

Weigh, to the nearest 0,001 g, 12,7 g of freshly sublimed iodine (DANGER: ⟨C⟩⟨O⟩) 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: ⟨B⟩⟨C⟩) 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

$$c(I_2) = \frac{m}{254}$$

7.1.2.6 Salicylic acid, HOC_6H_4COOH, 1 % (10 g/l) solution.

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 to 400 ml of boiling water. Boil until the starch dissolves and dilute to 1 litre with water.

7.1.2.8 Sodium thiosulfate, $c(Na_2S_2O_3) = 0{,}100$ mol/l (15,8 g/l)[2)].

NOTE This solution is not required for the direct-titration method (7.1.4.2).

7.1.2.9 Sulfuric acid, $c(H_2SO_4) = 0{,}05$ mol/l (4,9 g/l)[2)4)].

7.1.2.10 Ethanol, C_2H_5OH, 95 % (denatured).

7.1.2.11 Phenolphthalein indicator, 5 g/l.

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, HCHO, approximately 37 % (360 g/l) (DANGER: ⟨B⟩⟨C⟩⟨S⟩).

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.

7.1.3.3 Magnetic stirrer and bar, for direct-titration method (7.1.4.2).

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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.

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

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 (7.1.2.5) to a glass-stoppered 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.

7.1.4.2 Direct-titration method

Weigh, to the nearest 0,000 1 g, a test portion of about 0,12 g. Using a pipette (7.1.3.2), transfer 50,00 ml of the iodine solution (7.1.2.5) 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 (7.1.2.7). 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 volume exceeds 4 ml, repeat the test as this can result in test results lower than the actual assay. Adjust the sample appropriately.

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 titrate with the sulfuric acid (7.1.2.9) to the colour change.

7.1.5 Expression of results

7.1.5.1 Back-titration method

The assay, expressed as a percentage by mass of sodium metabisulfite ($Na_2S_2O_5$), is given by

$$\left[\frac{4{,}753\left[\left(100c_1\right)-\left(c_2\cdot V_2\right)\right]}{m_1}\right]-\left[\frac{9{,}505\left(c_3\cdot V_3\right)}{m_2}\right]$$

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 7.1.4.1;

V_3 is the volume, expressed in millilitres, of the sulfuric acid used for the second titration in 7.1.4.1;

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;

4,753 is the conversion factor obtained from the equivalent mass of sodium metabisulfite (i.e. 190,10/4) × 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);

9,505 is the conversion factor obtained from the equivalent mass of sodium metabisulfite (i.e. 190,10/4) × the number of equivalents of sodium metabisulfite for sodium 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 sodium metabisulfite ($Na_2S_2O_5$) is desired, the second titration in 7.1.4.1 is not required and the assay is given by

$$\frac{4,753\left[\left(100c_1\right)-\left(c_2\cdot V_2\right)\right]}{m_1}$$

7.1.5.2 Direct-titration method

The assay, expressed as a percentage by mass of sulfur dioxide (SO_2), is given by

$$\left[\frac{6,406c_1\left(50+V_1\right)}{m_1}\right]-\left[\frac{6,406\left(c_3\cdot V_3\right)}{m_3}\right]$$

The assay, expressed as a percentage by mass of sodium metabisulfite ($Na_2S_2O_5$), is given by

1,483 8 × percentage by mass of sulfur dioxide

where

c_1 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.5);

c_3 is the actual concentration, expressed in moles per litre, of the sulfuric acid (7.1.2.9);

V_1 is the volume, expressed in millilitres, of the iodine solution used for the first titration in 7.1.4.2;

V_3 is the volume, expressed in millilitres, of the sulfuric acid used for the second titration in 7.1.4.2;

m_1 is the mass, expressed in grams, of the test portion used for the first titration;

m_3 is the mass, expressed in grams, of the test portion used for the second titration;

6,406 is the conversion factor obtained from the mass of sulfur dioxide equivalent to 1 mole of iodine or 1 mole of sulfuric acid (i.e. 64,06) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

50 is the volume, expressed in millilitres, of the iodine solution added in 7.1.4.2;

1,483 8 is the ratio of the conversion factor obtained from the mass of sodium metabisulfite equivalent to 1 mole of iodine (i.e. 190,10/2 = 95,05) to the conversion factor obtained from the mass of sulfur dioxide equivalent to 1 mole of iodine (i.e. 64,06).

NOTE When an assay based on the mass fraction of sulfite but expressed as sodium metabisulfite ($Na_2S_2O_5$) is desired, the second titration in 7.1.4.2 is not required and the sulfur dioxide assay is given by

$$\frac{6,406 c_1 (50 + V_1)}{m_1}$$

The assay for sodium metabisulfite is calculated from the sulfur dioxide assay in the normal way.

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 of the sample 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 a 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 $S_2O_3^{2-}$ shall be 0,03 %.

7.6.2 Reagents

7.6.2.1 Potassium bromide, KBr.

7.6.2.2 Mercury(II) chloride, $HgCl_2$ (DANGER: ⟨⟨S⟩⟩).

7.6.2.3 Mercury(II) chloride reagent.

Dissolve 25 g of potassium bromide (7.6.2.1) and 25 g of mercury(ll) chloride (7.6.2.2) (DANGER: ⟨⟨S⟩⟩) 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 9,5 g. Dissolve it in water, dilute to 100 ml and mix well. Slowly transfer, using a pipette (7.6.3.1), 0,5 ml of this solution to 10 ml of the mercury(ll) 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(ll) 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. 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, 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.

ICS 37.040.30

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