INTERNATIONAL STANDARD

ISO 418

Second edition
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Photography — Processing chemicals — Specifications for anhydrous sodium sulfite

Photographie — Produits chimiques pour traitement — Spécifications relatives au sulfite de soude anhydre

Reference number
ISO 418:1994(E)

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.

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.

International Standard ISO 418 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 418:1976), which has been technically revised.

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Introduction

0.1 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 parts 1, 3, 5, 7 and 9 of ISO 10349.

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, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Normal precautions required in the performance of any chemical procedure are to be exercised at all times but care has been taken to provide warnings 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.

0.2 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 the minimum requirements necessary to assure 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 which 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.

0.3 Efforts have been made to employ tests which are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests which 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 sulfite

1 Scope

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO 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 (Na_2SO_3) 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 Handling and storage

Store in a closed container at room temperature.

4 Requirements

A summary of the requirements is shown in table 1.

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.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	97,0 % (*m/m*) min.	7.1	ISO 418
Insoluble matter (as precipitate of calcium, magnesium and ammonium hydroxides)	0,5 % (*m/m*) max.	7.2	ISO 10349-3
Heavy metals (as Pb)	0,002 % (*m/m*) max.	7.3	ISO 10349-5
Iron (Fe)	0,005 % (*m/m*) max.	7.4	ISO 10349-5
Alkalinity (as Na_2CO_3)	0,15 % (*m/m*) max.	7.5	ISO 10349-7
Reaction to ammoniacal silver nitrate	To pass test	7.6	ISO 10349-9
Thiosulfate (as $Na_2S_2O_3$)	0,03 % (*m/m*) max.	7.7	ISO 418
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.8	ISO 418

NOTE — *m/m* = mass/mass

6 Sampling

See ISO 10349-1.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of Na_2SO_3 shall be 97,0 % (*m/m*) min.

7.1.2 Reagents

7.1.2.1 Hydrochloric acid, ρ = 1,18 g/ml (approximately) (DANGER: < C >< B >)[1].

7.1.2.2 Potassium iodide, KI.

7.1.2.3 Iodine, standard volumetric solution of 0,05 mol/l (12,7 g/l)[2].

Weigh, to the nearest 0,001 g, 12,7 g of freshly sublimed iodine 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) (< C >< B >), and dilute to 1 litre at 20 °C in a one-mark volumetric flask. From the mass of iodine (*m*) calculate the concentration, c_I, in moles per litre.

$$c_I = m/254$$

7.1.2.4 Sodium thiosulfate solution, 0,100 mol/l (15,8 g/l)[2].

NOTE 1 This solution is not required for the direct titration method (7.1.4.2).

7.1.2.5 Salicylic acid, 1 % (10 g/l).

7.1.2.6 Starch, soluble.

7.1.2.7 Starch indicator solution, 5 g/l.

Stir in 5 g of soluble starch (7.1.2.6) with 100 ml of the salicylic acid (7.1.2.5). Add 300 ml to 400 ml of boiling water. Boil until the starch dissolves and finally dilute to 1 000 ml with water.

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

1) Hazard warning codes are defined in ISO 10349-1:1992, clause 4.

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

7.1.3.2 Pipette, of 50 ml capacity.

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

7.1.4 Procedure

Use either 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), deliver 50,00 ml of the iodine solution (7.1.2.3) into a glass stoppered flask. Weigh, to the nearest 0,0001 g, a test portion of about 0,25 g of the test sample and wash this into the flask. Add 5 ml of the hydrochloric acid (7.1.2.1) and titrate with the sodium thiosulfate solution (7.1.2.4), adding the starch indicator solution (7.1.2.7) just before the endpoint.

7.1.4.2 Direct titration method

Weigh, to the nearest 0,0001 g, a test portion of about 0,16 g of the test sample. Using a pipette (7.1.3.2), deliver 50,00 ml of the iodine solution (7.1.2.3) into 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 decolorized 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 solution (7.1.2.7). Immediately titrate with the iodine solution (7.1.2.3) 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 (Na_2SO_3), is given by

$$6{,}302(50c_I - c_S \cdot V)/m$$

where

- c_I is the actual concentration, in moles per litre, of the iodine solution (7.1.2.3);
- c_S is the actual concentration, in moles per litre, of the sodium thiosulfate solution (7.1.2.4);
- V is the volume, in millilitres, of the sodium thiosulfate solution (7.1.2.4) used for the titration;
- m is the mass, in grams, of the test portion;
- 6,302 is a conversion factor for the mass of sodium sulfite equivalent to 1 mole of iodine (i.e. 63,02) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.1.5.2 Direct titration method

The assay, expressed as a percentage by mass of sodium sulfite (Na_2SO_3), is given by

$$6{,}302 \cdot c_I \cdot V'/m'$$

where

- c_I is the exact molarity, in moles per litre, of the iodine solution (7.1.2.3);
- V' is the volume, in millilitres, of the iodine solution (7.1.2.3) used for the titration;
- m' is the mass, in grams, of the test portion;
- 6,302 is a conversion factor for the mass of sodium sulfite equivalent to 1 mole of iodine (i.e. 63,02) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Insoluble matter content (as a precipitate of calcium, magnesium and ammonium hydroxides)

7.2.1 Specification

Maximum content of insoluble matter shall be 0,5 % (*m/m*).

7.2.2 Procedure

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

7.3 Heavy metals content

7.3.1 Specification

Maximum content of heavy metals shall be 0,002 % (*m/m*).

7.3.2 Procedure

NOTE 2 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 Iron content

7.4.1 Specification

Maximum content of iron shall be 0,005 % (*m/m*).

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 Na_2CO_3)

7.5.1 Specification

Maximum free alkali content shall be 0,15 % (*m/m*).

7.5.2 Reagents

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

Dilute 30 % hydrogen peroxide (DANGER: <<C>>) (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 of the test sample and dissolve 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 Thiosulfate (as $Na_2S_2O_3$)

7.7.1 Specification

Maximum sodium thiosulfate content shall be 0,03 % (*m/m*).

7.7.2 Reagents

7.7.2.1 Mercury(II) chloride, $HgCl_2$ (DANGER:<S>).

7.7.2.2 Potassium bromide, KBr.

7.7.2.3 Mercury(II) chloride reagent solution.

Dissolve 25 g of potassium bromide (7.7.2.2) and 25 g of mercury(II) chloride (7.7.2.1) (<S>) in 900 ml of water at 50 °C. Cool, dilute to 1 000 ml and allow to stand overnight. Filter if not perfectly clear.

7.7.2.4 Thiosulfate standard solution.

Dilute 5 ml of the thiosulfate solution (7.1.2.4) to 1 000 ml.

7.7.3 Apparatus

7.7.3.1 Graduated pipette, of 1 ml capacity.

7.7.3.2 Two matched Nessler colour-comparison cylinders, of 50 ml capacity.

7.7.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 13 g of the sample. Dissolve it in water, dilute to 100 ml and mix well. Slowly pipette (7.7.3.1) 0,5 ml of this solution into 10 ml of the mercury chloride reagent solution (7.7.2.3) in one of the Nessler cylinders (7.7.3.2). To 10 ml of the mercury chloride reagent

solution (7.7.2.3) contained in the second Nessler cylinder, slowly add 0,25 ml of the thiosulfate standard solution (7.7.2.4). Allow both to stand for 10 min without agitation, then carefully agitate to distribute the opalescence. Immediately examine, in the Nessler 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 3 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

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.