INTERNATIONAL
STANDARD

ISO
3619

Second edition
1994-12-15

Photography — Processing chemicals — Specifications for ammonium thiosulfate solution

Photographie — Produits chimiques de traitement — Spécifications relatives au thiosulfate d'ammonium en solution

Reference number
ISO 3619:1994(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 3619 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3619:1976), which has been technically revised.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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, 4, 5, 10, 11 and 12 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 ammonium thiosulfate solution

1 Scope

This International Standard establishes criteria for the purity of photographic-grade ammonium thiosulfate solution and specifies 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 10349-4:1992, *Photography — Photographic-grade chemicals — Test methods — Part 4: Determination of residue after ignition.*

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

ISO 10349-10:1992, *Photography — Photographic-grade chemicals — Test methods — Part 10: Determination of sulfide content.*

ISO 10349-11:1992, *Photography — Photographic-grade chemicals — Test methods — Part 11: Determination of specific gravity.*

ISO 10349-12:1992, *Photography — Photographic-grade chemicals — Test methods — Part 12: Determination of density.*

3 General

3.1 Physical properties

Ammonium thiosulfate, $(NH_4)_2S_2O_3$, exists as a clear colourless solution; crystallization may occur at reduced temperatures (cold weather). It has a relative molecular mass of 148,20.

3.2 Hazardous properties

Ammonium thiosulfate solution is not hazardous when handled with normal precautions. Ammonium thiosulfate should not be mixed with large volumes of strong acids or alkalis.

3.3 Storage

Ammonium thiosulfate solution shall be stored 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.

6 Sampling

See ISO 10349-1.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay [as $(NH_4)_2S_2O_3$]	53,0 % (*m*/*m*) min. 61,0 % (*m*/*m*) max.	7.1	ISO 3619
Alkalinity (as NH_4OH)	0,3 % (*m*/*m*) min. 1,5 % (*m*/*m*) max.	7.1	ISO 3619
Sulfites [as $(NH_4)_2SO_3$]	1 % (*m*/*m*) max.	7.1	ISO 3619
Insoluble matter (as precipitate of calcium and magnesium in ammonium hydroxide)	0,2 % (*m*/*m*) max.	7.2	ISO 10349-3
Residue after ignition	0,2 % (*m*/*m*) max.	7.3	ISO 10349-4
Heavy metals (as Pb)	0,001 % (*m*/*m*) max.	7.4	ISO 10349-5
Iron (Fe)	0,001 % (*m*/*m*) max.	7.5	ISO 10349-5
Sulfides (as S^{2-})	0,000 25 % (*m*/*m*) max.	7.6	ISO 10349-10
Specific gravity (at 20 °C) or	1,290 min. 1,346 max.	7.7.1	ISO 10349-11
density (at 20 °C)	1,284 g/ml min. 1,340 g/ml max.	7.7.2	ISO 10349-12

NOTE — *m*/*m* = mass/mass

7 Test methods

7.1 Assay [as $(NH_4)_2S_2O_3$], alkalinity (as NH_4OH) and sulfite content [as $(NH_4)_2SO_3$]

7.1.1 Specification

Content of $(NH_4)_2S_2O_3$ shall be between 53,0 % (*m*/*m*) min. and 61,0 % (*m*/*m*) max.

Alkalinity shall be between 0,3 % (*m*/*m*) min. and 1,5 % (*m*/*m*) max.

Maximum content of sulfite shall be 1 % (*m*/*m*).

7.1.2 Reagents

7.1.2.1 Sodium thiosulfate, Na_2SO_3, standard volumetric solution of 0,100 mol/l (15,81 g/l)[1].

7.1.2.2 Hydrochloric acid, HCl, standard volumetric solution of 0,100 mol/l (3,65 g/l)[1] [2].

7.1.2.3 Sodium hydroxide, NaOH, standard volumetric solution of 0,100 mol/l (4,00 g/l)[1] [3].

7.1.2.4 Iodine solution, I_2, standard volumetric solution of 0,050 mol/l (12,7 g/l)[1] [4].

7.1.2.5 Ethanol, C_2H_5OH, 95 % (*V*/*V*), denatured.

7.1.2.6 Methyl red indicator, 0,1 g/l.

Warm a mixture of 25 mg of methyl red (finely powdered) and 0,95 ml of a (1+1) mixture of water and sodium hydroxide (7.1.2.3) and 5 ml of ethanol (7.1.2.5). After solution is complete, add 125 ml of ethanol (7.1.2.5) and dilute to 250 ml with water.

7.1.2.7 Salicylic acid, $C_6H_6O_3$, 1 % (10 g/l).

Prepare a solution of 1 g of salicylic acid in 100 ml of water.

7.1.2.8 Starch indicator, 5 g/l.

Stir 5 g of soluble starch with 100 ml of salicylic acid (7.1.2.7). Then add 300 ml to 400 ml of boiling water and boil until the starch dissolves. Finally dilute to 1 litre with water.

---

[1] Commercially available analysed reagents are recommended. If solutions are to be prepared, see any quantitative analytical chemistry text.

[2] This solution may be prepared from concentrated hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>). (Hazard warning codes are defined in ISO 10349-1.)

[3] This solution may be prepared from sodium hydroxide pellets (DANGER: <<C>>).

[4] Self-prepared iodine solutions require standardization before use.

7.1.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

7.1.3.1 One-mark volumetric flask, of 500 ml capacity.

7.1.3.2 Pipette, of 50 ml capacity.

7.1.3.3 Burette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 8 g of the test sample into a 500 ml volumetric flask (7.1.3.1). Dilute with 100 ml of water. Add a few drops of the methyl red indicator (7.1.2.6) and immediately titrate with the hydrochloric acid (7.1.2.2) to the first colour change. Dilute the neutralized solution to the mark with water. Take, using the pipette (7.1.3.2), a 50 ml aliquot, then add 75 ml of water and 2 ml of the starch indicator (7.1.2.8). Titrate with the iodine solution (7.1.2.4) to a permanent blue colour (second endpoint). Then carefully add just enough of the standard sodium thiosulfate (7.1.2.1) to discharge the blue colour. Add a few drops of the methyl red indicator (7.1.2.6) and titrate with the sodium hydroxide (7.1.2.3) to the first colour change (third endpoint).

7.1.5 Expression of results

7.1.5.1 The assay for ammonium thiosulfate, expressed as a percentage by mass of $(NH_4)_2S_2O_3$, is given by

$$296{,}4(c_2 \cdot V_2 - 0{,}333 \cdot c_3 \cdot V_3)/m$$

where

c_2 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.4);

V_2 is the volume, in millilitres, of the iodine solution used to reach the endpoint of the second titration (7.1.4);

c_3 is the actual concentration, expressed in moles per litre, of the sodium hydroxide solution (7.1.2.3);

V_3 is the volume, in millilitres, of the sodium hydroxide used to reach the endpoint of the third titration (7.1.4);

296,4 is the conversion factor obtained from the molar mass of ammonium thiosulfate (i.e. 148,2) × 2 moles of thiosulfate per mole of iodine (i.e. 2) × the sample ratio (i.e. 10) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

0,333 is a correction factor for the ratio of acid generated by the iodine used in the oxidation of the sulfite [three equivalents of acid are formed (see note 1), two of which are due to the consumption of iodine];

m is the mass, in grams, of the test portion.

7.1.5.2 The alkalinity, expressed as a percentage by mass of ammonium hydroxide (NH_4OH), is given by

$$3{,}5 \cdot c_1 \cdot V_1/m$$

where

c_1 is the actual concentration, expressed in moles per litre, of the hydrochloric acid (7.1.2.2);

V_1 is the volume, in millilitres, of the hydrochloric acid used to reach the endpoint of the first titration (7.1.4);

3,5 is the conversion factor obtained from the mass of ammonium hydroxide equivalent to 1 mole of hydrochloric acid (i.e. 35) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

m is the mass, in grams, of the test portion.

7.1.5.3 The sulfite content, expressed as a percentage by mass of ammonium sulfite $[(NH_4)_2SO_3]$, is given by

$$38{,}7 \cdot c_3 \cdot V_3/m$$

where

c_3 is the actual concentration, expressed in moles of sodium hydroxide per litre, of the sodium hydroxide solution (7.1.2.3);

V_3 is the volume, in millilitres, of the sodium hydroxide used to reach the endpoint of the third titration (7.1.4);

38,7 is the conversion factor obtained from the mass of ammonium sulfite equivalent to one-third mole of sodium hydroxide (see note 1) (i.e. 0,333 3 × 116) × the sample ratio (i.e. 10) × the conversion factor for millilitres to litre (i.e. 0,001) × 100 (for percentage);

m is the mass, in grams, of the test portion.

NOTE 1 The pertinent chemical reactions for this analysis are the following.

Oxidation of ammonium thiosulfate:

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-$$

Oxidation of sulfite species:

$$HSO_3^- + I_2 + H_2O \longrightarrow HSO_4^- + 2I^- + 2H^+$$

$$HSO_4^- \longrightarrow H^+ + SO_4^{2-}$$

Three equivalents of acid are formed in the oxidation of sulfite by iodine, two by the oxidation and the third is due to the increased acidity of biosulfate vs. bisulfite. Thus a factor of 0,333 is used to correct for the acid due to the acidity difference.

7.2 Insoluble matter content (as a precipitate of calcium and magnesium in ammonium hydroxide)

7.2.1 Specification

Maximum content of insoluble matter shall be 0,2 % (*m*/*m*).

7.2.2. Procedure

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

7.3 Residue after ignition

7.3.1 Specification

Maximum residue after ignition shall be 0,2 % (*m*/*m*).

7.3.2 Procedure

Determine the residue after ignition in accordance with ISO 10349-4. Weigh, to the nearest 0,01 g, a test portion of about 10 g of the test sample. Evaporate to dryness on a steam bath and then continue the procedure in accordance with ISO 10349-4. Incinerate at 600 °C ± 50 °C, for 4 h. Cool in a desiccator and then weigh to the nearest 0,001 g. Retain this residue for the heavy metals (7.4) and iron (7.5) tests.

7.4 Heavy metals content (as Pb)

7.4.1 Specification

Maximum content of heavy metals shall be 0,001 % (*m*/*m*).

7.4.2 Procedure

NOTE 2 The standard for the iron test (7.5) 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 the residue from the ignition test (7.3.2) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 5 ml of the 25 ml residue solution). Use 2 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.5 Iron content

7.5.1 Specification

Maximum content of iron shall be 0,001 % (*m*/*m*).

7.5.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of the residue from the ignition test (7.3.2) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 5 ml of the 25 ml residue solution). Use 2 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.6 Sulfide content (as S^{2-})

7.6.1 Specification

Maximum content of sulfide shall be 0,000 25 % (*m*/*m*).

7.6.2 Procedure

Determine the percentage of sulfide in accordance with ISO 10349-10. Use a test portion, weighed to the nearest 0,05 g, of 2,0 g of the sample and mix with 40 ml of water. Use a 5 ml portion of the sulfide standard.

7.7 Specific gravity or density

Either the specific gravity or density test will be sufficient to satisfy conformity to the specification.

7.7.1 Specific gravity

7.7.1.1 Specification

The specific gravity shall be between 1,290 and 1,346 at 20 °C.

7.7.1.2 Procedure

Determine the specific gravity in accordance with ISO 10349-11, using a hydrometer which includes the range 1,25 to 1,35.

7.7.2 Density

7.7.2.1 Specification

The density shall be between 1,288 g/ml and 1,343 g/ml at 20 °C.

7.7.2.2 Procedure

Determine the density in accordance with ISO 10349-12.

This page intentionally left blank