

INTERNATIONAL
STANDARD

ISO
3622

Second edition
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**Photography — Processing chemicals —
Specifications for ammonium thiocyanate**

*Photographie — Produits chimiques de traitement — Spécifications
relatives au thiocyanate d'ammonium*



Reference number
ISO 3622:1996(E)

Foreword

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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 3622 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3622:1976), 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, 4, 5 and 6 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 thiocyanate

1 Scope

This International Standard establishes criteria for the purity of photographic-grade ammonium thiocyanate and specifies the test methods 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 10349-4:1992, *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 10349-6:1992, *Photography — Photographic-grade chemicals — Test methods — Part 6: Determination of halide content.*

3 General

3.1 Physical properties

Ammonium thiocyanate (NH_4CNS) exists as colourless, deliquescent crystals. It has a relative molecular mass of 76,12.

3.2 Hazardous properties

Ammonium thiocyanate presents no special hazardous problems under normal handling.

3.3 Storage

Store in a cool dry place. Care shall be taken to avoid conditions of high heat and/or humidity which can cause decomposition and emission of toxic fumes.

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.

7 Test procedures

7.1 Assay

7.1.1 Specification

Content of NH_4CNS shall be 97,0 % (*m/m*) min.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	97,0 % (<i>m/m</i>) min.	7.1	ISO 3622
Residue after ignition	0,10 % (<i>m/m</i>) max.	7.2	ISO 10349-4
Heavy metals content	0,002 % (<i>m/m</i>) max.	7.3	ISO 10349-5
Iron content	0,000 5 % (<i>m/m</i>) max.	7.4	ISO 10349-5
Halide content (as Cl ⁻)	0,006 % (<i>m/m</i>) max.	7.5	ISO 10349-6
pH value	4,5 to 6,0	7.6	ISO 3622
Sulfur compounds		7.7	ISO 3622
a) For developer solution, use amount precipitated by ammoniacal silver nitrate	0,005 % (<i>m/m</i>) max.	7.7.1	ISO 3622
b) For fixer solution, use iodine consumption	To pass test	7.7.2	ISO 3622
Sulfate (as SO_4^{2-})	0,04 % (<i>m/m</i>) max.	7.8	ISO 3622
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.9	ISO 3622

NOTE — *m/m* = mass/mass

7.1.2 Reagents

7.1.2.1 Nitric acid (HNO₃), (1 + 9) solution ¹⁾.

7.1.2.2 Ammonium iron(III) sulfate [(NH₄)Fe(SO₄)₂], 50 g/l solution.

7.1.2.3 Silver nitrate (AgNO₃), standard volumetric solution of 0,100 mol/l (17,0 g/l) ^{2) 3)}.

7.1.2.4 Ammonium thiocyanate (NH₄SCN), standard volumetric solution of 0,100 mol/l (7,61 g/l) ²⁾.

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

7.1.3.2 Pipette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 0,3 g and dissolve it in 50 ml of water. Add 5 ml of the nitric acid (7.1.2.1), followed by 50,00 ml of the silver nitrate (7.1.2.3) using the pipette (7.1.3.2). Shake well, add 2 ml of the ammonium iron(III) sulfate (7.1.2.2) and titrate the excess of silver nitrate with the ammonium thiocyanate (7.1.2.4) to a permanent red endpoint.

7.1.5 Expression of results

The assay, expressed as a percentage by mass of NH₄CNS, is given by

$$7,612(50 \cdot c_1 - V \cdot c_2)/m$$

where

*c*₁ is the actual concentration, in moles per litre, of the silver nitrate (7.1.2.3);

V is the volume, in millilitres, of the ammonium thiocyanate (7.1.2.4) used for the titration;

*c*₂ is the actual concentration, in moles per litre, of the ammonium thiocyanate (7.1.2.4);

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

50 is the volume, in millilitres, of silver nitrate added (7.1.4);

7,612 is the conversion factor obtained from the mass of ammonium thiocyanate equivalent to 1 mole of silver nitrate (i.e. 76,12) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Residue after ignition

7.2.1 Specification

Maximum content of residue after ignition shall be 0,10 % (*m/m*).

1) This solution may be prepared from concentrated nitric acid, 69 % (*m/m*) (approx.) (DANGER: (C)(B)(O)). (Hazard warning codes are defined in ISO 10349-1.)

2) Commercially available analysed reagent solutions are recommended. Procedures for the preparations of these materials are available in any quantitative analysis text.

3) This solution may be prepared from solid silver nitrate (DANGER: (C)).

7.2.2 Procedure

Determine the percentage residue after ignition in accordance with ISO 10349-4. Weigh about 5,0 g of sample and ignite it in a platinum crucible ($600\text{ °C} \pm 50\text{ °C}$, 4 h, 0,001 g). Cool in a desiccator and then weigh to the nearest 0,001 g.

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 — 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, 7.2. Use 4 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.4 Iron content

7.4.1 Specification

Maximum content of iron shall be 0,000 5 % (*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, 7.2. Use 1 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.5 Halide content (as Cl^-)

7.5.1 Specification

Maximum content of halide shall be 0,06 % (*m/m*) [0,1 % (*m/m*) as NH_4Cl].

7.5.2 Reagents

7.5.2.1 Sodium hydroxide (NaOH), solid (DANGER: <<C>>).

7.5.2.2 Hydrogen peroxide (H_2O_2), 67 g/l (approx.) solution.

Dilute 1 volume of 30 % hydrogen peroxide (DANGER: <<C>>O) with 4 volumes of water.

7.5.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 1 g and dissolve it in 30 ml of the hydrogen peroxide (7.5.2.2). Add 1 g of sodium hydroxide (7.5.2.1) (DANGER: <<C>>) and swirl until the vigorous reaction ceases. Add another 30 ml of hydrogen peroxide (7.5.2.2) and boil for 2 min. Cool and dilute to 100 ml with water. Use a 10 ml aliquot of this test solution. Continue the procedure in accordance with ISO 10349-6:1992, clause 4, using 6 ml of the halide standard solution A.

7.6 pH value

7.6.1 Specification

The pH of the solution shall be between 4,5 and 6,0.

7.6.2 Apparatus

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

7.6.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g and dissolve it in about 80 ml of boiled water. Dilute to 100 ml. Measure the pH of the solution at 20 °C, using the pH-meter (7.6.2.1) in accordance with the manufacturer's instructions.

7.7 Sulfur compounds

For use in non-developer solutions (e.g. fixers), the less-sensitive iodine consumption test is adequate, whereas for use in developers the more-sensitive ammoniacal silver nitrate test is required.

7.7.1 Amount precipitated by ammoniacal silver nitrate ⁴⁾

7.7.1.1 Specification

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

7.7.1.2 Reagents

7.7.1.2.1 Sulfide standard solution (1 ml = 0,010 mg S²⁻).

Dissolve 1,5 g of sodium sulfide nonahydrate (Na₂S·9H₂O) (DANGER: <S>) in 100 ml of water in a 200 ml volumetric flask. Dilute to volume with water and mix well. Pipette 1 ml of this solution into a 100 ml volumetric flask, dilute to volume with water and mix well. Label this solution "sulfide standard".

7.7.1.2.2 Ammonium hydroxide (NH₄OH), ρ = 0,91 g/ml (DANGER: <C>).

7.7.1.2.3 Silver nitrate (AgNO₃), 100 g/l solution ³⁾.

7.7.1.2.4 Silver nitrate, standard volumetric solution of 0,001 mol/l (0,170 g/l) ^{2) 3)}.

7.7.1.3 Apparatus

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

7.7.1.4 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 1 g and dissolve it in 25 ml of water. Add this solution to a mixture of 20 ml of ammonium hydroxide (7.7.1.2.2) (DANGER: <C>) and 2 ml of the silver nitrate (7.7.1.2.3).

Also prepare a control by adding 5 ml of sulfide standard (7.7.1.2.1) to a mixture of 20 ml of water, 20 ml of ammonium hydroxide (7.7.1.2.2) and 3 ml of 0,001 mol/l silver nitrate (7.7.1.2.4). Heat both solutions in a boiling water bath for 15 min. Cool, transfer to the Nessler colour-comparison cylinders (7.7.1.3.1) and dilute to 50 ml.

4) The test method given here for sulfur compounds is not the same as the ammoniacal silver nitrate test given in ISO 10349-9:1992, *Photography — Photographic-grade chemicals — Test methods — Part 9: Reaction to ammoniacal silver nitrate*.

Compare the colours produced in the two Nessler colour-comparison cylinders. Any colour produced in the sample shall not exceed that produced in the sulfide standard control.

CAUTION — Dispose of all test solutions and rinse all used apparatus immediately. Explosive compounds may be formed on standing.

7.7.2 Iodine consumption

7.7.2.1 Specification

To pass test.

7.7.2.2 Reagents

7.7.2.2.1 Iodine (I_2), standard volumetric solution of 0,100 mol/l²⁾.

7.7.2.2.2 Sulfuric acid (H_2SO_4), 10 % solution⁵⁾.

7.7.2.2.3 Salicylic acid ($HOC_6H_4CO_2H$), 1 % (10 g/l) solution.

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

7.7.2.2.4 Starch indicator, 5 g/l solution.

Stir 5 g of soluble starch with 100 ml of 1 % salicylic acid (7.7.2.2.3). Then add 300 ml to 400 ml of boiling water and boil until the starch dissolves. Finally dilute to 1 000 ml with water.

7.7.2.3 Procedure

Dissolve 4,95 g to 5,05 g of the sample in 50 ml of water. Add 1,7 ml of the sulfuric acid (7.7.2.2.2), 1 g of potassium iodide (KI) and 1 ml of starch indicator (7.7.2.2.4). Titrate with the iodine solution (7.7.2.2.1). Not more than 1,0 ml of the iodine solution shall be required.

7.8 Sulfate content (as SO_4^{2-})

7.8.1 Specification

Maximum content of sulfate shall be 0,04 % (m/m) [0,06 % (m/m) as $(NH_4)_2SO_4$].

7.8.2 Reagents

7.8.2.1 Hydrochloric acid (HCl), (1 + 9) solution⁶⁾.

7.8.2.2 Barium chloride

Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) (DANGER: (S)) in 1 000 ml of water.

7.8.2.3 Sulfate standard solution (1 ml = 0,10 mg SO_4^{2-}).

Dissolve a soluble sulfate salt in water [e.g. 181 mg of potassium sulfate (K_2SO_4) in 1 000 ml of water].

5) This solution may be prepared from concentrated sulfuric acid, $\rho \approx 1,84$ g/ml (DANGER: <<C>>).

6) This solution may be prepared from concentrated hydrochloric acid, $\rho \approx 1,18$ g/ml (DANGER: (C)(B)).

7.8.3 Apparatus

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

7.8.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 0,9 g and dissolve it in 30 ml of water in one of the Nessler colour-comparison cylinders (7.8.3.1). Transfer 4 ml of the sulfate standard (7.8.2.3) to the other Nessler colour-comparison cylinder. Add 0,5 ml of the hydrochloric acid (7.8.2.1) and 1 ml of the barium chloride (7.8.2.2) to each cylinder. Dilute each to 50 ml, mix well and allow to stand for 15 min.

Compare the turbidities produced in the two cylinders. The test sample turbidity shall not be greater than that of the reference sample.

7.9 Appearance of solution

7.9.1 Specification

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

7.9.2 Procedure

Prepare a 100 g/l solution of the sample in water. Observe the solution for colour and clarity.

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