INTERNATIONAL STANDARD

ISO 420

Second edition
1994-10-15

Photography — Processing chemicals — Specifications for potassium bromide

Photographie — Produits chimiques de traitement — Spécifications pour le bromure de potassium

Reference number
ISO 420:1994(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 420 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 420:1976), 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, 6, 7, 8 and 10 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 potassium bromide

1 Scope

This International Standard establishes criteria for the purity of photographic-grade potassium bromide and specifies the test methods to be used to determine the purity.

2 Normative references

The following International 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 10349-6:1992, *Photography — Photographic-grade chemicals — Test methods — Part 6: Determination of halide content.*

ISO 10349-7:1992, *Photography — Photographic-grade chemicals — Test methods — Part 7: Determination of alkalinity or acidity.*

ISO 10349-8:1992, *Photography — Photographic-grade chemicals — Test methods — Part 8: Determination of volatile matter.*

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

3 General

3.1 Physical properties

Potassium bromide (KBr) exists in the form of white crystals or a crystalline powder. It has a relative molecular mass of 119,00.

3.2 Hazardous properties

Potassium bromide is not hazardous when handled with normal precautions.

3.3 Storage

Potassium bromide 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.

7 Test methods

7.1 Assay

7.1.1. Specification

Content of KBr shall be between 99,0 % (*m/m*) min. and 100,3 % (*m/m*) max.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	99,0 % (*m/m*) min. 100,3 % (*m/m*) max.	7.1	ISO 420
Insoluble matter (as precipitate of calcium and magnesium in ammonium hydroxide)	0,3 % (*m/m*) max.	7.2	ISO 10349-3
Heavy metals (as Pb)	0,002 % (*m/m*) max.	7.3	ISO 10349-5
Iron (Fe)	0,002 % (*m/m*) max.	7,4	ISO 10349-5
Halides (as KCl)	0,50 % (*m/m*) max.	7.5	ISO 10349-6
Alkalinity (as KOH)	0,015 % (*m/m*) max.	7.6	ISO 10349-7
Acidity (as HBr)	0,010 % (*m/m*) max.	7.7	ISO 10349-7
Volatile matter	0,3 % (*m/m*) max.	7.8	ISO 10349-8
Sulfide (as K_2S)	0,000 8 % (*m/m*) max.	7.9	ISO 10349-10
Iodide	To pass test	7.10	ISO 420
Oxidizing substances	To pass test	7.11	ISO 420
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.12	ISO 420
NOTE – *m/m* = mass/mass			

7.1.2 Reagents

7.1.2.1 Nitric acid, HNO_3 (1 + 9)[1) 2)].

7.1.2.2 Ammonium iron(III) sulfate, $(NH_4)Fe(SO_4)_2$, 50 g/l.

7.1.2.3 Silver nitrate, $AgNO_3$, standard volumetric solution of 0,100 mol/l (16,987 g/l)[3)].

7.1.2.4 Ammonium thiocyanate, NH_4SCN, standard volumetric solution of 0,100 mol/l (7,612 g/l)[3)].

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 0,4 g of the sample and dissolve it in 50 ml of water. Add 5 ml of the nitric acid (7.1.2.1), followed by 50 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 silver ions with the ammonium thiocyanate (7.1.2.4) to the first persistent colour change.

7.1.5 Expression of results

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

$$11{,}90(50c_1 - c_2V)/m$$

where

c_1 is the actual concentration, expressed in moles per litre, of the silver nitrate (7.1.2.3);

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

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

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

2) This solution may be prepared from nitric acid, 69 % (*m/m*) (DANGER:<C><O>).

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

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

11,90 is the conversion factor obtained from the mass of potassium bromide equivalent to 1 mole of silver nitrate (i.e. 119,0) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

NOTE 1 The assay limit is based on materials as received and is not corrected for potassium chloride content. The presence of potassium chloride will increase the assay value; i.e. 1 g of potassium chloride is equivalent to 1,6 g of potassium bromide.

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 0,3 % (*m/m*).

7.2.2 Procedure

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

7.3 Heavy metals content (as Pb)

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 4,90 g to 5,10 g of the sample prepared in accordance with ISO 10349-5:1992, 7.2. Use 10 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 0,002 % (*m/m*).

7.4.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 4,90 g to 5,10 g of the sample prepared in accordance with ISO 10349-5:1992, 7.2. Use 10 ml of the iron standard prepared in accordance with ISO 10349-5: 1992, 8.1.1.

7.5 Halide content

7.5.1 Specification

Maximum content of halides shall be 0,25 % (*m/m*) as Cl^- (0,50 % as KCl).

7.5.2 Reagents

7.5.2.1 Nitric acid, HNO_3 (1+2) (DANGER: <C><O>)[1) 2)].

7.5.2.2 Hydrogen peroxide, H_2O_2 (DANGER: <C><O>), approximately 167 g/l) (1+1).

Dilute 30 % hydrogen peroxide (DANGER:<<C>> <O>) with an equal volume of water.

7.5.3 Procedure

Weigh, to the nearest 0,01 g, a test portion of 1 g and dissolve it in 15 ml of the nitric acid (7.5.2.1) (<C><O>) in a conical flask. Add 6 ml of the hydrogen peroxide (7.5.2.2) (<C><O>) and digest on a steam bath until the solution is colourless. Wash down the sides of the flask with water, digest for an additional 15 min and dilute to 250 ml with water.

Transfer a 25 ml aliquot of the test solution into a 100 ml beaker. Pipette a 25 ml portion of the halide standard A in accordance with sentence 3 of clause 7 of ISO 10349-6:1992 and then continue in accordance with the rest of the procedure.

7.6 Alkalinity (as KOH)

7.6.1 Specification

Maximum free alkali content shall be 0,015 % (*m/m*).

7.6.2 Procedure

Prepare a test solution in accordance with ISO 10349-7 using a test portion of 3,9 g to 4,1 g. If the prepared test solution turns pink when the indicator is added, determine the percentage alkalinity as potassium hydroxide using a factor K equal to 5,61 in the calculation given in ISO 10349-7.

7.7 Acidity (as HBr)

7.7.1 Specification

Maximum free acid content shall be 0,010 % (*m/m*).

7.7.2 Procedure

If the prepared test solution in the alkalinity determination remains clear when the indicator is added, determine the percentage acidity as hydrobromic acid in accordance with ISO 10349-7. Use a factor K equal to 8,09 in the calculation given in ISO 10349-7.

7.8 Volatile matter

7.8.1 Specification

Maximum percentage of volatile matter shall be 0,3 % (*m/m*).

7.8.2 Procedure

Determine the percentage of volatile matter in accordance with ISO 10349-8. Use a test portion of about 5,0 g, weighed to the nearest 0,000 1 g, and heat in an oven at 105 °C± 5 °C for 4 h.

7.9 Sulfide content

7.9.1 Specification

Maximum content of sulfides shall be 0,000 24 % (*m/m*) as S^{2-} (0,000 8 % as K_2S).

7.9.2 Procedure

Determine the percentage of sulfides in accordance with ISO 10349-10. Use a test portion of 1,90 g to 2,10 g, and mix with 40 ml of water. Use a 5 ml portion of the sulfide standard.

7.10 Iodide content

7.10.1 Specification

To pass test.

7.10.2 Reagents

7.10.2.1 Carbon tetrachloride (CCl_4) or **chloroform** ($CHCl_3$) (DANGER:<B><S>)[4].

7.10.2.2 Sulfuric acid, H_2SO_4 (1 + 9)[5].

7.10.2.3 Iron(III) chloride, $FeCl_3$, 100 g/l.

Dissolve 100 g of iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) in 800 ml of water and dilute to 1 litre.

7.10.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g and dissolve it in 25 ml of water. Add 1 ml of the sulfuric acid (7.10.2.2), 0,5 ml of the iron(III) chloride (7.10.2.3) and 1 ml of the carbon tetrachloride or chloroform (7.10.2.1) (<B><S>). Shake the mixture vigorously and allow the layers to separate.

There shall be no violet tint in the organic layer.

7.11 Oxidizing substances content

7.11.1 Specification

To pass test.

7.11.2 Reagent

7.11.2.1 Carbon tetrachloride (CCl_4) or **chloroform** ($CHCl_3$) (DANGER:<B><S>)[4].

7.11.2.2 Potassium iodide, KI.

7.11.2.3 Sulfuric acid, H_2SO_4 (1 + 9)[5].

7.11.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g and dissolve it in 10 ml of water in a test tube. Treat this solution, and 10 ml of water in a second test tube (control), in the following manner. Add 0,5 g of potassium iodide (7.11.2.2), 1 ml of sulfuric acid (7.11.2.3) and 1 ml of the carbon tetrachloride or chloroform (7.11.2.1) (<B><S>). Stopper the test tube and shake the mixture vigorously. Allow the layers to separate.

Compare, in the test tubes, the colours produced in the organic layers of the test and control samples. The sample shall be no greater in colour than the control sample.

7.12 Appearance of solution

7.12.1 Specification

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

7.12.2 Procedure

Dissolve a test portion of 20 g in 100 ml of water. Observe the solution for colour and clarity.

4) Concern over the use of halogenated hydrocarbons as solvents (chloroform or carbon tetrachloride) has resulted in a request for alternatives to the use of these solvents in this test procedure. Iodine is soluble in the following solvents, some of which may be suitable for use in this test procedure: toluene, *p*-xylene, mesitylene, hexane (petroleum ether or ligroin), cyclohexane, diethyl ether <<F>>, *n*-butanol and *n*-octenol. These solvents have not yet been evaluated in this test procedure and their usage in lieu of the chlorinated solvents requires confirmation of the validity of the procedure by the user.

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

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ICS 37.040.30

Descriptors: photography, photographic materials, photographic chemicals, potassium bromide, specifications, materials specifications, purity criteria, tests, chemicals analysis.

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