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INTERNATIONAL STANDARD

ISO 3620

Second edition
1994-12-15

Corrected and reprinted
1995-02-01

Photography — Processing chemicals — Specifications for aluminium potassium sulfate

Photographie — Produits chimiques de traitement — Spécifications pour le sulfate double d'aluminium et de potassium

Reference number
ISO 3620:1994(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 3620 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3620:1976), which has been technically revised.

Annex A of this International Standard is for information only.

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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 and 5 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.

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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.

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Photography — Processing chemicals — Specifications for aluminium potassium sulfate

1 Scope

This International Standard establishes criteria for the purity of photographic-grade aluminium potassium sulfate dodecahydrate, $AlK(SO_4)_2 \cdot 12H_2O$, 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 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.*

3 General

3.1 Physical properties

Aluminium potassium sulfate dodecahydrate [$AlK(SO_4)_2 \cdot 12H_2O$] exists in the form of colourless crystals or a white powder. It has a relative molecular mass of 474,39.

3.2 Hazardous properties

Aluminium potassium sulfate is not hazardous when handled with normal precautions.

3.3 Storage

Aluminium potassium sulfate should 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 $AlK(SO_4)_2 \cdot 12H_2O$]	99,5 % (*m/m*) min.	7.1	ISO 3620
Heavy metals (as Pb)	0,005 % (*m/m*) max.	7.2	ISO 10349-5
Iron (Fe)	0,01 % (*m/m*) max.	7.3	ISO 10349-5
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.4	ISO 3620
NOTE — *m/m* = mass/mass			

7 Test methods

7.1 Assay

7.1.1 Specification

Content of $AlK(SO_4)_2 \cdot 12H_2O$ shall be 99,5 % (*m/m*) min.

7.1.2 Reagents

7.1.2.1 EDTA solution, standard volumetric solution of 0,05 mol/l (16,8 g/l of the disodium salt, $C_{10}H_{14}Na_2N_2O_8$)[1].

7.1.2.2 Lead nitrate, $Pb(NO_3)_2$, standard volumetric solution of 0,05 mol/l (16,56 g/l) (DANGER: <<S>>)[2].

Dissolve 16,56 g ± 0,01 g of lead nitrate (<<S>>) in 500 ml of water in a 1 litre volumetric flask and make up to the mark with water.

7.1.2.3 Xylenol orange indicator, 1 g/l[3].

7.1.2.4 Hexamine (hexamethylene tetramine)[4], $C_6H_{12}N_4$, solid.

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

7.1.3.2 Pipette, of 20 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 2,25 g of the test sample. Dissolve it in water and dilute to 100 ml. Using the pipette (7.1.3.2), take 20 ml of this solution and add 40,0 ml of the EDTA solution (7.1.2.1) followed by 100 ml of water. Heat on a water bath for 10 min then cool. Add 1 g of hexamine (7.1.2.4) and, using the burette (7.1.3.1) titrate with the lead nitrate (7.1.2.2) (<<S>>) using 0,4 ml of the xylenol orange indicator (7.1.2.3). The endpoint occurs when the solution goes from red to yellow in colour.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, of aluminium potassium sulfate dodecahydrate $[AlK(SO_4)_2 \cdot 12H_2O]$ is given by the formula

$$(474,4 - 237,2 \cdot c \cdot V)/m$$

where

c is the actual concentration, in moles per litre, of the lead nitrate (7.1.2.2);

V is the volume, in millilitres, of the lead nitrate used for the titration (7.1.4);

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

474,4 is the conversion factor obtained from the mass of aluminium potassium sulfate dodecahydrate $[AlK(SO_4)_2 \cdot 12H_2O]$ (i.e. 474,4) equivalent to 1 mole of EDTA × the EDTA used (i.e. 40 × 0,05) × the sample ratio (i.e. 5) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

237,2 is a conversion factor inclusive of the factors above (i.e. 474,4) corrected by a factor of 2 to compensate for EDTA consumption by lead nitrate vs. that of aluminium potassium sulfate.

7.2 Heavy metals content

7.2.1 Specification

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

7.2.2 Procedure

NOTE 1 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 0,99 g to 1,01 g prepared in accordance with ISO 10349-5:1992, 7.2. Use 5 ml of the heavy metals standard prepared in accordance with ISO 10349-5: 1992, 8.1.2.

7.3 Iron content

7.3.1 Specification

Maximum content of iron shall be 0,01 % (*m/m*).

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1) Commercially analytical reagent is recommended. A procedure for the preparation and standardization of EDTA solution is given in annex A.

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

3) Xylenol orange solutions have been reported to be unstable. It is recommended that solutions be prepared on a daily basis.

4) Also known as: methenamine, hexamethylenamine, 1,3,5,7-tetraazaadamantane, aminoform, cystamin, cystogen, formin uritone and urotropin.

7.3.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 0,49 g to 0,51 g of the sample prepared in accordance with ISO 10349-5: 1992, 7.2. Use 5 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.4 Appearance of solution

7.4.1 Specification

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

7.4.2 Procedure

Prepare a 50 g/l solution of the sample in water. Examine the solution for colour and clarity.

Annex A

(informative)

Preparation of EDTA solution: Standard volumetric solution of 0,05 mol/l (16,8 g)

A.1 Reagents

A.1.1 Ethylenediaminetetraacetic acid (EDTA) dihydrate, disodium salt, $C_{10}H_{14}N_2Na_2O_8{\cdot}2H_2O$.

NOTE 2 The relative molecular mass for the dihydrate salt is 372,23. The relative molecular mass for the non-hydrated salt is 336,20.

A.1.2 Calcium carbonate, $CaCO_3$, chelometric standard grade material[4].

A.1.3 Hydrochloric acid, HCl, (1 + 3)[5].

A.1.4 Sodium hydroxide, NaOH, standard volumetric solution of 1 mol/l (40,0 g/l)[6].

A.1.5 Hydroxynaphthol blue indicator.

A.2 Apparatus

A.2.1 Burette, of 50 ml capacity.

A.2.2 Pipette, of 2 ml capacity.

A.3 Procedure

Dissolve 20 g of disodium EDTA dihydrate (A.1.1) in water and dilute to 1 litre.

Weigh, to the nearest 0,000 1 g, about 0,200 g of the calcium carbonate (A.1.2). Transfer the calcium carbonate to a 400 ml beaker and add 10 ml of water. Cover the beaker with a watch glass and add 2 ml of hydrochloric acid (A.1.3) from a pipette (A.2.2) inserted between the watch glass and the lip of the beaker. Swirl the beaker to assist dissolution of the calcium carbonate. Wash down the sides of the beaker, the watch glass and the outside of the pipette and dilute the solution to about 100 ml with water. While stirring with a magnetic stirrer, add about 30 ml of the prepared EDTA solution using the burette (A.2.1), followed by 15 ml of sodium hydroxide (A.1.4) and 0,300 g of hydroxynaphthol blue indicator (A.1.5). Continue the titration with the EDTA solution to the endpoint when a blue colour appears.

A.4 Expression of results

The actual concentration of the EDTA solution, c, in moles per litre, is given by the formula

$$c = m/(0{,}100\ 09 \cdot V)$$

where

m is the mass, in grams, of the calcium carbonate (A.1.2);

V is the volume, in millilitres, of the prepared EDTA solution (A.1.1);

0,100 09 is the relative molecular mass of calcium carbonate (i.e. 100,09) × the conversion factor for millilitres to litres (i.e. 0,001).

Adjust the concentration of the EDTA solution to exactly 0,05 mol/l by diluting with water. The volume of water required, in millilitres, V_W, is given by the formula

$$V_W = (c \cdot S/0{,}05) - S$$

where

c is the actual concentration of the prepared EDTA solution, in moles per litre;

S is the volume, in millilitres, of the prepared EDTA solution to be diluted.

4) It is recommended that the calcium carbonate be dried to constant weight in a low-temperature oven and maintained in a desiccator to prevent absorption of water.

5) This solution can be prepared from concentrated hydrochloric acid, $\rho \approx 1{,}18$ (DANGER: <C><B>).

6) This solution can be prepared from solid sodium hydroxide (DANGER: <<C>>).

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ICS 37.040.30

Descriptors: photography, photographic materials, chemical reagents, aluminium potassium sulphate, specifications, materials specifications, purity criteria, tests, chemical analysis.

Price based on 4 pages