INTERNATIONAL
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Photography — Processing chemicals — Specifications for anhydrous potassium carbonate

Photographie — Produits chimiques de traitement — Spécifications pour le carbonate de potassium anhydre

Reference number
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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 3623 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3623:1976), which has been technically revised.

Annex A of this International Standard is for information only.

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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, 5, 6, 7, 8 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 potassium carbonate

1 Scope

This International Standard establishes criteria for the purity of photographic-grade anhydrous potassium carbonate 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.*

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 10349-9:1992, *Photography — Photographic-grade chemicals — Test methods — Part 9: Determination of reaction to ammoniacal silver nitrate.*

3 General

3.1 Physical properties

Anhydrous potassium carbonate, K_2CO_3, exists in the form of colourless white granules or powder. It has a relative molecular mass of 138,21.

3.2 Hazardous properties

Potassium carbonate is irratating to the eyes, skin and throat.

3.3 Storage

Potassium carbonate 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.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of K_2CO_3 shall be 97,0 % (m/m) min.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay (as K_2CO_3)	97,0 % (*m/m*) min.	7.1	ISO 3623
Heavy metals (as Pb)	0,001 % (*m/m*) max.	7.2	ISO 10349-5
Iron (Fe)	0,001 % (*m/m*) max.	7.3	ISO 10349-5
Halides (as Cl^-)	0,1 % (*m/m*) max.	7.4	ISO 10349-6
Volatile matter at 150 °C	2,0 % (*m/m*) max.	7,5	ISO 10349-8
Reaction to ammoniacal silver nitrate	To pass test	7.6	ISO 10349-9
Free alkali (as KOH)	1,0 % (*m/m*) max.	7.7	ISO 3623
Bicarbonate (as $KHCO_3$)	0,4 % (*m/m*) max.	7.8	ISO 3623
Calcium and magnesium (as Mg)	0,015 % (*m/m*) max.	7.9	ISO 3623
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.10	ISO 3623

NOTE — *m/m* = mass/mass

7.1.2 Reagents

7.1.2.1 Hydrochloric acid, HCl, standard volumetric solution of 1,00 mol/1 [1] [2].

7.1.2.2 Methyl orange indicator, 0,4 g/l.

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 2,5 g and dissolve it in 50 ml of water. Add two drops of the methyl orange indicator (7.1.2.2) and titrate with the hydrochloric acid (7.1.2.1) to the endpoint when the colour changes from yellow to red.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for K_2CO_3, is given by

$$6{,}91 \cdot c \cdot V/m$$

where

c is the actual concentration, expressed in moles per litre, of the hydrochloric acid (7.1.2.1);

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

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

6,91 is the conversion factor obtained from the mass of potassium carbonate equivalent to 1 mole of hydrochloric acid (i.e. 138,2/2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Heavy metals content

7.2.1 Specification

Maximum content of heavy metals shall be 0,001 % (*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 4,90 g to 5,10 g prepared in accordance with ISO 10349-5:1992, 7.3. 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 0,001 % (*m/m*).

7.3.2 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

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

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

the sample prepared in accordance with ISO 10349-5:1992, 7.3. Use 5 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.4 Halides content (as Cl⁻).

7.4.1 Specification

Maximum content of halides shall be 0,1 % (*m*/*m*) as Cl⁻.

7.4.2 Procedure

Determine the percentage of halides (expressed as Cl⁻) in accordance with ISO 10349-6. Use 10 ml of the Halide A standard.

7.5 Volatile matter

7.5.1 Specification

Maximum percentage of volatile matter shall be 2,0 % (*m*/*m*).

7.5.2 Procedure

Determine the percentage of volatile matter in accordance with ISO 10349-8. Use a test portion of about 5,0 g and place in an oven at 150 °C for 4 h (150 °C, 4 h, 0,000 1 g) [3].

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 Free alkali content (as KOH)

7.7.1 Specification

Maximum free alkali content, as KOH, shall be 1,0 % (*m*/*m*).

7.7.2 Reagents

7.7.2.1 Carbon-dioxide-free water

Prepare carbon-dioxide-free water in accordance with ISO 10349-7.

7.7.2.2 Phenolphthalein indicator, 5 g/l.

Dissolve 0,5 g of phenolphthalein in 50 ml of methanol or ethanol then dilute to 100 ml with water. Filter if necessary.

7.7.2.3 Sodium hydroxide, NaOH, standard volumetric solution of 0,100 mol [1].

7.7.2.4 Barium chloride, $BaCl_2$, neutral solution.

Dissolve 244 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) (DANGER: <C><B><S>)[4] in 1 litre of carbon-dioxide-free water (7.7.2.1). Check that the solution is neutral to phenolphthalein indicator (7.7.2.2) and, if not, adjust with a few drops of sodium hydroxide solution (7.7.2.3).

7.7.2.5 Hydrochloric acid, HCl, standard volumetric solution of 0,100 mol/l [1] [2].

7.7.3 Apparatus

7.7.3.1 Pipette, of 25 ml capacity.

7.7.3.2 Burette, of 50 ml capacity.

7.7.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g and dissolve it in 30 ml of freshly boiled water in a 125 ml stoppered conical flask. Add 25 ml of the barium chloride (7.7.2.4), followed by 10 drops of the phenolphthalein indicator (7.7.2.2) and mix well. If the solution has a pink colour, determine the alkalinity in accordance with ISO 10349-7:1992, 7.1, titrating with the hydrochloric acid (7.7.2.5) until the colour is discharged. Use a *K* factor equal to 5,61 in the calculation. (If no pink colour is produced, proceed to 7.8, using the same solution.)

7.7.5 Test limit

A maximum alkali content of 1,0 % (*m*/*m*), as KOH, is equivalent to no more than 8,9 ml of the hydrochloric acid (7.7.2.5) being used to discharge the pink colour.

7.8 Bicarbonate content (as $KHCO_3$)

7.8.1 Specification

Maximum content of bicarbonate, as $KHCO_3$, shall be 0,4 % (*m*/*m*).

7.8.2 Reagents

As specified in 7.7.2.

7.8.3 Apparatus

As specified in 7.7.3.

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[3] The notation system used for the drying procedure is described in ISO 10349-1:1992, clause 9.

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

7.8.4 Procedure

If the solution from 7.7.4 is not pink, determine the bicarbonate content in accordance with ISO 10349-7:1992, 7.2 titrating with the sodium hydroxide solution (7.7.2.3) until a faint pink colour, which persists for 30 s, is obtained. Use a K factor equal to 10,01 in the calculation.

7.8.5 Test limit

A maximum bicarbonate content of 0,4 % (m/m), as $KHCO_3$, is equivalent to no more than 2,0 ml of the sodium hydroxide solution (7.7.2.3) being used to produce the pink colour.

7.9 Calcium and magnesium content (as Mg)

7.9.1 Specification

Maximum content of calcium and magnesium shall be 0,015 % (m/m), as Mg.

7.9.2 Reagents

7.9.2.1 Ammonium hydroxide, NH_4OH, $\rho \approx 0{,}91$ g/ml (DANGER: <B>).

7.9.2.2 Buffer solution, pH 9,5 to 10,0.

Dissolve 54 g of ammonium chloride (NH_4Cl) in 200 ml of water. Add 350 ml of ammonium hydroxide (7.9.2.1) (<C><B>) and dilute to 1 litre.

7.9.2.3 EDTA solution, standard volumetric solution of 0,01 mol/l (3,36 g/l of the disodium salt, $C_{10}H_{14}Na_2N_2O_8$)[2] [5].

7.9.2.4 Hydrochloric acid, HCl, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>).

7.9.2.5 Magnesium standard solution (1 ml = 1 mg Mg).

Dissolve 10,141 g of magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) in water containing 1 ml of hydrochloric acid (7.9.2.4) (<C><B>) in a 1 litre volumetric flask. Dilute to the mark with water and mix.

7.9.2.6 Mordant black indicator

Grind 0,25 g of Mordant black 11[6] with 25 g of sodium chloride (NaCl) in a mortar.

7.9.3 Apparatus

7.9.3.1 One-mark volumetric flask, of 1 litre capacity.

7.9.3.2 Burette, of 50 ml capacity.

7.9.4 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 2,5 g and dissolve it in 90 ml of water. Using litmus paper, neutralize with hydrochloric acid (7.9.2.4) (<C><B>), boil for 5 min and cool. Prepare a ten-fold dilution (1 + 9) of a portion of the magnesium standard solution (7.9.2.5) and add 1 ml of this diluted solution, followed by 5 ml of the buffer solution (7.9.2.2) and 1,0 g of the Mordant black 11 indicator (7.9.2.6). Titrate with the EDTA solution (7.9.2.3) to the endpoint when the colour changes from violet-red to blue. Carry out a similar titration on a blank solution which is treated the same way except for the addition of the test portion.

7.9.5 Expression of results

The calcium and magnesium content, as Mg, expressed as a percentage by mass of the K_2CO_3, is given by

$$0{,}024\,3(V_1 - V_2)/m$$

where

V_1 is the volume, in millilitres, of the EDTA solution (7.9.2.3) used to reach the endpoint of the test solution;

V_2 is the volume, in millilitres, of the EDTA solution (7.9.2.3) used to reach the endpoint of the blank;

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

0,024 3 is the conversion factor obtained from the mass of magnesium equivalent to 1 mole of EDTA (i.e. 24,3) × the concentration of the EDTA solution (i.e. 0,01) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.10 Appearance of solution

7.10.1 Specification

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

7.10.2 Procedure

Dissolve a test portion of 200 g of the test sample and dissolve in 1 litre of water. Observe the solution for colour and clarity.

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[5] A procedure for the preparation and standardization of EDTA solution is given in annex A.

[6] Listed in colour index as C.I. 14645. Chrome Fast Black CAT, KIT*TS, Eriochrome Black DW, T and TDW, and Potting Black C are examples of suitable products available commercially. This information is given for the convenience of users of the International Standard and does not constitute an endorsement by ISO of these products.

Annex A

(informative)

Preparation of EDTA solution: Standard volumetric solution of 0,01 mol/l (3,36 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[7].

A.1.3 Hydrochloric acid, HCl (1 + 3)[8].

A.1.4 Sodium hydroxide, NaOH, standard volumetric solution of 1 mol/l (40,0 g/l)[9].

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 0,050 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 1 + 3 (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 40 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 0,01 mol/l by diluting with water. The volume of water required, in millilitres, V_W, is given by the formula

$$V_W = (c \cdot S/0{,}01) - S$$

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.

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[7] It is recommended that the calcium carbonate be dried to constant mass in a low-temperature oven and maintained in a desiccator to prevent absorption of water.

[8] This solution can be prepared from concentrated hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>).

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