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# INTERNATIONAL STANDARD ISO 3623

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# Photographic grade potassium carbonate, anhydrous — Specification

*Carbonate de potassium anhydre de qualité photographique — Spécifications*

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# Photographic grade potassium carbonate, anhydrous — Specification

## 0 INTRODUCTION

This International Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality, and to furnish manufacturers, suppliers, and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

Photographic grade chemicals are those which meet the requirements specified in the appropriate International Standards. These specifications set out purity standards and state the limiting concentrations and test methods for certain inert or photographically harmful impurities that may be present.

Originally these specifications were based on known requirements for black-and-white photographic processing, but increased attention has been paid to the requirements of colour processing. Experience to date indicates that chemicals meeting these specifications are satisfactory for colour processes in general use.

### 0.1 Specification requirements

These specifications set out chemical and physical requirements. While it is recognized that the ultimate criterion of the quality of a photographic chemical is its successful performance in a photographic test, present knowledge indicates that, from a practical standpoint, chemical and physical methods of testing are generally adequate. The photographic industry has accumulated a comprehensive collection of such chemical tests for impurities. These tests, which correlate with objectionable photographic effects, have been drawn upon in the formulation of these specifications. Chemical tests are generally more sensitive, less variable, and less costly than photographic tests.

Purity requirements have been set as low as possible, consistent with the objectives mentioned. If, however, 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 higher-quality materials.

Every effort has been made to keep the number of requirements in each specification to a minimum. The requirements generally include only those photographically harmful impurities which, through experience, are likely to be present. Inert impurities are limited to amounts which will not unduly reduce the assay.

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. All assays are intended to be made on undried samples in view of the fact that photographic processing chemicals are normally used "as received".

Identity tests have been included in the specifications wherever a possibility exists that another chemical or a mixture of chemicals could pass the other tests.

All requirements listed in clause 3 of each specification are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

### 0.2 Selection of test methods

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.

While the test methods set out in the specifications are recommended, the use of other equally reliable methods is allowed. 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.

### 0.3 Reagents

An effort has been made to minimize the number of reagents employed in this series of specifications. The methods of preparation and of standardization have been included in all cases where these are not common, or where a preferred method is desirable.

Details of reagent preparation and standardization are included in each specification in which the reagent is called for so that each specification shall be self-sufficient.

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## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the purity requirements of, and test methods for, photographic grade potassium carbonate, anhydrous.

## 2 CHARACTERISTICS

Potassium carbonate, anhydrous, is in the form of white granules or powder, of chemical formula $K_2CO_3$ and relative molar mass 69,1.

## 3 REQUIREMENTS

### 3.1 Assay

The assay shall be not less than 97,0 % (*m/m*), expressed as $K_2CO_3$, when determined by the method described in 4.1.

### 3.2 Appearance of solution

An aqueous solution shall be clear and free from sediment, other than a slight flocculence, when examined by the method described in 4.2.

### 3.3 Calcium and magnesium content

The calcium and magnesium content, expressed as magnesium (Mg), shall be not greater than 0,015 % (*m/m*), when determined by the method described in 4.3.

### 3.4 Volatile matter at 150 °C

The volatile matter at 150 °C shall be not greater than 2,0 % (*m/m*), when determined by the method described in 4.4.

### 3.5 Free alkali contant

The free alkali content, expressed as KOH, shall be not greater than 1,0 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.5, when not more than 8,9 ml of 0,1 M hydrochloric acid solution shall be required to discharge the colour from the test solution.

### 3.6 Halide content

The halide content, expressed as KCl, shall be not greater than 0,2 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.6, when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

### 3.7 Bicarbonate content

The bicarbonate content, expressed as $KHCO_3$, shall be not greater than 0,4 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.7, when not more than 2,0 ml of 0,1 M sodium hydroxide solution shall be required to produce a faint pink colour, persisting for 30 s, in the test solution.

### 3.8 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 10 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.8, when the colour produced in the test solution shall be not greater than that produced in the control solution.

### 3.9 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 10 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.9, when the colour produced in the test solution shall be not greater than that produced in the control solution.

### 3.10 Reaction to ammoniacal silver nitrate

The colour or precipitate produced in the test solution by ammoniacal silver nitrate solution shall be not greater than that produced in the control solution by ammonia solution, when examined by the method described in 4.10.

## 4 TEST METHODS

Reagents used in the tests shall be recognized reagent grade chemicals normally used for careful analytical work. In all the directions the acids and ammonia solution referred to shall be of full strength unless dilution is specified. Dilution is specified in terms of molar concentration (molarity)[1] when standardization of the reagent is required. When dilution is indicated as (1 + *x*), it means that 1 volume of the reagent or strong solution is added to *x* volumes of distilled water.

Distilled water, or water otherwise produced of at least equal purity, shall be used whenever water is required.

### 4.1 Assay

#### 4.1.1 *Reagents*

**4.1.1.1 Hydrochloric acid,** 1 M standard volumetric solution.

**4.1.1.2 Methyl orange** indicator solution, 0,4 g/l.

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[1] 1 mol/l = 1 kmol/m$^3$ = 1 mol/dm$^3$ = 1 M

### 4.1.2 *Apparatus*

Ordinary laboratory apparatus and

**4.1.2.1 Burette,** 50 ml capacity, conforming to class A of ISO/R 385.

### 4.1.3 *Procedure*

Weigh, to the nearest 0,001 g, a test portion of about 2,5 g of the laboratory sample and dissolve in 50 ml of water. Add 2 drops of the methyl orange indicator solution (4.1.1.2) and titrate with the hydrochloric acid solution (4.1.1.1) to the colour change from yellow to red.

### 4.1.4 *Calculation*

The assay, expressed as a percentage by mass of potassium carbonate ($K_2CO_3$), is given by the formula

$$\frac{6{,}91\,VT}{m}$$

where

$V$ is the volume, in millilitres, of the hydrochloric acid solution (4.1.1.1) used for the titration;

$T$ is the exact molarity of the hydrochloric acid solution (4.1.1.1);

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

### 4.2 Appearance of solution test

Dissolve 200 g of the laboratory sample in 1 000 ml of water and examine for clarity and sediment.

### 4.3 Determination of calcium and magnesium content

### 4.3.1 *Reagents*

**4.3.1.1 Hydrochloric acid** solution, $\rho$ approximately 1,18 g/ml.

**4.3.1.2 Buffer solution,** pH 9,5 to 10,0.

Dissolve 54 g of ammonium chloride in 200 ml of water, add 350 ml of ammonia solution, $\rho$ approximately 0,910 g/ml, and dilute to 1 000 ml.

**4.3.1.3 Magnesium,** standard solution.

Dissolve 10,140 g of magnesium sulphate heptahydrate ($MgSO_4.7H_2O$) in water containing 1 ml of the hydrochloric acid solution (4.3.1.1) in the one-mark volumetric flask (4.3.2.1). Dilute to the mark and mix.

1 ml of this solution contains 1 mg of magnesium.

**4.3.1.4 Zinc,** 0,5 M standard volumetric solution.

**4.3.1.5 (Ethylenedinitrilo)tetraacetic acid (EDTA), *di*sodium salt,** dihydrate, 0,001 M standard volumetric solution.

Dissolve 18,6 g of *di*sodium EDTA, dihydrate, in water and dilute to 1 000 ml. Standardize by titrating against the standard volumetric zinc solution (4.3.1.4) in 5 ml of the buffer solution (4.3.1.2) and 70 ml of water, using 0,1 g of the Mordant black 11 indicator (4.3.1.6). The end-point is indicated by a colour change from violet-red to blue. Using the factor of the zinc solution (4.3.1.4), dilute the *di*sodium EDTA solution with water to exactly 0,01 M.

**4.3.1.6 C.I. 14645, C.I. Mordant black 11** indicator.[1]

Grind 0,25 g of mordant black 11 with 25 g of sodium chloride in a mortar.

**4.3.2.1 One-mark volumetric flask,** 1 000 ml capacity, conforming to class A of ISO 1042.

### 4.3.2 *Apparatus*

Ordinary laboratory apparatus and

**4.3.2.2 Burette,** 50 ml capacity, conforming to class A of ISO/R 385.

### 4.3.3 *Procedure*

Weigh, to the nearest 0,01 g, a test portion of about 2,5 g of the laboratory sample and dissolve in 90 ml of water. Using litmus paper, neutralize with the hydrochloric acid solution (4.3.1.1), boil for 5 min and cool. Dilute a portion of the standard magnesium solution (4.3.1.3) 1 + 9 and add 1 ml of this, followed by 5 ml of the buffer solution (4.3.1.2) and 0,1 g of the Mordant black 11 indicator (4.3.1.6). Titrate with the standard volumetric *di*sodium EDTA solution (4.3.1.5) to the colour change from violet-red to blue. Carry out a similar titration on a blank solution omitting the test portion but containing the same quantities of all the other reagents.

### 4.3.4 *Calculation*

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

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

where

$V_1$ is the volume, in millilitres, of the *di*sodium EDTA solution (4.3.1.5) used for the titration of the test solution;

$V_2$ is the volume, in millilitres, of the *di*sodium EDTA solution (4.3.1.5) used for the titration of the blank;

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

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[1] Listed in Colour Index as C.I. 14645. Trade names include Chrome Fast Black CAT, KIT & TC, Eriochrome Black DW, T and TDW, Potting Black C, etc.

### 4.4 Determination of volatile matter at 150 °C

#### 4.4.1 *Apparatus*

Ordinary laboratory apparatus and

**4.4.1.1 Oven,** capable of being controlled at 150 ± 5 °C.

#### 4.4.2 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample into a previously weighed low-form, glass-stoppered weighing bottle. Remove the stopper and heat in the oven (4.4.1.1), controlled at 150 ± 5 °C, for 4 h. Cool in a desiccator, replace the cover and weigh to the nearest 1 mg.

#### 4.4.3 *Calculation*

The volatile matter at 150 °C, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

$m_1$ is the mass, in grams, of the weighing bottle;

$m_2$ is the mass, in grams, of the weighing bottle and test portion before heating;

$m_3$ is the mass, in grams, of the weighing bottle and test portion after heating.

### 4.5 Limit test for free alkali content

#### 4.5.1 *Reagents*

**4.5.1.1 Barium chloride,** neutral solution.

Dissolve 243 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in 1 000 ml of carbon dixoide-free water. Check that it is neutral to the phenolphthalein indicator solution (4.5.1.4) and, if not, adjust with a few drops of the sodium hydroxide solution (4.5.1.3).

**4.5.1.2 Hydrochloric acid,** 0,1 M standard volumetric solution.

**4.5.1.3 Sodium hydroxide,** 0,1 M standard volumetric solution.

**4.5.1.4 Phenolphthalein** indicator, ethanol/water solution, 5 g/l.

Dissolve 5 g of phenolphthalein in 500 ml of ethanol and add 500 ml of water, with contant stirring. Filter if necessary.

#### 4.5.2 *Apparatus*

Ordinary laboratory apparatus and

**4.5.2.1 Pipette,** 25 ml capacity, conforming to class A of ISO/R 648.

**4.5.2.2 Burette,** 50 ml capacity, conforming to class A of ISO/R 385.

#### 4.5.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample and dissolve in 30 ml of freshly boiled water in a 125 ml stoppered conical flask. Add 25 ml of the neutral barium chloride solution (4.5.1.1) and 10 drops of the phenolphthalein indicator solution (4.5.1.4) and mix well. If the solution has a pink colour, titrate with the standard volumetric hydrochloric acid solution (4.5.1.2) until the colour is discharged. (If no pink colour is produced, proceed to 4.7, using the same solution.)

### 4.6 Limit test for halides

#### 4.6.1 *Reagents*

**4.6.1.1 Nitric acid** solution, dilute (1 + 9).

**4.6.1.2 Silver nitrate** solution, 100 g/l.

**4.6.1.3 Chloride,** standard solution.

Dissolve 20 mg of potassium chloride in 1 000 ml of water.

#### 4.6.2 *Apparatus*

Ordinary laboratory apparatus and

**4.6.2.1 One-mark volumetric flask,** 100 ml capacity, conforming to class A of ISO 1042.

**4.6.2.2 Pipette,** 2 ml capacity, conforming to class A of ISO/R 648.

**4.6.2.3 Two matched Nessler cylinders,** 50 ml capacity.

#### 4.6.3 *Procedure*

Weigh, to the nearest 0,01 g, a test portion of about 1 g of the laboratory sample, dissolve in water in the one-mark volumetric flask (4.6.2.1) and dilute to the mark. Using the pipette (4.6.2.2), transfer 2 ml aliquots of this solution and of the standard chloride solution (4.6.1.3) to separate Nessler cylinders (4.6.2.3). Then add 10 ml of the nitric acid solution (4.6.1.1) and 1 ml of the silver nitrate solution (4.6.1.2) to each. Dilute to 50 ml and mix well.

Compare, in the Nessler cylinders, the turbidities produced in the test and control solutions.

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