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Photographic grade potassium hydroxide — Specification

Hydroxyde de potassium de qualité photographique — Spécifications

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3625 was drawn up by Technical Committee ISO/TC 42, *Photography*, and circulated to the Member Bodies in September 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Italy	Turkey
Austria	Japan	United Kingdom
Belgium	Mexico	U.S.A.
Bulgaria	Romania	U.S.S.R.
Canada	South Africa, Rep. of	Yugoslavia
France	Spain	
Germany	Sweden	

No Member Body expressed disapproval of the document.

Photographic grade potassium hydroxide — 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.

1 SCOPE AND FIELD OF APPLICATION

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

2 CHARACTERISTICS

Potassium hydroxide is in the form of white sticks, pellets, flakes, granules or powder, of chemical formula KOH and relative molar mass 56,11.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 85 % (*m/m*), expressed as KOH, 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 0,03 % (*m/m*), when determined by the method described in 4.3.

3.4 Halides content

The total halides content, expressed as potassium chloride (KCl), shall be not greater than 0,5 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.4, when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

3.5 Carbonate content

The carbonate content, expressed as potassium carbonate (K_2CO_3), shall be not greater than 0,3 % (*m/m*), when determined by the method described in 4.5.

3.6 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 30 mg/kg.

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

3.7 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 20 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.7, when the colour produced in the test solution shall be not greater than that produced in the control solution.

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 solutions 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 Barium chloride, neutral solution.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in 1 000 ml of carbon dioxide-free water. Check that it is neutral to the phenolphthalein indicator solution (4.1.1.4). If not, adjust with a few drops of the 0,1 M sodium hydroxide solution (4.1.1.3).

4.1.1.2 Hydrochloric acid, approximately 1 M standard volumetric solution.

4.1.1.3 Sodium hydroxide, 0,1 M standard volumetric solution.

4.1.1.4 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 constant stirring. Filter if necessary.

4.1.2 Apparatus

Ordinary laboratory apparatus and

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

4.1.2.2 Pipette, 50 ml capacity, conforming to class A of ISO/R 648.

4.1.2.3 One-mark volumetric flask, 500 ml capacity, conforming to class A of ISO 1042.

1) 1 mol/l = 1 kmol/m³ = 1 mol/dm³ = 1 M

4.1.3 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 20 g of the laboratory sample in a glass-stoppered weighing bottle. Transfer to the 500 ml one-mark volumetric flask (4.1.2.3), dissolve in carbon dioxide-free water and dilute to the mark with the same water. Using the pipette (4.1.2.2), take a 50 ml aliquot and dilute to about 200 ml with carbon dioxide-free water, retaining the balance of the solution for the halide test under 4.4.3. Add 5 ml of the barium chloride solution (4.1.1.1) to the test solution, stopper, shake and allow to stand for 5 min. Add 3 drops of the phenolphthalein indicator solution (4.1.1.4) and titrate with the hydrochloric acid solution (4.1.1.2) to the disappearance of the pink colour, retaining this solution for the determination of carbonate content under 4.5.3.

4.1.4 Calculation

The assay, expressed as a percentage by mass of potassium hydroxide (KOH), is given by the formula

$$\frac{56,11 VT}{m}$$

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

Prepare a 100 g/l solution of the sample 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, ρ approximately 1,18 g/ml.

4.3.1.2 Buffer solution, pH 9,5 to 10.

Dissolve 54 g of ammonium chloride in 200 ml of water, add 350 ml of ammonia solution, ρ approximately 0,91 g/ml, and dilute to 1 000 ml.

4.3.1.3 Magnesium, standard solution.

Dissolve 10,140 g of magnesium sulphate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in water containing 1 ml of the hydrochloric acid solution (4.3.1.1) in the 1 000 ml 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, 0,05 M standard volumetric solution.

4.3.1.5 (Ethylenedinitrilo)tetraacetic acid (EDTA), disodium salt, dihydrate, 0,01 M standard volumetric solution.

Dissolve 18,6 g of disodium 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 disodium EDTA solution with water to exactly 0,01 M.

4.3.1.6 C.I. 14645, C.I. Mordant black 11 indicator¹⁾.

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

4.3.2 Apparatus

Ordinary laboratory apparatus and

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

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

4.3.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample and dissolve in 75 ml of water.

Neutralize with the hydrochloric acid solution (4.3.1.1), using litmus paper, boil for 5 min and then 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 Mordant black 11 indicator (4.3.1.6). Titrate with the standard volumetric disodium 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 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

$$\frac{0,0243(V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of the disodium EDTA solution (4.3.1.5) used to titrate the test solution;

V_2 is the volume, in millilitres, of the disodium EDTA solution (4.3.1.5) used to titrate the blank solution;

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

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

4.4 Limit test for halides

4.4.1 Reagents

4.4.1.1 Nitric acid solution, dilute (1 + 9).

4.4.1.2 Silver nitrate solution, 100 g/l.

4.4.1.3 Chloride, standard solution.

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

4.4.2 Apparatus

Ordinary laboratory apparatus and

4.4.2.1 One-mark volumetric flask, 50 ml capacity, conforming to class A of ISO 1042.

4.4.2.2 Pipette, 20 ml capacity, conforming to class A of ISO/R 648.

4.4.2.3 Two matched Nessler cylinders, 50 ml capacity.

4.4.3 Procedure

Transfer 5 ml of the test solution, as prepared in 4.1.3, to the one-mark volumetric flask (4.4.2.1) and dilute to 50 ml. Using the pipette (4.4.2.2), transfer 20 ml aliquots of this solution and of the standard chloride solution (4.4.1.3) to separate Nessler cylinders (4.4.2.3). Then add to each solution 5 ml of water, 5 ml of the nitric acid solution (4.4.1.1) and 2 ml of the silver nitrate solution (4.4.1.2). Dilute to 50 ml and mix well.

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

4.5 Determination of carbonate content

4.5.1 Reagents

4.5.1.1 Hydrochloric acid, 0,1 M standard volumetric solution.

4.5.1.2 Methyl orange indicator solution, 0,4 g/l.

4.5.2 Apparatus

Ordinary laboratory apparatus and

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

4.5.3 Procedure

Take the solution from the end of the assay titration (4.1.3) add 2 drops of the methyl orange indicator solution (4.5.1.2) and continue the titration with the hydrochloric acid solution (4.5.1.1) until the indicator changes colour.

4.5.4 Calculation

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

$$\frac{69,11 VT}{m}$$

where

V is the volume, in millilitres, of the hydrochloric acid solution (4.5.1.1) used for the titration;

T is the exact molarity of the hydrochloric acid solution (4.5.1.1);

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

4.6 Limit test for heavy metals

4.6.1 Reagents

4.6.1.1 Hydrochloric acid solution, ρ approximately 1,18 g/ml.

4.6.1.2 Hydrochloric acid solution, dilute (1 + 99).

4.6.1.3 Ammonia solution, dilute (1 + 9).

4.6.1.4 Heavy metals, standard solution.

Dissolve a soluble lead salt in water to give a solution containing 10 mg of lead per 1 000 ml.

4.6.1.5 Water, saturated at room temperature with hydrogen sulphide.

4.6.1.6 *p*-Nitrophenol indicator solution, 2,5 g/l.

4.6.2 Apparatus

Ordinary laboratory apparatus and

4.6.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.6.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample and dissolve in 15 ml of water. Also take 15 ml of the standard heavy metals solution (4.6.1.4) and treat this and the test solution in the following manner. Add 10 ml of the hydrochloric acid solution (4.6.1.1) and evaporate to dryness on a steam bath. Take up the residue in 5 ml of the hydrochloric acid solution (4.6.1.2) and then add 25 ml of water.

To each, add 1 drop of the *p*-nitrophenol indicator solution (4.6.1.6) followed by the ammonia solution (4.6.1.3), drop by drop, until the solutions turn yellow. Add the hydrochloric acid solution (4.6.1.2), drop by drop, until the solutions become colourless and add 0,5 ml in excess. Dilute each to 50 ml with water.

Transfer 20 ml aliquots of each solution to separate Nessler cylinders (4.6.2.1), retaining the balance of the test solution for the iron test under 4.7.3. Finally, add 5 ml of the hydrogen sulphide water (4.6.1.5), dilute to 50 ml and mix well.

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

4.7 Limit test for iron

4.7.1 Reagents

4.7.1.1 Acetate buffer solution, pH 5,0.

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2 M acetic acid and dilute to 1 000 ml with water. Adjust the final pH of the solution to $5,0 \pm 0,1$ with glacial acetic acid or 100 g/l sodium hydroxide solution.

4.7.1.2 Iron, standard solution.

Dissolve a soluble iron(III) salt in water to give a solution containing 10 mg of iron(III) per 1 000 ml.

4.7.1.3 1,10-Phenanthroline reagent solution.

Thoroughly mix equal volumes of a 1 g/l aqueous solution of 1,10-phenanthroline, a 100 g/l aqueous solution of hydroxylammonium chloride and the acetate buffer solution (4.7.1.1).

4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.7.3 Procedure

Take 10 ml of the standard iron solution (4.7.1.2) and treat in the same manner as the 15 ml of the standard heavy metals solution (4.6.1.4) under 4.6.3, as far as the dilution to 50 ml.

Transfer a 20 ml aliquot of this treated standard iron solution to one of the Nessler cylinders (4.7.2.1) and 20 ml of the balance of the treated test solution from 4.6.3 to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.7.1.3) to each, mix and allow to stand for 10 min. Dilute each to 50 ml and mix well.

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

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