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Photographic grade potassium bromide — Specification

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 42 has reviewed ISO Recommendation R 420 and found it technically suitable for transformation. International Standard ISO 420 therefore replaces ISO Recommendation R 420-1965.

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ISO Recommendation R 420 was approved by the Member Bodies of the following countries :

Belgium	Italy	Switzerland
Brazil	Japan	United Kingdom
Canada	Netherlands	U.S.A.
Chile	New Zealand	U.S.S.R.
France	Romania	
Germany	Sweden	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 420 into an International Standard.

Photographic grade potassium bromide – 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 bromide.

2 CHARACTERISTICS

Potassium bromide is in the form of white crystals or crystalline powder, of chemical formula KBr and relative molar mass 119,0.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 99,0 % (*m/m*) nor more than 100,3 % (*m/m*), expressed as KBr, 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, magnesium and other matter insoluble in ammonia solution

The matter insoluble in ammonia solution shall be not greater than 0,3 % (*m/m*), when determined by the method given in 4.3.

3.4 Volatile matter at 105 °C

The volatile matter at 105 °C shall be not greater than 0,3 % (*m/m*), when determined by the method described in 4.4.

3.5 Alkalinity

The alkalinity, expressed as potassium hydroxide (KOH), shall be not greater than 0,015 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.5.

3.6 Acidity

The acidity, expressed as hydrogen bromide (HBr), shall be not greater than 0,010 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.6.

3.7 Chloride content

The chloride 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 4.7, when the turbidity produced

in the test solution shall be not greater than that produced in the control solution.

3.8 Iodide content

The iodide content shall be not greater than that permitted by the limit test described in 4.8, when the carbon tetrachloride or chloroform layer shall not acquire a violet tint.

3.9 Sulphide content

The sulphide content, expressed as potassium sulphide (K₂S), shall be not greater than 8 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 Oxidizing substances content

The oxidizing substances content shall be not greater than that permitted by the limit test described in 4.10, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.11 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 20 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.11, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.12 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 4.12, 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 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.

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

4.1 Assay

4.1.1 Reagents

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

4.1.1.2 Ammonium iron(III) sulphate solution, 50 g/l.

4.1.1.3 Silver nitrate, 0,1 M standard volumetric solution.

4.1.1.4 Ammonium thiocyanate, 0,1 M standard volumetric solution.

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.

4.1.3 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 0,4 g of the laboratory sample and dissolve in 50 ml of water. Add 5 ml of the nitric acid solution (4.1.1.1), followed by 50,00 ml of the standard volumetric silver nitrate solution (4.1.1.3), using the pipette (4.1.2.2). Shake well, add 2 ml of the ammonium iron(III) sulphate solution (4.1.1.2) and titrate the excess of silver nitrate with the standard volumetric ammonium thiocyanate solution (4.1.1.4) to the first persistent colour change.

4.1.4 Calculation

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

$$\frac{11,90 (50 T_1 - V T_2)}{m}$$

where

V is the volume, in millilitres, of the ammonium thiocyanate solution (4.1.1.4) used for the titration;

T_1 is the exact molarity of the silver nitrate solution (4.1.1.3);

T_2 is the exact molarity of the ammonium thiocyanate solution (4.1.1.4);

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

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

4.2 Appearance of solution test

Prepare a 200 g/l solution of the laboratory sample in water and examine for clarity and sediment.

4.3 Determination of calcium, magnesium and other matter insoluble in ammonia solution

4.3.1 Reagents

4.3.1.1 Ammonium oxalate solution, 40 g/l.

4.3.1.2 Diammonium hydrogen orthophosphate solution, 100 g/l.

4.3.1.3 Ammonia solution, dilute (1 + 9).

4.3.1.4 Ammonia solution, dilute (1 + 39).

4.3.2 Apparatus

Ordinary laboratory apparatus and

4.3.2.1 Crucible.

4.3.2.2 Muffle furnace, capable of being controlled at 600 ± 50 °C.

4.3.3 Procedure

Weigh, to the nearest 0,5 g, a test portion of about 10 g of the laboratory sample and dissolve in 75 ml of water. Add 5 ml of the ammonium oxalate solution (4.3.1.1) and 2 ml of the diammonium hydrogen orthophosphate solution (4.3.1.2), followed by 10 ml of the ammonia solution (4.3.1.3). Allow to stand overnight. If any precipitate is formed, filter through an ashless filter paper and wash the precipitate with the ammonia solution (4.3.1.4). Transfer the filter paper with the precipitate to the previously weighed crucible (4.3.2.1), dry and ignite the residue in the furnace (4.3.2.2), controlled at 600 ± 50 °C, for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

4.3.4 Calculation

The matter insoluble in ammonia solution is given, as a percentage by mass, by the formula

$$\frac{100 (m_2 - m_1)}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the crucible;

m_2 is the mass, in grams, of the crucible plus residue.

4.4 Determination of volatile matter at 105 °C

4.4.1 Apparatus

Ordinary laboratory apparatus and

4.4.1.1 Oven, capable of being controlled at 105 ± 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 105 ± 5 °C, for 4 h. Cool in a desiccator, replace the stopper and weigh to the nearest 1 mg.

4.4.3 Calculation

The volatile matter at 105 °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 alkalinity

4.5.1 Reagents

4.5.1.1 Hydrochloric acid, 0,01 M standard volumetric solution.

4.5.1.2 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.

4.5.2 Apparatus

Ordinary laboratory apparatus and

4.5.2.1 Burette, 10 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 4 g of the laboratory sample and dissolve in 60 ml of freshly boiled water in a 125 ml stoppered conical flask. Add 3 drops of the phenolphthalein indicator solution (4.5.1.2) and mix well. If the solution has a pink colour, titrate with the standard volumetric hydrochloric acid solution (4.5.1.1) until the colour is discharged. (If no pink colour is produced, proceed to 4.6, using the same solution.)

4.5.4 Calculation

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

$$\frac{5,6 V T}{m}$$

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 acidity

4.6.1 Reagent

4.6.1.1 Sodium hydroxide, 0,01 M standard volumetric solution.

4.6.2 Procedure

If the solution prepared under 4.5.3 is not pink, titrate with the standard volumetric sodium hydroxide solution (4.6.1.1) until a faint pink colour, which persists for 30 s, is obtained.

4.6.3 Calculation

The acidity, expressed as a percentage by mass of hydrogen bromide (HBr), is given by the formula

$$\frac{8,1 V T}{m}$$

4.6.3.1 where

V is the volume, in millilitres, of the sodium hydroxide solution (4.6.1.1) used for the titration;

T is the exact molarity of the sodium hydroxide solution (4.6.1.1);

m is the mass, in grams, of the test portion taken under 4.5.3.

4.7 Limit test for chloride

4.7.1 Reagents

4.7.1.1 Nitric acid solution, dilute (1 + 2).

4.7.1.2 Hydrogen peroxide solution, approximately 167 g/l.

Dilute "30 % hydrogen peroxide" solution (1 + 1).

4.7.1.3 Chloride, standard solution.

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

4.7.1.4 Silver nitrate solution, 100 g/l.

4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

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4.7.3 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 1 g of the laboratory sample and dissolve in 15 ml of the nitric acid solution (4.7.1.1) in a conical flask. Add 6 ml of the hydrogen peroxide solution (4.7.1.2) 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, cool, filter and dilute to 250 ml with water.

Transfer 10 ml aliquots of this test solution and of the standard chloride solution (4.7.1.3) to separate Nessler cylinders (4.7.2.1) and treat each solution as follows. Add 1 ml of the nitric acid solution (4.7.1.1), dilute to 50 ml and then add 0,5 ml of the silver nitrate solution (4.7.1.4) and mix well.

Compare, in the Nessler cylinders, the turbidities produced after 5 min in the test and control solutions.

4.8 Limit test for iodide

4.8.1 Reagents

4.8.1.1 Carbon tetrachloride (CCl_4) or **chloroform** (CHCl_3).

4.8.1.2 Sulphuric acid solution, dilute (1 + 9).

4.8.1.3 Iron(III) chloride solution, 100 g/l.

Dissolve 100 g of iron(III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 800 ml of water and dilute to 1 000 ml.

4.8.2 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g of the laboratory sample and dissolve in 25 ml of water. Add 1 ml of the sulphuric acid solution (4.8.1.2), 0,5 ml of the iron(III) chloride solution (4.8.1.3) and 1 ml of the carbon tetrachloride or chloroform (4.8.1.1). Shake the mixture vigorously and allow the layers to separate. Observe any colour in the carbon tetrachloride or chloroform layer.

4.9 Limit test for sulphide

4.9.1 Reagents

4.9.1.1 Lead, alkaline solution.

Prepare a 100 g/l solution of lead acetate trihydrate [$\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$] and add sufficient 100 g/l sodium hydroxide solution to dissolve the precipitate formed and to provide a slight excess of sodium hydroxide.

4.9.1.2 Sulphide, standard solution.

Immediately before use, dissolve 6,1 g of sodium sulphide nonahydrate ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) in 1 000 ml of boiled and cooled water. Dilute 1 ml of this solution (1 + 999) with boiled and cooled water.

1 ml of this solution is equivalent to 0,002 8 mg of potassium sulphide (K_2S).

NOTE — Some workers have found alkaline earth sulphide solutions more stable than sodium sulphide solutions.

4.9.2 Apparatus

Ordinary laboratory apparatus and

4.9.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.9.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 3,5 g of the laboratory sample and dissolve in 10 ml of water. Treat this and 10 ml of the freshly prepared standard sulphide solution (4.9.1.2) in separate Nessler cylinders (4.9.2.1) in the following manner. Add 0,5 ml of the alkaline lead solution (4.9.1.1) to each, dilute to 50 ml and mix well.

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

4.10 Limit test for oxidizing substances

4.10.1 Reagents

4.10.1.1 Carbon tetrachloride (CCl_4) or **chloroform** (CHCl_3).

4.10.1.2 Potassium iodide (KI).

4.10.1.3 Sulphuric acid solution, dilute (1 + 9).

4.10.2 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g of the laboratory sample and dissolve in 10 ml of water in a test tube. Treat this solution and 10 ml of water in a second test tube in the following manner. Add 0,5 g of the potassium iodide (4.10.1.2), 1 ml of the sulphuric acid solution (4.10.1.3) and 1 ml of the carbon tetrachloride or chloroform (4.10.1.1). Shake the mixture vigorously and allow the layers to separate.

Compare, in the test tubes, the colours produced in the carbon tetrachloride or chloroform layer of the test and control solutions.

4.11 Limit test for heavy metals

4.11.1 Reagents

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

4.11.1.2 Ammonia solution, dilute (1 + 9).

4.11.1.3 Heavy metals, standard solution.

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

4.11.1.4 Water, saturated at room temperature with hydrogen sulphide.

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

4.11.2 Apparatus

Ordinary laboratory apparatus and

4.11.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.11.2.2 Procedure

Weigh, to the nearest 0,05 g, a test portion of about 2 g of the laboratory sample and dissolve in 20 ml of water in one of the Nessler cylinders (4.11.2.1). Also transfer 4 ml of the standard heavy metals solution (4.11.1.3) to the other Nessler cylinder. To each, add 1 drop of the *p*-nitrophenol indicator solution (4.11.1.5) followed by the ammonia solution (4.11.1.2), drop by drop, until the solutions turn yellow. Add the hydrochloric acid solution (4.11.1.1), drop by drop, until the solutions become colourless and then add 0,5 ml in excess. Finally add 5 ml of the hydrogen sulphide water (4.11.1.4), dilute to 50 ml and mix well.

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

4.12 Limit test for iron

4.12.1 Reagents

4.12.1.1 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 to $5,0 \pm 0,1$ with glacial acetic acid or 100 g/l sodium hydroxide solution.

4.12.1.2 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.

4.12.1.3 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 (4.12.1.1).

4.12.2 Apparatus

Ordinary laboratory apparatus and

4.12.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.12.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample and dissolve in 20 ml of water in one of the Nessler cylinders (4.12.2.1). Transfer 4 ml of the standard iron solution (4.12.1.2) to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.12.1.3) 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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