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Photographic grade potassium metabisulphite — Specification

Métadisulfite de potassium de qualité photographique — Spécifications

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FOREWORD

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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 3629 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.

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Photographic grade potassium metabisulphite — 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 metabisulphite.

2 CHARACTERISTICS

Potassium metabisulphite is in the form of white, glassy crystals, of chemical formula $K_2S_2O_5$ and relative molar mass 222,3.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 95,0 % (*m/m*), expressed as $K_2S_2O_5$, 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 fluocculence, when examined by the method described in 4.2.

3.3 pH value

The pH of a 50 g/l solution, at 20 °C, shall be between 4,0 and 4,6 when determined by the method described in 4.3.

3.4 Thiosulphate content

The thiosulphate content, expressed as potassium thiosulphate ($K_2S_2O_3$), shall be not greater than 0,07 % (*m/m*).

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

3.5 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 50 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.5, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.6 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 50 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 that produced in the control solution.

3.7 Reaction to ammoniacal silver nitrate solution

The colour or turbidity 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 4.7.

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 Acetic acid, solution approximately 2 M.

4.1.1.2 Formaldehyde, neutralized to phenolphthalein, approximately 360 g/l solution.

4.1.1.3 Iodine, 0,05 M standard volumetric solution, 12,7 g of iodine per litre.

4.1.1.4 Sodium thiosulphate, 0,1 M standard volumetric solution.

4.1.1.5 Sulphuric acid, 0,05 M standard volumetric solution.

4.1.1.6 Starch indicator solution, 5 g/l.

Stir 5 g of soluble starch with 100 ml of a 10 g/l salicylic acid solution. Then add 300 to 400 ml of boiling water, boil until the starch dissolves then finally dilute to 1 000 ml with water.

4.1.1.7 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.

1) 1 mol/l = 1 kmol/m^3 = 1 mol/dm^3 = 1 M

4.1.3 *Procedure*

Using a pipette (4.1.2.2), deliver 50,00 ml of the standard iodine solution (4.1.1.3) into a flask. Weigh, to the nearest 0,000 1 g, a test portion of about 0,23 g of the laboratory sample and wash this into the flask. Add 5 ml of the acetic acid solution (4.1.1.1) and titrate with the standard sodium thiosulphate solution (4.1.1.4), adding the starch indicator solution (4.1.1.6) just before the end-point.

Weigh, to the nearest 0,001 g, a further test portion of about 5 g of the laboratory sample, dissolve in 50 ml of water and add 50 ml of the formaldehyde solution (4.1.1.2). Add a few drops of the phenolphthalein indicator solution (4.1.1.7) and titrate with the standard sulphuric acid solution (4.1.1.5) to the colour change.

4.1.4 *Calculation*

The assay, expressed as a percentage by mass of potassium metabisulphite ($K_2S_2O_5$), is given by the formula

$$\frac{5,56}{m_1}(100\,T_1 - V_2T_2) - \frac{11,1}{m_2}\,V_3T_3$$

where

T_1 is the exact molarity of the iodine solution (4.1.1.3);

T_2 is the exact molarity of the sodium thiosulphate solution (4.1.1.4);

T_3 is the exact molarity of the sulphuric acid solution (4.1.1.5);

V_2 is the volume, in millilitres, of the sodium thiosulphate solution (4.1.1.4) used for the titration;

V_3 is the volume, in millilitres, of the sulphuric acid solution (4.1.1.5) used for the titration;

m_1 is the mass, in grams, of the test portion used for the first titration;

m_2 is the mass, in grams, of the test portion used for the second titration.

NOTE – When an assay (based on its sulphite content), expressed as a percentage by mass of potassium metabisulphite ($K_2S_2O_5$) is desired, the second titration of 4.1.3 is not required and is given by the formula

$$\frac{5,56}{m_1}(100\,T_1 - V_2T_2)$$

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 Measurement of the pH value

4.3.1 *Apparatus*

Electronic pH meter equipped with a glass electrode and standard reference electrode.

4.3.2 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample, dissolve in about 80 ml of freshly boiled water and dilute to 100 ml. Measure the pH of this solution at 20 °C using the pH meter in accordance with the manufacturer's instructions.

4.4 Limit test for thiosulphate

4.4.1 *Reagents*

As specified under 4.1 and

4.4.1.1 Thiosulphate, standard solution.

Dilute 5 ml of the sodium thiosulphate solution (4.1.1.4) to 1 000 ml.

4.4.1.2 Mercury(II) chloride reagent solution.

Dissolve 25 g of potassium bromide and 25 g of mercury(II) chloride in 900 ml of water at 50 °C. Cool, dilute to 1 000 ml and allow to stand overnight. Filter if not perfectly clear.

4.4.2 *Apparatus*

Ordinary laboratory apparatus and

4.4.2.1 Graduated pipette, 1 ml capacity.

4.4.2.2 Two matched Nessler cylinders, 50 ml capacity.

4.4.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 6 g of the laboratory sample, dissolve in water and dilute to 100 ml. Slowly pipette 0,5 ml of this solution into 10 ml of the mercury(II) chloride reagent solution (4.4.1.2) in one of the Nessler cylinders (4.4.2.2). To 10 ml of the mercury(II) chloride reagent (4.4.1.2) contained in the second Nessler cylinder slowly add 0,25 ml of the standard thiosulphate solution (4.4.1.1). Allow both to stand for 10 min without agitation, then carefully agitate to distribute the opalescence. Immediately examine, in the Nessler cylinders, the opalescence produced in the test and control solutions.

NOTE – If the solutions are allowed to stand for more than 15 min, secondary reactions occur which will affect the result.

4.5 Limit test for heavy metals

4.5.1 *Reagents*

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

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

4.5.1.3 Ammonia solution, dilute (1 + 9).

4.5.1.4 Heavy metals, standard solution.

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

4.5.1.5 Water, saturated at room temperature with hydrogen sulphide.

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

4.5.2 *Apparatus*

Ordinary laboratory apparatus and

4.5.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.5.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample and dissolve in 25 ml of water. Also take 10 ml of the standard heavy metals solution (4.5.1.4) and treat this and the test solution in the following manner. Add 15 ml of the hydrochloric acid solution (4.5.1.1) and evaporate to dryness on a steam-bath. Take up the residues with 25 ml of water. To each, add 2 drops of the *p*-nitrophenol indicator solution (4.5.1.6) followed by the ammonia solution (4.5.1.3), drop by drop, until the solutions turn yellow. Add the hydrochloric acid solution (4.5.1.2), drop by drop, until the solutions become colourless and add 2,5 ml in excess. Dilute each to 50 ml with water.

Treat 20 ml aliquots of each solution separately in the Nessler cylinders (4.5.2.1), retaining the balance of the test solution for the iron test under 4.6.3. Add 5 ml of the hydrogen sulphide water (4.5.1.5), dilute to 50 ml and mix well.

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

4.6 Limit test for iron

4.6.1 *Reagents*

As specified under 4.5.1 and

4.6.1.1 Acetate buffer solution, pH 5,0.

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

4.6.1.2 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.

4.6.1.3 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 (4.6.1.1).

4.6.2 *Apparatus*

Ordinary laboratory apparatus and

4.6.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.6.3 *Procedure*

Take 10 ml of the standard iron solution (4.6.1.2) and treat in the same manner as 10 ml of the standard heavy metals solution (4.5.1.4) under 4.5.3, 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 (4.6.2.1) and 20 ml of the balance of the treated test solution from 4.5.3 to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.6.1.3) 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.

4.7 Reaction to ammoniacal silver nitrate test

4.7.1 *Reagent*

4.7.1.1 Silver nitrate, ammoniacal solution.

Immediately before use, mix equal volumes of ammonia solution, ρ approximately 0,910 g/ml, and 100 g/l aqueous silver nitrate solution.

4.7.2 *Apparatus*

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.7.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample and dissolve in 40 ml of water. Divide this volume equally between the two Nessler cylinders (4.7.2.1). To one, the test solution, add 10 ml of the freshly prepared ammoniacal silver nitrate solution (4.7.1.1) and mix well. To the other, the control solution, add 5 ml of ammonia solution, ρ approximately 0,910 g/ml, and 5 ml of water and mix well. Allow each to stand for 2 min.

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

CAUTION : Dispose of all test solutions and rinse apparatus used immediately. Explosive compounds may be formed on standing.