International Standard 5990

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Photographic grade potassium sulfite, 650 g/l aqueous solution — Specification

Sulfite de potassium en solution aqueuse à 650 g/l 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.

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Photographic grade potassium sulfite, 650 g/l aqueous solution — 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 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 up 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 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.

References ISO 835

1 Scope and field of application

This International Standard specifies the purity requirements of, and test methods for, photographic grade potassium sulfite, 650 g/l aqueous solution.

2 Characteristics

Potassium sulfite, 650 g/l aqueous solution, is in the form of a clear, colourless or almost colourless liquid. The chemical formula is K_2SO_3 and relative molar mass 158,3.

3 Requirements

3.1 Assay

The assay shall be not less than 44,5 % (*m*/*m*) and not more than 46,0 % (*m*/*m*), expressed as K_2SO_3, when determined by the method described in 4.1.

3.2 Density

The density shall be not less than 1,445 g/ml and not more than 1,460 g/ml at 20 °C when determined by the method described in 4.2.

3.3 Appearance of solution

The aqueous solution shall be clear and free from sediment, other than a slight flocculence, when examined by the method described in 4.3. The solution shall be odourless.

3.4 pH value

The pH of a (1 + 9) aqueous dilution shall be between 8,0 and 10,0 at 20 °C when determined by the method described in 4.4.

3.5 Thiosulfate content

The thiosulfate content, expressed as potassium thiosulfate ($K_2S_2O_3$), shall not be greater than 0,01 % (*m*/*m*).

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

3.6 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 4.6, 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.

3.8 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 4.8.

4 Test methods

Reagents shall be handled in conformity to health and safety precautions as shown on containers or as given in other sources of such information. Proper labelling of prepared reagents includes adequate health and safety precautions. The discharge of reagents shall conform to applicable environmental regulations.

Reagents used in the tests shall be certified reagent grade chemicals or chemicals of a purity acceptable for the analysis. 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 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 Iodine, $c(I_2) = 0{,}05$ mol/l standard volumetric solution, 12,7 g of iodine per litre.

4.1.1.2 Sodium thiosulfate, $c(Na_2S_2O_3) = 0{,}1$ mol/l standard volumetric solution.

4.1.1.3 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 and finally dilute to 1 000 ml with water.

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[1] $1 \text{ mol/l} = 1 \text{ kmol/m}^3 = 1 \text{ mol/dm}^3$.

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, 10 ml capacity, conforming to class A of ISO 648.

4.1.2.3 Pipette, 25 ml capacity, conforming to class A of ISO 648.

4.1.2.4 Volumetric flask, 250 ml capacity, conforming to class A of ISO 1042.

4.1.3 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 7 g of the sample, transfer to the 250 ml volumetric flask (4.1.2.4), and make up to the mark with water, mixing well. Using the pipette (4.1.2.3) deliver 25 ml of the standard volumetric iodine solution (4.1.1.1) into a flask, and then add 10 ml of the diluted sample to this, using the pipette (4.1.2.2). Titrate the excess iodine with the standard volumetric sodium thiosulfate solution (4.1.1.2) using the burette (4.1.2.1). Add a few drops of the starch indicator (4.1.1.3) just before the end-point.

4.1.4 Calculation

The assay, expressed as a percentage by mass of potassium sulfite (K_2SO_3), is given by the formula

$$\frac{3{,}956\ V_1\ c(I_2) - 1{,}978\ V_2 c(Na_2S_2O_3)}{m} \times 100$$

where

$c(I_2)$ is the exact concentration of the iodine solution (4.1.1.1);

$c(Na_2S_2O_3)$ is the exact concentration of the sodium thiosulfate solution (4.1.1.2);

V_1 is the volume, in millilitres, of the iodine solution added (i.e. 25 ml);

V_2 is the volume, in millilitres, of the sodium thiosulfate solution (4.1.1.2) used for the titration;

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

$3{,}956\ V_1 c(I_2)$ is the mass, in grams, of K_2SO_3 which is equivalent to I_2 standard volumetric solution added. This is calculated by (25 times molar mass of K_2SO_3 equivalent to 1 ml of 1 mol/l I_2 solution) × (volume, in millilitres, of the I_2 solution added) × (exact concentration of the I_2 solution);

$1{,}978\ V_2 c(Na_2S_2O_3)$ is the mass, in grams, of K_2SO_3 which is equivalent to $Na_2S_2O_3$ standard volumetric solution used for the titration of excess I_2. This is calculated by (25 times molar mass of K_2SO_3 equivalent to 1 ml of 1 mol/l $Na_2S_2O_3$ solution) × (volume, in millilitres, of the $Na_2S_2O_3$ solution used for the titration of excess I_2) × (exact concentration of the $Na_2S_2O_3$ solution).

In these calculations, 25 equals the dilution, that is, sampling volume (10 ml) divided by made-up volume (250 ml).

4.2 Determination of density

4.2.1 Apparatus

Ordinary laboratory apparatus and

4.2.1.1 Hydrometer 20/20, including the range 1,4 to 1,5 g/ml.

4.2.2 Procedure

Float the hydrometer (4.2.1.1) in an adequate volume of original sample at 20 °C and read the value shown.

4.3 Appearance of solution test

Dilute 100 ml of sample with 400 ml of water and examine for clarity, sediment and smell.

4.4 Determination of the pH value

4.4.1 Apparatus

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

4.4.2 Procedure

Measure, to the nearest 0,5 ml, 10 ml of the sample, add 90 ml of water and mix well. Measure the pH of the solution at 20 °C, using the pH meter in accordance with the manufacturer's instructions.

4.5 Limit test for thiosulfate

4.5.1 Reagents

4.5.1.1 Thiosulfate, standard solution.

Dilute 5 ml of the sodium thiosulfate solution (4.1.1.2) to 1 000 ml.

4.5.1.2 Mercury(II) chloride reagent.

Dissolve 25 g of potassium bromide and 25 g of mercury(II) chloride in 600 ml of water at 50 °C. Cool, add 200 ml of glacial acetic acid, dilute to 1 000 ml and allow to stand overnight. Filter if not perfectly clear.

4.5.2 Apparatus

Ordinary laboratory apparatus and

4.5.2.1 Graduated pipette, 1 ml capacity, conforming to class A of ISO ~~385~~. 835 (851 [illegible])

4.5.2.2 Two matched Nessler cylinders, 50 ml capacity.

4.5.3 Procedure

Dilute 10 ml of the sample, measured to the nearest 0,5 ml, with 90 ml of water or use the solution prepared for pH measurement (4.4). Slowly pipette 1,4 ml of this solution into 10 ml of the mercury(II) chloride reagent solution (4.5.1.2) in one of the Nessler cylinders (4.5.2.2). To 10 ml of the mercury(II) chloride reagent solution (4.5.1.2) contained in the second Nessler cylinder slowly add 0,25 ml of the standard thiosulfate solution (4.5.1.1), and 1,0 ml of water. Swirl to mix thoroughly and allow to stand for 10 min. At the end of this time, swirl again to distribute the opalescence. Without proper mixing, a repeatable turbidity may not be obtained.

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.

4.6 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 sulfide.

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, 5 g of the sample and dilute to 15 ml with water. Also take 10 ml of the standard heavy metals solution (4.1.6.4) and treat this and the test solution in the following manner. Add 15 ml of the hydrochloric acid solution (4.6.1.1) 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 (4.6.1.6) followed by the ammonia solution (4.6.1.3), drop by drop, until the solutions turn yellow. Add the dilute hydrochloric acid solution (4.6.1.2), drop by drop, until the solutions become colourless and add 2,5 ml in excess. Dilute each to 100 ml with water.

Treat 30 ml aliquots of each solution separately in the Nessler cylinders (4.6.2.1), retaining the balance of the test solution for the iron test under 4.7.3. Add 5 ml of the hydrogen sulfide 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

As specified under 4.6.1 and

4.7.1.1 Acetate buffer solution, pH 5,0.

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 120 g/l acetic acid solution and dilute to 1 000 ml. Adjust the final pH to 5,0 ± 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 **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 10 ml of the standard heavy metals solution (4.6.1.4) under 4.6.3, as far as the dilution to 100 ml.

Transfer a 10 ml aliquot of this treated standard iron solution to one of the Nessler cylinder (4.7.2.1) and 10 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.