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Photographic grade sodium thiosulphate, anhydrous — Specification

Thiosulfate de sodium anhydre 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 3300 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	Japan	Turkey
Austria	Mexico	United Kingdom
Belgium	Netherlands	U.S.A.
Canada	Poland	U.S.S.R.
France	Romania	Yugoslavia
Germany	South Africa, Rep. of	
Italy	Spain	

No Member Body expressed disapproval of the document.

Photographic grade sodium thiosulphate, 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.

1 SCOPE AND FIELD OF APPLICATION

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

2 CHARACTERISTICS

Sodium thiosulphate, anhydrous, is in the form of a white powder, of chemical formula $\text{Na}_2\text{S}_2\text{O}_3$ and relative molar mass 158,1.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 97,0 % (m/m), expressed as $\text{Na}_2\text{S}_2\text{O}_3$, 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, magnesium and other matter insoluble in ammonia solution

The matter insoluble in ammonia solution shall be not greater than 0,4 % (m/m), when determined by the method given in 4.3.

3.4 pH value

The pH of a 100 g/l aqueous solution when prepared and measured by the method described in 4.4 shall be between 6,5 and 9,5 at 20 °C.

3.5 Sulphide content

The sulphide content, expressed as Na_2S , shall be not greater than 20 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 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 50 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 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 Iodine, 0,15 M standard volumetric solution, 12,7 g of iodine per litre.

4.1.1.2 Starch indicator solution.

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 and boil until the starch dissolves, finally diluting to 1 000 ml with water.

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 0,6 g of the laboratory sample, transfer to a conical flask and dissolve in about 50 ml of water. Titrate with the standard volumetric iodine solution (4.1.1.1) from the burette (4.1.2.1), using the starch indicator solution (4.1.1.2), until the blue colour produced at the end-point remains for at least 1 min.

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

4.1.4 Calculation

The assay, expressed as a percentage by mass of sodium thiosulphate, anhydrous, is given by the formula

$$\frac{31,6 VT}{m}$$

where

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

T is the exact molarity of the iodine solution (4.1.1.1);

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

4.2 Appearance of test solution

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 5 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

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 Measurement of pH value

4.4.1 Apparatus

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

4.4.2 Procedure

Weigh, to the nearest 0,1 g, about 10 g of the laboratory sample, dissolve in about 80 ml of previously boiled water having a pH no lower than 6,5, and make up to 100 ml. Measure the pH of this solution at 20 °C using the pH meter in accordance with the manufacturer's instructions.

4.5 Limit test for sulphide

4.5.1 Reagents

4.5.1.1 Lead, alkaline solution.

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

4.5.1.2 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 mg of sodium sulphide (Na_2S).

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

4.5.2 Apparatus

Ordinary laboratory apparatus and

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

4.5.2.2 Two matched Nessler cylinders, 50 ml capacity.

4.5.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of 1 g of the laboratory sample and dissolve in 10 ml of water. Treat this and 10 ml of the freshly prepared standard sulphide solution (4.5.1.2) in separate Nessler cylinders (4.5.2.2) in the following manner. Add 0,5 ml of the alkaline lead solution (4.5.1.1) to each, dilute to 50 ml and mix well.

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

4.6 Limit test for heavy metals

4.6.1 Reagents

4.6.1.1 Hydrochloric acid solution, dilute (1 + 1).

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

4.6.1.3 Ammonia solution, dilute (1 + 2).

4.6.1.4 Hydrogen peroxide solution, approximately 1,67 g/l.

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

4.6.1.5 Heavy metals, standard solution.

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

4.6.1.6 Water, saturated at room temperature with hydrogen sulphide.

4.6.1.7 *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 0,05 g, a test portion of 2,0 g of the laboratory sample and dissolve in 20 ml of water. Also take 4 ml of the standard heavy metals solution (4.6.1.5) and treat this and the test solution in the following manner. Add 5 ml of the ammonia solution (4.6.1.3) and then add slowly, with great care, in several portions, 40 ml of the hydrogen peroxide solution (4.6.1.4). Allow to stand for 10 min. Evaporate to dryness on a steam bath, and then add 5 ml of the hydrochloric acid solution (4.6.1.1) and 5 ml of water. Heat to boiling and dilute each to 50 ml with water.

To each, add 2 drops of *p*-nitrophenol indicator solution (4.6.1.7) followed by the ammonia solution (4.6.1.3), drop by drop, until the solutions turn yellow. Then add the 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.

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 (4.6.1.6), 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

Reagents as specified in 4.6.1 and

4.7.1.1 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.

4.7.1.2 Potassium thiocyanate, butanolic solution.

Dissolve 10 g of potassium thiocyanate in 10 ml of water and then add, with vigorous shaking, 90 ml of butan-1-ol.

4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 Two glass-stoppered test tubes, 50 ml capacity.

4.7.3 Procedure

Take 10 ml of the standard iron solution (4.7.1.1) and treat in the same manner as the 4 ml of the standard heavy metals solution (4.6.1.5) under 4.6.3 as far as the boiling and dilution to 100 ml.

Transfer 5 ml of this treated standard iron solution to one of the glass-stoppered test tubes (4.7.2.1) and 5 ml of the balance of the treated test solution from 4.6.3 to the other test tube. Add 15 ml of the butanolic potassium thiocyanate solution (4.7.1.2) to each and shake for 1 min.

Compare, in the test tubes, the colours produced in the test and control solutions.