INTERNATIONAL STANDARD 419

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Photographic grade sodium thiosulphate, crystalline – Specification

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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 419 was drawn up by Technical Committee ISO/TC 42, *Photography.* It was submitted directly to the ISO Council, in accordance with clause F.7.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 419-1965, which was approved in Jaunuary 1962 by the Member Bodies of the following countries :

Belgium
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No Member Body expressed disapproval of the document.

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Photographic grade sodium thiosulphate, crystalline – Specification

0 INTRODUCTION

This document is one of a number of ISO publications giving 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.

A photographic grade chemical is one which meets the requirements specified in the applicable ISO publication. These specifications set forth 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.

These specifications set forth 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 those objectives listed herein. If the purity of a commonly available grade of chemical exceeds photographic processing requirements, however, and if there is no economic penalty in its use, the purity requirements have been set to take advantage of the higher quality material.

Every effort has been made to keep the number of requirements in this specification to a minimum. The requirements generally include only those photographically harmful impurities which, through experience, are known likely to be present. Inert impurities are limited to amounts which will not unduly reduce the assay.

An assay procedure has been included. An effective assay requirement serves not only as a safeguard of chemical purity but is also a valuable complement to the identity test. The assay is 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 under the summary of requirements in this specification are binding. The physical appearance of the material and any footnotes are for general information only and are not a part of the requirements.

Efforts have been made to employ tests which are capable of being run in any normally equipped laboratory and to avoid tests wherever possible which require highly specialized equipment or techniques. Instrumental methods have been specified only as alternative methods or alone in those cases where no other method is available.

While the test methods set forth in this specification 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 employed.

An effort has been made to minimize the number of reagents employed in this series of specifications. The methods of preparation and 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 in order that each specification be self-sufficient.

1 SCOPE AND FIELD OF APPLICATION

This International Standard gives a specification for photographic grade crystalline sodium thiosulphate. It is one of a series to establish criteria of purity of chemicals suitable for processing photographic materials. A "photographic grade" chemical is one which meets purity requirements as described. This specification states the limiting concentrations and the test methods for certain inert or photographically harmful impurities that may be present.

2 PHYSICAL APPEARANCE

Crystalline sodium thiosulphate (known as crystal Hypo) ($Na_2S_2O_3 . 5H_2O$) is in the form of colourless crystals.

3 SUMMARY OF REQUIREMENTS

Assay (as $Na_2S_2O_3 . 5H_2O$) : 99 % minimum.

pH value (20 % solution) : 6.5 to 9.5.

Sulphide (as Na_2S) : 0.001 % maximum.

Insoluble matter, calcium, magnesium and ammonium hydroxide precipitate : 0.2 % maximum residue.

Heavy metals (as Pb) : 0.001 % maximum.

Iron (Fe) : 0.003 % maximum.

Appearance of solution : to pass test.

4 ASSAY (as $Na_2S_2O_3 . 5H_2O$) *(99.0 % minimum)*

4.1 Reagents

4.1.1 *Standard iodine solution*

Weigh accurately about 12.75 g of freshly resublimed iodine[1] into a tared weighing flask. Add 36 g of potassium iodide and 100 ml of distilled water. After solution is complete, dilute to exactly 1 l at 20 °C in a volumetric flask. From the mass of the iodine, calculate the normality.

4.1.2 *Starch indicator*

Stir 5 g of soluble starch with 100 ml of 1 % salicylic acid solution. Add 300 to 400 ml of boiling distilled water and boil until the starch dissolves. Dilute to 1 l.

4.2 Procedure

Take about 1 g of the sample, weigh accurately and dissolve in 50 ml of distilled water. Titrate with the standard iodine solution, using the starch indicator.

$$\% \ Na_2S_2O_3.5H_2O = \frac{24.8\,NV}{m}$$

where

N is the normality of the iodine solution;

V is the volume of the iodine solution, in millilitres;

m is the mass of sample taken, in grams.

5 pH VALUE *(6.5 to 9.5)*

Dissolve 20.0 ± 0.1 g of the sample in about 80 ml of freshly boiled and cooled distilled water, and dilute to 100 ml. Determine the pH of the solution at 20 °C using a pH meter equiped with a glass electrode and a suitable reference electrode.

6 SULPHIDE (as Na_2S) *(0.001 % maximum)*

Dissolve 2 ± 0.1 g of the sample in 10 ml of distilled water and add 0.5 ml of alkaline lead solution (prepared by adding sufficient 10 % sodium hydroxide solution to a 10 % lead acetate [$Pb(C_2H_3O_2)_2 . 3H_2O$] solution to dissolve the precipitate and provide a slight excess of sodium hydroxide). Dilute to 50 ml and mix well. Any dark colour produced should be not greater than is produced by 0.02 mg of sodium sulphide (Na_2S) or an equi-molecular mass of any other water-soluble sulphide treated in the same manner. The test control sulphide solution should be freshly prepared using boiled and cooled distilled water. Use Nessler tubes for comparison.

NOTE — Some workers have found alkaline earth sulphide solutions more stable than Na_2S solutions.

7 INSOLUBLE MATTER, CALCIUM, MAGNESIUM AND AMMONIUM HYDROXIDE PRECIPITATE *(0.2 % maximum residue)*

Dissolve 10 ± 0.1 g of the sample in 75 ml of distilled water. Add 5 ml of 4 % ammonium oxalate solution, 2 ml of 10 % ammonium phosphate solution and 10 ml of dilute ammonium hydroxide (1 + 9). Allow to stand overnight. If any precipitate is formed, filter and wash with dilute ammonium hydroxide (1 + 39). Dry, ignite at a dull red heat (approximately 600 °C), cool in a desiccator and weigh. The residue mass should be not more than 0.020 g.

8 HEAVY METALS (as Pb) *(0.001 % maximum)*

Prepare a 20 ml heavy-metals test control containing 0.05 mg of lead ion and a 20 ml iron test control containing a soluble iron salt equivalent to 0.15 mg of iron (see 9.2). Dissolve 5 ± 0.1 g of the sample to be tested in 20 ml of distilled water. Treat both test controls and the sample solution in the same manner. Add 5 ml of dilute ammonium hydroxide (1 + 2) and then add slowly, in several portions, 40 ml of 15 % hydrogen peroxide solution (prepared by diluting 1 volume of 30 % hydrogen peroxide with 1 volume of distilled water). Allow to stand for 10 min. Evaporate to dryness on a steam bath. Add 5 ml of dilute hydrochloric acid (1 + 1) and 5 ml of distilled water. Heat to boiling. Dilute to 100 ml with distilled water. Neutralize a 20 ml aliquot of both the heavy-metals test control and the sample solution to *p*-nitrophenol indicator with dilute ammonium hydroxide (1 + 2). (Save the iron test control and the balance of the sample solution for the iron test.) Add hydrochloric acid (1 + 99), drop by drop, until neutral to the indicator, then add 1.0 ml excess, followed by 5 ml of hydrogen sulphide water. Dilute to 50 ml and mix well. Any colour produced in the sample solution should be not stronger than that produced in the lead test control. Use Nessler tubes for comparison.

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[1] Reagents used in making the tests should be recognized reagent grade chemicals normally used for careful analytical work. In all the directions, the acids and ammonium hydjroxide referred to should be of full strength, unless dilution is specified. Dilution is specified in terms of normality when standardization of the reagent is required. When dilution is indicated as (1 + *x*) it means 1 volume of the reagent or strong solution diluted with *x* volumes of distilled water.