UDC 77

Ref. No.: ISO / R 419 - 1965 (E)

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION



# ISO RECOMMENDATION
# R 419

## SPECIFICATION FOR PHOTOGRAPHIC GRADE SODIUM THIOSULPHATE, CRYSTALLINE

1st EDITION

March 1965



Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

# BRIEF HISTORY

The ISO Recommendation R 419, *Specification for Photographic Grade Sodium Thiosulphate, Crystalline*, was drawn up by Technical Committee ISO/TC 42, *Photography*, the Secretariat of which is held by the American Standards Association, Inc. (ASA).

Work on this question by the Technical Committee began in 1956 and led, in 1958, to the adoption of a Draft ISO Recommendation.

In August 1961, this Draft ISO Recommendation (No. 394) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 419 March 1965

# SPECIFICATION FOR PHOTOGRAPHIC GRADE SODIUM THIOSULPHATE, CRYSTALLINE

## 1. SCOPE

This ISO Recommendation 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 (crystal hypo) ($Na_2S_2O_3 \cdot 5H_2O$) is in the form of colourless crystals.

## 3. SUMMARY OF REQUIREMENTS

*Assay* (as $Na_2S_2O_3 \cdot 5H_2O$) : 99.0 per cent minimum.
*Alkalinity* (as NaOH) : 0.016 per cent maximum.
*Acidity* (as $H_2SO_4$) : 0.005 per cent maximum.
*Sulphide* (as $Na_2S$) : 0.001 per cent maximum.
*Insoluble matter, calcium, magnesium and ammonium hydroxide precipitate* : 0.2 per cent maximum residue.
*Heavy metals* (as Pb) : 0.001 per cent maximum.
*Iron* (Fe) : 0.005 per cent maximum.
*Appearance of solution* : to pass test.

## 4. ASSAY (as $Na_2S_2O_3 \cdot 5H_2O$)

*(99.0 per cent minimum)*

### 4.1 Reagents

**4.1.1** *Standard iodine solution.* Weigh accurately about 12.75 g of freshly resublimed iodine * into a tared weighing flask. Add 36 g of potassium iodine and 100 ml of distilled water. After solution is complete, dilute to exactly 1 litre 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 per cent salicylic acid solution. Add 300 to 400 ml of boiling distilled water and boil until the starch dissolves. Dilute to 1 litre.

### 4.2 Procedure

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

$$1 \text{ ml } 0.1 \text{ N I} = 0.0248 \text{ g } Na_2S_2O_3 \cdot 5H_2O$$

## 5. ALKALINITY (as NaOH)

*(0.016 per cent maximum)*

Dissolve 5 ± 0.1 g of the sample in 100 ml of freshly boiled distilled water. Add 3 drops of phenolphthalein indicator. If a pink colour is produced, it should be discharged by the addition of not more than 0.2 ml of 0.1 N hydrochloric acid. If no pink colour is produced, proceed as in section 6.

* 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 hydroxide 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.

### 6. ACIDITY (as $H_2SO_4$)

*(0.005 per cent maximum)*

If no pink colour is produced at the stage described in section 5, the addition of not more than 0.5 ml of 0.01 N sodium hydroxide should produce a pink colour.

### 7. SULPHIDE (as $Na_2S$)

*(0.001 per cent 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 per cent sodium hydroxide solution to a 10 per cent lead acetate ($Pb(C_2H_3O_2)_2 \cdot 3H_2O$) 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. Use Nessler tubes for comparison. Some workers have found alkaline earth sulphide solutions more stable than $Na_2S$ solutions.

NOTE. — The test control sulphide solution should be freshly prepared, using boiled and cooled distilled water.

### 8. INSOLUBLE MATTER, CALCIUM, MAGNESIUM AND AMMONIUM HYDROXIDE PRECIPITATE

*(0.2 per cent maximum residue)*

Dissolve 10 ± 0.1 g of the sample in 75 ml of distilled water. Add 5 ml of 4 per cent ammonium oxalate solution, 2 ml of 10 per cent 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.

### 9. HEAVY METALS (as Pb)

*(0.001 per cent 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 0.25 mg of iron (see clause 10.2). 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 per cent hydrogen peroxide solution (prepared by diluting 1 volume of 30 per cent 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), dropwise, 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.