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**INTERNATIONAL STANDARD**



**3619**

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**Photographic grade ammonium thiosulphate solution —  
Specification**

*Thiosulfate d'ammonium en solution 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 3619 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	Germany	Sweden
Austria	Italy	Turkey
Belgium	Japan	United Kingdom
Bulgaria	Mexico	U.S.A.
Canada	South Africa, Rep. of	U.S.S.R.
France	Spain	Yugoslavia

No Member Body expressed disapproval of the document.

# Photographic grade ammonium thiosulphate 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 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 ammonium thiosulphate solution.

## 2 CHARACTERISTICS

Ammonium thiosulphate solution is a clear, colourless liquid of chemical formula  $(\text{NH}_4)_2\text{S}_2\text{O}_3$  and relative molar mass 148,2.

NOTE — Crystallization may occur at reduced temperatures (cold weather).

## 3 REQUIREMENTS

### 3.1 Assay

The assay shall be not lower than 57,0 % (*m/m*) and not greater than 61,0 % (*m/m*), when determined by the method described in 4.1.

### 3.2 Density

The apparent density, at 20 °C, shall be between 1,315 g/ml and 1,340 g/ml, when determined 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 0,2 % (*m/m*), when determined by the method described in 4.3.

### 3.4 Alkalinity

The alkalinity, expressed as ammonium hydroxide ( $\text{NH}_4\text{OH}$ ), shall be not lower than 0,3 % (*m/m*) and not greater than 1,5 % (*m/m*), when determined by the method described in 4.1.

### 3.5 Sulphide content

The sulphide content, expressed as ammonium sulphide  $(\text{NH}_4)_2\text{S}$ , shall be not greater than 5 mg/kg.

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

### 3.6 Sulphite content

The sulphite content, expressed as ammonium sulphite  $(\text{NH}_4)_2\text{SO}_3$ , shall be not greater than 1 % (*m/m*), when determined by the method described in 4.1.

### 3.7 Residue after ignition

The residue after ignition shall not exceed 0,2 % (*m/m*) when determined by the method described in 4.5.

### 3.8 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 10 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.

### 3.9 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 10 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 solutions referred to shall be of full strength unless dilution is specified. Dilution is specified in terms of molar concentration (molarity)<sup>1)</sup> 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 and determination of alkalinity and sulphite content

#### 4.1.1 Reagents

4.1.1.1 Sodium thiosulphate solution, 0,1 M.

4.1.1.2 Hydrochloric acid, 0,1 M standard volumetric solution.

4.1.1.3 Sodium hydroxide, 0,1 M standard volumetric solution.

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

4.1.1.5 Methyl red indicator solution, 0,1 g/l.

Warm 25 mg of methyl red (finely powdered) with 0,95 ml of 0,05 M sodium hydroxide solution and 5 ml of 95 % (V/V) ethanol. After solution is complete, add 125 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

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 and finally dilute to 1 000 ml with water.

1) 1 mol/l = 1 kmol/m<sup>3</sup> = 1 mol/dm<sup>3</sup> = 1 M.

**4.1.2 Apparatus**

Ordinary laboratory apparatus and

**4.1.2.1 Volumetric flask**, 500 ml capacity, conforming to class A of ISO 1042.

**4.1.2.2 Pipette**, 50 ml capacity, conforming to class A of ISO/R 648.

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

**4.1.3 Procedure**

Weigh, to the nearest 0,01 g, a test portion of about 8 g of the laboratory sample and dilute with 100 ml of water. Add a few drops of the methyl red indicator solution (4.1.1.5) and immediately titrate to the first colour change with the hydrochloric acid solution (4.1.1.2). Dilute the neutralized solution to 500 ml in the volumetric flask (4.1.2.1). To a 50 ml aliquot, add 75 ml of water and 2 ml of the starch indicator solution (4.1.1.6). Titrate with the iodine solution (4.1.1.4) to a permanent blue colour. Then carefully add just enough of the sodium thiosulphate solution (4.1.1.1) to destroy the blue colour, add a few drops of the methyl red indicator solution and titrate with the sodium hydroxide solution (4.1.1.3) to the first colour change.

**4.1.4 Calculation**

The assay, expressed as a percentage by mass of ammonium thiosulphate  $[(\text{NH}_4)_2\text{S}_2\text{O}_3]$ , is given by the formula

$$\frac{296,4 V_2 T_2 - 0,667 V_3 T_3}{m}$$

The alkalinity, expressed as a percentage by mass of ammonium hydroxide  $(\text{NH}_4\text{OH})$ , is given by the formula

$$\frac{3,5 V_1 T_1}{m}$$

The sulphite content, expressed as a percentage by mass of ammonium sulphite  $[(\text{NH}_4)_2\text{SO}_3]$ , is given by the formula

$$\frac{38,8 V_3 T_3}{m}$$

where

$V_1$  is the volume, in millilitres, of the hydrochloric acid solution (4.1.1.2) used for the titration;

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

$V_3$  is the volume, in millilitres, of the sodium hydroxide solution (4.1.1.3) used for the titration;

$T_1$  is the exact molarity of the hydrochloric acid solution (4.1.1.2);

$T_2$  is the exact molarity of the iodine solution (4.1.1.4);

$T_3$  is the exact molarity of the sodium hydroxide solution (4.1.1.3);

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

**4.2 Determination of density**

Measure with a hydrometer including the range 1,30 g/ml to 1,35 g/ml at 20 °C.

**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 Platinum crucible**

**4.3.2.2 Muffle furnace**, capable of being controlled at  $600 \pm 50$  °C.

**4.3.3 Procedure**

Weigh, to the nearest 0,5 g, a test portion of about 10 g of the laboratory sample and dilute with 75 ml of water. Add 5 ml of the ammonium oxalate solution (4.3.1.1), 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) and ignite the residue in the furnace (4.3.2.2), controlled at  $600 \pm 50$  °C, for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

**4.3.4 Calculation**

Matter insoluble in ammonia solution, expressed as a percentage by mass, is given 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 Limit test for sulphide**

**4.4.1 Reagents**

**4.4.1.1 Lead, alkaline solution.**

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

**4.4.1.2 Sulphide, standard solution.**

Immediately before use, dissolve 1,5 g of sodium sulphide nonahydrate  $(\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O})$  in 200 ml of boiled and cooled water. Dilute 1 ml of this solution (1 + 99) with boiled and cooled water. 1 ml of this solution is equivalent to 0,021 mg of ammonium sulphide  $[(\text{NH}_4)_2\text{S}]$ .

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

**4.4.2 Apparatus**

Ordinary laboratory apparatus and

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

**4.4.2.2 Two matched Nessler cylinders, 50 ml capacity.**

**4.4.3 Procedure**

Weigh, to the nearest 0,5 g, a test portion of 20 g of the laboratory sample and mix with 40 ml of water. Treat this and 4,75 ml of the freshly prepared standard sulphide solution (4.4.1.2) in separate Nessler cylinders (4.4.2.2) in the following manner. Add 2 ml of the alkaline lead solution (4.4.1.1) to each, dilute to 50 ml and mix well.

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

**4.5 Determination of residue after ignition**

**4.5.1 Apparatus**

Ordinary laboratory apparatus and

**4.5.1.1 Platinum crucible.**

**4.5.1.2 Muffle furnace, capable of being controlled at  $600 \pm 50^\circ\text{C}$ .**

**4.5.2 Procedure**

Weigh, to the nearest 0,05 g, a test portion of about 2,5 g of the laboratory sample into the previously weighed crucible (4.5.1.1). Evaporate to dryness on a steam-bath, and then heat gently over a burner. Finally, heat the residue in the muffle furnace (4.5.1.2), controlled at  $600 \pm 50^\circ\text{C}$ , for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

**4.5.3 Calculation**

The residue after ignition, expressed as a percentage by mass, is given by the formula

$$\frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

$m_1$  is the mass, in grams, of the crucible;

$m_2$  is the mass, in grams, of the crucible and test portion;

$m_3$  is the mass, in grams, of the crucible and residue.

**4.6 Limit test for heavy metals**

**4.6.1 Reagents**

**4.6.1.1 Hydrochloric acid solution, dilute (1 + 99).**

**4.6.1.2 Ammonia solution, dilute (1 + 2).**

**4.6.1.3 Hydrogen peroxide solution, approximately 167 g/l.**

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

**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 sulphide.**

**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, a test portion of about 5 g of the laboratory sample and dilute with 30 ml of water. Also take 5 ml of the standard heavy metals solution (4.6.1.4) and treat this and the test solution in the following manner. Add 5 ml of the ammonia solution (4.6.1.2) and then add slowly, with great care, in several portions, 40 ml of the hydrogen peroxide solution (4.6.1.3). Allow to stand for 10 min and then evaporate to dryness on a steam-bath. Dissolve the residues in 30 ml of water.

To each, add 2 drops of the *p*-nitrophenol indicator solution (4.6.1.6), followed by the ammonia solution, drop by drop, until the solutions turn yellow. Then add the hydrochloric acid solution (4.6.1.1), drop by drop, until the solutions become colourless and then add 1 ml in excess. Dilute each to 100 ml with water.