# INTERNATIONAL STANDARD

# ISO 5990

Third edition
2000-12-15

# Photography — Processing chemicals — Specifications for potassium sulfite, 650 g/l solution

*Photographie — Produits chimiques de traitement — Spécifications relatives au sulfite de potassium en solution à 650 g/l*

Reference number
ISO 5990:2000(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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## Contents

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 5990 was prepared by Technical Committee ISO/TC 42, *Photography*.

This third edition cancels and replaces the second edition (ISO 5990:1996), of which it constitutes a technical revision.

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# Introduction

This International Standard is one of a series that establishes criteria of purity for chemicals used in processing photographic materials. General test methods and procedures cited in this International Standard are compiled in ISO 10349-1.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques, which may not always be the case. Some of the procedures utilize caustic, toxic or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks or aprons. Normal precautions for the safe performance of any chemical procedure must be exercised at all times, but specific details have been provided for hazardous materials. Hazard warnings designated by a letter enclosed in angle brackets "〈 〉" are used as a reminder in those steps detailing handling operations and are defined in ISO 10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

This International Standard provides chemical and physical requirements for the suitability of a photographic-grade chemical. The tests correlate with undesirable photographic effects. Purity requirements are set as low as possible, consistent with these photographic effects. These criteria are considered to be the minimum requirements necessary to ensure sufficient purity for use in photographic processing solutions, except that if 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 availability of the higher-quality material.

Every effort has been made to keep the number of requirements to a minimum. Inert impurities are limited to amounts that will not unduly reduce the assay. All tests are performed on samples "as received" to reflect the condition of materials furnished for use. Although the ultimate criterion for suitability of such a chemical is its successful performance in an appropriate use test, the shorter, more economical test methods described in this International Standard are generally adequate.

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. Identity tests have been included whenever a possibility exists that another chemical or mixture of chemicals could pass the other tests.

All requirements listed in clause 4 are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

Efforts have been made to employ tests that are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests that 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.

Over the past few years, great improvements have been made in instrumentation for various analyses. Where such techniques have equivalent or greater precision, they may be used in place of the tests described in this International Standard. Correlation of such alternative procedures with the given method is the responsibility of the user. In the case of disagreement in results, the method called for in the specification should prevail. Where a requirement states "to pass test", however, alternative methods should not be used.

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# Photography — Processing chemicals — Specifications for potassium sulfite, 650 g/l solution

## 1 Scope

This International Standard establishes criteria for the purity of photographic-grade potassium sulfite, 650 g/l aqueous solution, and specifies the tests to be used to determine the purity.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10349-1:1992, *Photography — Photographic-grade chemicals — Test methods — Part 1: General*.

ISO 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content*.

ISO 10349-9:1992, *Photography — Photographic-grade chemicals — Test methods — Part 9: Reaction to ammoniacal silver nitrate*.

ISO 10349-11:1992, *Photography — Photographic-grade chemicals — Test methods — Part 11: Determination of specific gravity*.

ISO 10349-12:1992, *Photography — Photographic-grade chemicals — Test methods — Part 12: Determination of density*.

## 3 General

### 3.1 Physical properties

Potassium sulfite ($K_2SO_3$), 650 g/l aqueous solution, is a clear, colourless or almost colourless liquid. Potassium sulfite has a relative molecular mass of 158,27.

### 3.2 Hazardous properties

Potassium sulfite, 650 g/l aqueous solution, is not hazardous when handled with normal precautions. Avoid contact with acids.

### 3.3 Storage

Potassium sulfite solution shall be stored in a closed container at room temperature.

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## 4 Requirements

A summary of the requirements is shown in Table 1.

**Table 1 — Summary of requirements**

| Test | Limit | Subclause | International Standard in which test method is given |
|---|---|---|---|
| Assay | Minimum: 44,5 %<br>Maximum: 46,0 % | 7.1 | ISO 5990 |
| Mass fraction of heavy metals (as Pb) | Maximum: 0,002 % | 7.2 | ISO 10349-5 |
| Mass fraction of iron (Fe) | Maximum: 0,002 % | 7.3 | ISO 10349-5 |
| Reaction to ammoniacal silver nitrate | To pass test | 7.4 | ISO 10349-9 |
| Specific gravity or density: | | | |
| specific gravity | Minimum: 1,445<br>Maximum: 1,460 | 7.5.1 | ISO 10349-11 |
| density | Minimum: 1,441 g/ml<br>Maximum: 1,457 g/ml | 7.5.2 | ISO 10349-12 |
| pH value of (1 + 9) solution | 8,0 to 10,0 | 7.6 | ISO 5990 |
| Mass fraction of thiosulfate (as $S_2O_3^{2-}$) | Maximum: 0,006 % | 7.7 | ISO 5990 |
| Appearance of solution | Clear and free from insoluble matter except for a slight flocculence | 7.8 | ISO 5990 |

## 5 Reagents and glassware

All reagents, materials and glassware shall conform to the requirements specified in ISO 10349-1 unless otherwise noted. The hazard warning symbols, used as a reminder in those steps detailing handling operations, are defined in ISO 10349-1. These symbols are used to provide information to the user and are not meant to provide conformance with hazardous labelling requirements, as these vary from country to country.

## 6 Sampling

See ISO 10349-1.

## 7 Test methods

### 7.1 Assay

#### 7.1.1 Specification

The mass fraction of potassium sulfite shall be between 44,5 % and 46,0 %.

#### 7.1.2 Reagents

**7.1.2.1 Hydrochloric acid** (HCl), $\rho \approx 1{,}18$ g/ml (DANGER: ⟨B⟩⟨C⟩)1).

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1) Hazard warning codes are defined in ISO 10349-1.

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**7.1.2.2 Potassium iodide** (KI).

**7.1.2.3 Iodine**, $c(I_2)$ = 0,05 mol/l (12,7 g/l)2) 3).

Weigh, to the nearest 0,001 g, 12,7 g of freshly sublimed iodine (DANGER: ⟨C⟩⟨O⟩) into a tared weighing flask. Add 36 g of potassium iodide (7.1.2.2) and 100 ml of water. After solution is complete, add three drops of hydrochloric acid (7.1.2.1) (DANGER: ⟨B⟩⟨C⟩), and dilute to 1 litre at 20 °C in a volumetric flask. From the mass of iodine, $m$, calculate the concentration, $c$, in moles per litre, from

$$c = \frac{m}{254}$$

**7.1.2.4 Sodium thiosulfate**, $c(Na_2S_2O_3)$ = 0,100 mol/l (15,8 g/l)2).

NOTE This solution is not required for the direct-titration method (7.1.4.3).

**7.1.2.5 Salicylic acid**, $c(HOC_6H_4COOH)$ = 1 % (10 g/l).

**7.1.2.6 Starch indicator**, 5 g/l solution.

Stir 5 g of soluble starch into 100 ml of 1 % salicylic acid solution (7.1.2.5). Add 300 ml of this solution to 400 ml of boiling water. Boil until the starch dissolves and dilute to 1 litre with water.

### 7.1.3 Apparatus

**7.1.3.1 Burette**, of capacity 50 ml.

**7.1.3.2 Pipette**, of capacity 25 ml.

**7.1.3.3 Pipette**, of capacity 50 ml.

**7.1.3.4 Magnetic stirrer and bar**, for the direct-titration method (7.1.4.3).

### 7.1.4 Procedure

#### 7.1.4.1 Introduction

Use either the back-titration method (7.1.4.2) or the direct-titration method (7.1.4.3).

#### 7.1.4.2 Back-titration method

Using a pipette (7.1.3.2), transfer 25,00 ml of the iodine solution (7.1.2.3) to a glass-stoppered flask. Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g and wash this into the flask. Add 5 ml of the hydrochloric acid (7.1.2.1) (DANGER: ⟨B⟩⟨C⟩) and, using a burette (7.1.3.1), titrate with the sodium thiosulfate solution (7.1.2.4), adding 2 ml of the starch indicator (7.1.2.6) just before the endpoint.

#### 7.1.4.3 Direct-titration method

Weigh, to the nearest 0,000 1 g, a test portion of 0,90 g. Using a pipette (7.1.3.3), transfer 50,00 ml of the iodine solution (7.1.2.3) to a completely dry 250 ml beaker that contains a magnetic stirring bar (7.1.3.4). While stirring the

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2) Commercially available analysed reagent solution is recommended. If the solution is to be prepared, see any quantitative analytical chemistry text.

3) It is recommended that self-prepared iodine solutions be standardized before use.

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iodine solution in the beaker, add the test portion to the centre of the beaker using a camel-hair brush. Avoid contact of the sample with the sides of the beaker.

If the iodine is not decolorized after addition of the sample, discard the trial and restart the procedure. If necessary, increase the test portion by 0,10 g.

Wash down the side walls of the beaker using about 2 ml of the starch indicator (7.1.2.6). Using a burette (7.1.3.1) immediately titrate with the iodine solution to the first permanent light-purple colour. Wash any iodine solution remaining on the burette tip into the solution with deionized water.

If the titration exceeds 10 ml, repeat the test as this can result in test results lower than the actual assay. Adjust the sample appropriately.

### 7.1.5 Expression of results

### 7.1.5.1 Back-titration method

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

$$\frac{7{,}914\left(50\cdot c_1-c_2\cdot V\right)}{m}$$

where

$c_1$ is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.3);

$c_2$ is the actual concentration, expressed in moles per litre, of the sodium thiosulfate solution (7.1.2.4);

$V$ is the volume, expressed in millilitres, of the sodium thiosulfate solution used for the titration in 7.1.4.2;

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

7,914 is the conversion factor obtained from the equivalent mass of potassium sulfite (i.e. 158,27/2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

50 is the conversion factor obtained from the volume, expressed in millilitres, of the iodine solution added in 7.1.4.2 (i.e. 25) × the number of equivalents of the iodine solution (i.e. 2).

### 7.1.5.2 Direct-titration method

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

$$\frac{15{,}827\cdot c_1\left(50+V\right)}{m}$$

where

$c_1$ is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.3);

$V$ is the volume, expressed in millilitres, of the iodine solution used for the titration in 7.1.4.3;

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

15,827 is the conversion factor obtained from the mass of potassium sulfite equivalent to 1 mole of iodine (i.e. 158,27) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

50 is the volume, expressed in millilitres, of the iodine solution added in 7.1.4.3.

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## 7.2 Mass fraction of heavy metals

### 7.2.1 Specification

The maximum mass fraction of heavy metals shall be 0,002 %.

### 7.2.2 Procedure

NOTE The standard for the iron test (7.3) is prepared in the same way as the heavy metals standard.

Determine the percentage of heavy metals in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g, prepared in accordance with ISO 10349-5:1992, 7.3. Use 4 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

## 7.3 Mass fraction of iron

### 7.3.1 Specification

The maximum mass fraction of iron shall be 0,002 %.

### 7.3.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g, prepared in accordance with ISO 10349-5:1992, 7.3. Use 4 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

## 7.4 Reaction to ammoniacal silver nitrate

### 7.4.1 Specification

To pass test.

### 7.4.2 Procedure

Determine the reaction to ammoniacal silver nitrate in accordance with ISO 10349-9.

## 7.5 Specific gravity or density

Either the specific gravity or density is required for conformance.

### 7.5.1 Specific gravity

#### 7.5.1.1 Specification

The specific gravity shall be between 1,445 and 1,460.

#### 7.5.1.2 Procedure

Determine the specific gravity in accordance with ISO 10349-11, using a hydrometer that includes the range from 1,4 to 1,5.

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### 7.5.2 Density

#### 7.5.2.1 Specification

The density shall be between 1,441 g/ml and 1,457 g/ml.

#### 7.5.2.2 Procedure

Determine the density in accordance with ISO 10349-12.

## 7.6 pH value

### 7.6.1 Specification

The pH shall be between 8,0 and 10,0.

### 7.6.2 Apparatus

**7.6.2.1 Electronic pH-meter**, equipped with a glass electrode and standard reference electrode.

### 7.6.3 Procedure

Measure, to the nearest 0,5 ml, a test portion of 10 ml; add to 90 ml of water and mix well. Measure the pH of the solution at 20 °C, using the pH-meter (7.6.2.1) in accordance with the manufacturer's instructions.

## 7.7 Mass fraction of thiosulfate (as $S_2O_3^{2-}$)

### 7.7.1 Specification

The maximum content of thiosulfate shall be 0,006 % (0,01 % as $K_2S_2O_3$).

### 7.7.2 Reagents

**7.7.2.1 Potassium bromide** (KBr).

**7.7.2.2 Mercury(II) chloride**, $HgCl_2$ (DANGER: 〈〈S〉〉).

**7.7.2.3 Mercury(II) chloride reagent**.

Dissolve 25 g of potassium bromide (7.7.2.1) and 25 g of mercury(II) chloride (7.7.2.2) (DANGER: 〈〈S〉〉) in 900 ml of water at 50 °C. Cool, dilute to 1 litre and allow to stand overnight. Filter the solution if it is not perfectly clear.

**7.7.2.4 Thiosulfate standard solution**, $\rho(S_2O_3^{2-})$ = 0,056 mg/ml.

Dilute 5 ml of the thiosulfate solution (7.1.2.4) to 1 litre.

### 7.7.3 Apparatus

**7.7.3.1 Graduated pipettes**, of capacity 1 ml.

**7.7.3.2 Two matched Nessler colour-comparison cylinders**, of capacity 50 ml.

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### 7.7.4 Procedure

Measure, to the nearest 0,5 ml, a test portion of 10 ml and add to 90 ml of water. Then mix well [or use the solution prepared for the pH test (7.6.3)]. Using a pipette (7.7.3.1), slowly transfer 1,6 ml of this solution into 10 ml of the mercury(II) chloride reagent (7.7.2.3) contained in one of the Nessler colour-comparison cylinders (7.7.3.2). To 10 ml of the mercury(II) chloride reagent contained in the second Nessler colour-comparison cylinder, slowly add 0,25 ml of the thiosulfate standard solution (7.7.2.4) using a second pipette. Swirl to mix and allow both 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 colour-comparison cylinders, the opalescence produced in the test and control solutions. The opalescence in the test solution shall not exceed that of the control solution.

NOTE If the solutions are allowed to stand for more than 15 min, reactions occur which will affect the results.

## 7.8 Appearance of solution

### 7.8.1 Specification

The solution shall be clear and free from insoluble matter except for a slight flocculence.

### 7.8.2 Procedure

Dilute 100 ml of the test sample with 400 ml of water. Observe the solution for colour and clarity.

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