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Photographic grade potassium thiocyanate — Specification

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FOREWORD

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Photographic grade potassium thiocyanate – 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 potassium thiocyanate.

2 CHARACTERISTICS

Potassium thiocyanate is in the form of colourless, deliquescent crystals of chemical formula KCNS and relative molar mass 97,18.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 98,0 % (*m*/*m*), expressed as KCNS, when determined by the method described in 4.1.

3.2 Appearance of aqueous 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 Appearance of ethanolic solution

An ethanolic solution shall be colourless or almost colourless, and clear and free from sediment, other than a slight flocculence, when examined by the method described in 4.3.

3.4 pH value

The pH value of a 50 g/l aqueous solution shall be between 4,5 and 6,0 at 20 °C, when determined by the method described in 4.4.

3.5 Chloride content

The chloride content, expressed as potassium chloride (KCl), shall be not greater than 0,1 % (*m*/*m*).

Conformity with this requirement shall be determined by the limit test described in 4.5, when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

3.6 Sulphate content

The sulphate content, expressed as potassium sulphate (K_2SO_4), shall be not greater than 0,1 % (*m*/*m*).

Conformity with this requirement shall be determined by the limit test described in 4.6, when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

3.7 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 4.7, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.8 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 2 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.8, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.9 Copper content

The copper content, expressed as copper (Cu), shall be not greater than 1 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.9, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.10 Content of sulphur compounds precipitated by ammoniacal silver nitrate

The content of sulphur compounds precipitated by ammoniacal silver nitrate, expressed as sulphur (S), shall be not greater than 10 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.10, 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 test 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)[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.

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[1] 1 mol/l = 1 kmol/m^3 = 1 mol/dm^3 = 1 M

4.1 Assay

4.1.1 *Reagents*

4.1.1.1 Nitric acid solution, dilute (1 + 9).

4.1.1.2 Ammonium iron(III) sulphate solution, 50 g/l.

4.1.1.3 Silver nitrate, 0,1 M standard volumetric solution.

4.1.1.4 Ammonium thiocyanate, 0,1 M standard volumetric solution.

4.1.2 *Apparatus*

Ordinary laboratory apparatus and

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

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

4.1.3 *Procedure*

Weigh, to the nearest 0,000 1 g, a test portion of about 0,3 g of the laboratory sample and dissolve in 50 ml of water. Add 5 ml of the nitric acid solution (4.1.1.1), followed by 50,00 ml of the silver nitrate solution (4.1.1.3), using the pipette (4.1.2.2). Shake well, add 2 ml of the ammonium iron(III) sulphate solution (4.1.1.2) and titrate the excess of silver nitrate with the ammonium thiocyanate solution (4.1.1.4).

4.1.4 *Calculation*

The assay, expressed as a percentage by mass of potassium thiocyanate (KCNS), is given by the formula

$$\frac{9{,}718\,(50\,T_1 - VT_2)}{m}$$

where

V is the volume, in millilitres, of the ammonium thiocyanate solution (4.1.1.4) used for the titration;

T_1 is the exact molarity of the silver nitrate solution (4.1.1.3);

T_2 is the exact molarity of the ammonium thiocyanate solution (4.1.1.4);

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

4.2 Appearance of aqueous solution test

Prepare a 100 g/l solution of the laboratory sample and examine for clarity and sediment at 20 °C.

4.3 Appearance of ethanolic solution test

Prepare a 100 g/l solution of the laboratory sample in ethanol and examine for clarity and sediment at 20 °C.

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, a test portion of about 5 g of the laboratory sample, dissolve in about 80 ml of freshly boiled water and dilute to 100 ml. Measure the pH of the solution at 20 °C, using the pH meter in accordance with the manufacturer's instructions.

4.5 Limit test for chloride

4.5.1 *Reagents*

4.5.1.1 Sodium hydroxide, solid.

4.5.1.2 Nitric acid solution, dilute (1 + 9).

4.5.1.3 Hydrogen peroxide solution, approximately 67 g/l.

Dilute (1 + 4) hydrogen peroxide solution "30 %".

4.5.1.4 Chloride, standard solution.

Dissolve 0,01 g of potassium chloride in 1 000 ml of the nitric acid solution (4.5.1.2).

4.5.1.5 Silver nitrate solution, 100 g/l.

4.5.2 *Apparatus*

Ordinary laboratory apparatus and

4.5.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.5.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 1 g of the laboratory sample, and dissolve in 30 ml of the hydrogen peroxide solution (4.5.1.3) in a conical flask. Add 1 g of the sodium hydroxide (4.5.1.1) and rotate the flask until the vigorous reaction ceases. Then add a further 30 ml of the hydrogen peroxide solution (4.5.1.3) and boil for 2 min. Cool and dilute to 100 ml.

Transfer 10 ml aliquots of this test solution and of the standard chloride solution (4.5.1.4) to separate Nessler cylinders (4.5.2.1) and treat each solution as follows. Add 10 ml of the nitric acid solution (4.5.1.2), dilute to 50 ml and then add 1 ml of the silver nitrate solution (4.5.1.5) and mix well.

Compare, in the Nessler cylinders, the turbidities produced after 5 min in the test and control solutions.

4.6 Limit test for sulphate

4.6.1 *Reagents*

4.6.1.1 Hydrochloric acid solution, dilute (1 + 9).

4.6.1.2 Barium chloride solution.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in 1 000 ml of water.

4.6.1.3 Sulphate, standard solution.

Dissolve a soluble sulphate salt in water to give a solution containing 100 mg of sulphate (SO_4^{--}) per 1 000 ml.

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 0,9 g of the laboratory sample and dissolve in 30 ml of water in one of the Nessler cylinders (4.6.2.1). Transfer 5 ml of the standard sulphate solution (4.6.1.3) to the other Nessler cylinder. Add 0,5 ml of the hydrochloric acid solution (4.6.1.1) and 1 ml of the barium chloride solution (4.6.1.2) to each, dilute to 50 ml, mix well and let stand 15 min.

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

4.7 Limit test for heavy metals

4.7.1 *Reagents*

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

4.7.1.2 Ammonia solution, dilute (1 + 9).

4.7.1.3 Heavy metals, standard solution.

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

4.7.1.4 Water, saturated at room temperature with hydrogen sulphide.

4.7.1.5 *p*-Nitrophenol indicator solution, 2,5 g/l.

4.7.2 *Apparatus*

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.7.3 *Procedure*

Weigh, to the nearest 0,05 g, a test portion of about 2 g of the laboratory sample and dissolve in 20 ml of water in one of the Nessler cylinders (4.7.2.1). Also transfer 4 ml of the standard heavy metals solution (4.7.1.3) to the other Nessler cylinder. To each, add 1 drop of the *p*-nitrophenol indicator solution (4.7.1.5) followed by the ammonia solution (4.7.1.2), drop by drop, until the solutions turn yellow. Add the hydrochloric acid solution (4.7.1.1), drop by drop, until the solutions become colourless and then add 0,5 ml in excess. Finally add 5 ml of the hydrogen sulphide water (4.7.1.4), dilute to 50 ml and mix well.

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

4.8 Limit test for iron

4.8.1 *Reagents*

4.8.1.1 Acetate buffer solution, pH 5,0.

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2M acetic acid and dilute to 1 000 ml with water. Adjust the final pH to 5,0 ± 0,1 with glacial acetic acid or 100 g/l sodium hydroxide solution.

4.8.1.2 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.

4.8.1.3 1,10-Phenanthroline reagent solution.

Thoroughly mix equal volumes of a 1 g/l aqueous solution of 1,10-phenanthroline, a 100 g/l aqueous solution of hydroxylammonium chloride and the acetate buffer solution (4.8.1.1).

4.8.2 *Apparatus*

Ordinary laboratory apparatus and

4.8.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.8.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 10 g of the laboratory sample and dissolve in 20 ml of water in one of the Nessler cylinders (4.8.2.1). Transfer 2 ml of the standard iron solution (4.8.1.2) to the Nessler cylinder. Add 10 ml of the 1,10-phenanthroline reagent solution (4.8.1.3) to each, mix and allow to stand for 10 min. Dilute each to 50 ml and mix well.

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

4.9 Limit test for copper

4.9.1 *Reagents*

4.9.1.1 Citric acid solution, 200 g/l.