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Photographic grade aluminium potassium sulphate dodecahydrate — Specification

*Sulfate double d'aluminium et de potassium dodécahydraté de qualité photographique —
Spécifications*

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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 3620 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 aluminium potassium sulphate dodecahydrate — 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 aluminium potassium sulphate, dodecahydrate.

2 CHARACTERISTICS

Aluminium potassium sulphate, dodecahydrate, is in the form of colourless crystals or a white powder, of chemical formula $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ and relative molar mass 474,4.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 99,5 % (*m/m*), expressed as $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, when determined by the method described in 4.1.

3.2 Appearance of 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 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 50 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.3, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.4 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 100 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.

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 solution 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.

1) 1 mol/l = 1 kmol/m³ = 1 mol/dm³ = 1 M

2) Listed in colour index as C.I. 14645. Trade names include Chrome Fast Black CAT, KIT * TS, Eriochrome Black DW, T and TDW, Potting Black C, etc.

Distilled water, or water otherwise produced of at least equal purity, shall be used whenever water is required.

4.1 Assay

4.1.1 Reagents

4.1.1.1 Hexamine, solid.

4.1.1.2 Buffer solution, pH 9,5 to 10,0.

Dissolve 54 g of ammonium chloride in 200 ml of water, add 350 ml of ammonia solution, ρ approximately 0,910 g/ml, and dilute to 1 000 ml.

4.1.1.3 Zinc, 0,1 M standard volumetric solution.

4.1.1.4 (Ethylenedinitrilo) tetraacetic acid (EDTA), *d/sodium salt*, dihydrate, 0,05 M standard volumetric solution.

Dissolve 37 g of *d/sodium* EDTA, dihydrate, in water and dilute to 1 000 ml. Standardize by titrating against the standard volumetric zinc solution (4.1.1.3) in 5 ml of the buffer solution (4.1.1.2) and 70 ml of water, using 0,1 g of the Mordant black 11 indicator (4.1.1.6). The end-point is indicated by the colour change from violet-red to blue. Using the factor of the zinc solution (4.1.1.3), dilute the *d/sodium* EDTA solution with water to exactly 0,05 M.

4.1.1.5 Lead nitrate, 0,05 M standard volumetric solution.

4.1.1.6 C.I. 14645, C.I. Mordant black 11 indicator.²⁾

Grind 0,25 g of Mordant black 11 with 25 g of sodium chloride in a mortar.

4.1.1.7 Xylenol orange indicator solution, 1 g/l.

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, 20 ml capacity, conforming to class A of ISO/R 648.

4.1.3 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 2,25 g of the laboratory sample, dissolve in water and dilute to 1 000 ml. Using the pipette (4.1.2.2), take 20 ml of this solution and add 40,0 ml of the standard volumetric *d/sodium* EDTA solution (4.1.1.4), followed by 100 ml of water. Heat on a water-bath for 10 min, cool, add

1 g of hexamine (4.1.1.1) and, using the burette (4.1.2.1), titrate with the standard volumetric lead nitrate solution (4.1.1.5), using 0,4 ml of the xylenol orange indicator solution (4.1.1.7).

4.1.4 Calculation

The assay, expressed as a percentage by mass of aluminium potassium sulphate, dodecahydrate $[\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$, is given by the formula

$$\frac{474 - 237VT}{m}$$

where

V is the volume, in millilitres, of the lead nitrate solution (4.1.1.5) used for the titration;

T is the exact molarity of the lead nitrate solution (4.1.1.5);

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

4.2 Appearance of solution test

Prepare a 50 g/l solution of the laboratory sample in water and examine for clarity and sediment.

4.3 Limit test for heavy metals

4.3.1 Reagents

4.3.1.1 Hydrochloric acid solution dilute (1 + 99)

4.3.1.2 Heavy metals, standard solution.

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

4.3.1.3 Water, saturated at room temperature with hydrogen sulphide.

4.3.2 Apparatus

Ordinary laboratory apparatus and

4.3.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.3.3 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 1 g of the laboratory sample, transfer to one of the Nessler cylinders (4.3.2.1) and dissolve in 25 ml of water. Also

transfer 5 ml of the standard heavy metals solution (4.3.1.2) to the other Nessler cylinder and treat this and the test solution in the following manner. Add 1 ml of the hydrochloric acid solution (4.3.1.1), followed by 10 ml of the hydrogen sulphide water (4.3.1.3), dilute to 50 ml and mix well.

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

4.4 Limit test for iron

4.4.1 Reagents

4.4.1.1 Acetate buffer solution, pH 5,0.

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

4.4.1.2 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.

4.4.1.3 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 (4.4.1.1).

4.4.2 Apparatus

Ordinary laboratory apparatus and

4.4.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.4.3 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 0,5 g of the laboratory sample, transfer to one of the Nessler cylinders (4.4.2.1) and dissolve in 25 ml of water. Also transfer 5 ml of the standard iron solution (4.4.1.2) to the other Nessler cylinder, add 20 ml of water and treat this and the test solution in the following manner. Add 5 ml of the 1,10-phenanthroline reagent solution, 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.

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