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Photographic grade glacial acetic acid — Specification

Acide acétique cristallisable 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 3298 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	Spain
Austria	Italy	Turkey
Belgium	Japan	United Kingdom
Bulgaria	Mexico	U.S.A.
Canada	Netherlands	U.S.S.R.
Czechoslovakia	Romania	Yugoslavia
France	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Photographic grade glacial acetic acid — 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 recognised 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 glacial acetic acid.

2 CHARACTERISTICS

Glacial acetic acid is a colourless liquid, of chemical formula CH_3COOH and relative molar mass 60.

3 REQUIREMENTS

3.1 Melting point (assay)

The melting point shall be not lower than $15,7^\circ\text{C}$ when determined by the method described in 4.1.

This requirement corresponds to a minimum acetic acid content (assay) of 99,5 % (m/m).

3.2 Non-volatile matter content

The non-volatile matter content, when determined by the method described in 4.2, shall not be greater than 50 mg/l.

3.3 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 4.3, 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 10 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.

3.5 Appearance of solution

An aqueous solution shall be clear and colourless, as compared to water, when prepared and examined by the method described in 4.5.

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.

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

4.1 Determination of melting point

4.1.1 Apparatus

4.1.1.1 Thermometer, covering the range 10 to 20°C or greater, subdivided at intervals of $0,1^\circ\text{C}$.

4.1.1.2 Test tube, about 25 mm in diameter.

4.1.1.3 Ice bath.

4.1.2 Procedure

Insert the thermometer (4.1.1.1) into the test tube (4.1.1.2) containing sufficient of the sample to reach the immersion mark on the thermometer when its bulb is about 12 mm above the bottom of the test tube, and chill in the ice bath (4.1.1.3) with stirring until a semi-solid mass is obtained. Remove from the ice bath and allow the temperature to rise, stirring the mass gently with the thermometer. Plot a graph of time as abscissa versus temperature as ordinate. The melting point is that temperature at which the graph first remains horizontal.

4.2 Determination of non-volatile matter content

4.2.1 Apparatus

Ordinary laboratory apparatus and

4.2.1.1 Platinum dish, of capacity 100 ml or greater.

4.2.1.2 Oven, capable of being controlled at $105 \pm 2^\circ\text{C}$.

4.2.2 Procedure

Dry the clean platinum dish (4.2.1.1) for 60 ± 5 min in the oven (4.2.1.2) maintained at $105 \pm 2^\circ\text{C}$, allow the dish to cool in a desiccator and weigh it to the nearest 1 mg.

Measure 95 ± 1 ml of the sample into the dish, and evaporate to dryness on a steam bath in a fume cupboard. Dry the residue in the dish in the oven, maintained at $105 \pm 2^\circ\text{C}$, for 60 ± 5 min, cool in a desiccator, and weigh the dish and contents to the nearest 1 mg.

NOTE — Retain the residue in the dish for preparation of the test solution (4.3.3) used in the limit tests for heavy metals and iron.

1) $1 \text{ mol/l} = 1 \text{ kmol/m}^3 = 1 \text{ mol/dm}^3 = 1 \text{ M}$

4.2.3 Expression of results

The non-volatile matter content is given, in milligrams per litre, by the formula

$$\frac{m_2 - m_1}{95} \times 10^6$$

where

m_1 is the mass, in grams, of the dish;

m_2 is the mass, in grams, of the dish plus residue.

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 One-mark volumetric flask, 1 000 ml capacity, conforming to class A of ISO 1042.

4.3.2.2 Two matched Nessler cylinders, 50 ml capacity.

4.3.3 Test solution

Dissolve the residue from the determination of non-volatile matter (4.2.2) in 1,0 ml of the hydrochloric acid solution (4.3.1.1), transfer quantitatively to the volumetric flask (4.3.2.1), dilute to the mark with water and mix well.

4.3.4 Procedure

Transfer 5 ml of the standard heavy metals solution (4.3.1.2) to one of the Nessler cylinders (4.3.2.2) and add 1,0 ml of the hydrochloric acid solution (4.3.1.1). Transfer 25 ml of the test solution (4.3.3) to the other Nessler cylinder, retaining the balance of the test solution for the

iron test (4.4.3). Add 10 ml of the hydrogen sulphide water (4.3.1.3) to each cylinder, 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 of the solution 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 hydrochloride 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

Transfer 2,5 ml of the standard iron solution (4.4.1.2) to one of the Nessler cylinders (4.4.2.1) and 25 ml of the balance of the test solution from 4.3.4 to the other Nessler cylinder. Add 10 ml of the 1,10-phenanthroline reagent solution (4.4.1.3) to each, mix well 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.

4.5 Solution test

Dilute 10 ml of the sample to 100 ml with water, and compare, in Nessler cylinders, the clarity and colour with that of water.

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