INTERNATIONAL STANDARD 423

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Photographic grade hydroquinone — Specification

Hydroquinone 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 42 has reviewed ISO Recommendation R 423 and found it technically suitable for transformation. International Standard ISO 423 therefore replaces ISO Recommendation R 423-1965.

ISO Recommendation R 423 was approved by the Member Bodies of the following countries :

Belgium
Brazil
Canada
Chile
France
Germany
Italy
Japan
Netherlands
New Zealand
Romania
Sweden
Switzerland
United Kingdom
U.S.A.
U.S.S.R.

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 423 into an International Standard.

Printed in Switzerland

Photographic grade hydroquinone — 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 hydroquinone.

2 CHARACTERISTICS

Hydroquinone is in the form of white or almost white crystals or crystalline powder, of chemical formula $C_6H_4(OH)_2$ and relative molar mass 110,1.

3 REQUIREMENTS

3.1 Assay

The assay shall be not lower than 99,0 % (*m*/*m*) and not greater than 101 % (*m*/*m*), expressed as $C_6H_4(OH)_2$, when determined by the method described in 4.1.

3.2 Appearance of solution

A dilute acetic acid solution shall be clear and colourless when prepared and examined by the method described in 4.2.

3.3 Residue after ignition

The residue after ignition shall be not greater than 0,10 % (*m*/*m*), when determined by the method described in 4.3.

3.4 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 4.4, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.5 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 20 mg/kg.

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

3.6 Identity

3.6.1 *Melting point*

The melting point shall be not lower than 171 °C and not higher than 175 °C, when determined by the method described in 4.6.1.

3.6.2 *Mixed melting point*

The melting point of the mixture of sample and standard shall be not lower than the melting point of either the sample or the standard, when determined by the method described in 4.6.1.

3.6.3 *Infra-red spectrum*

The infra-red absorption curve shall be essentially the same as that of the reference spectrum (see the figure), when determined by the method described in 4.6.2.

This requirement is an optional identity requirement supplementary to those of 3.6.1 and 3.6.2.

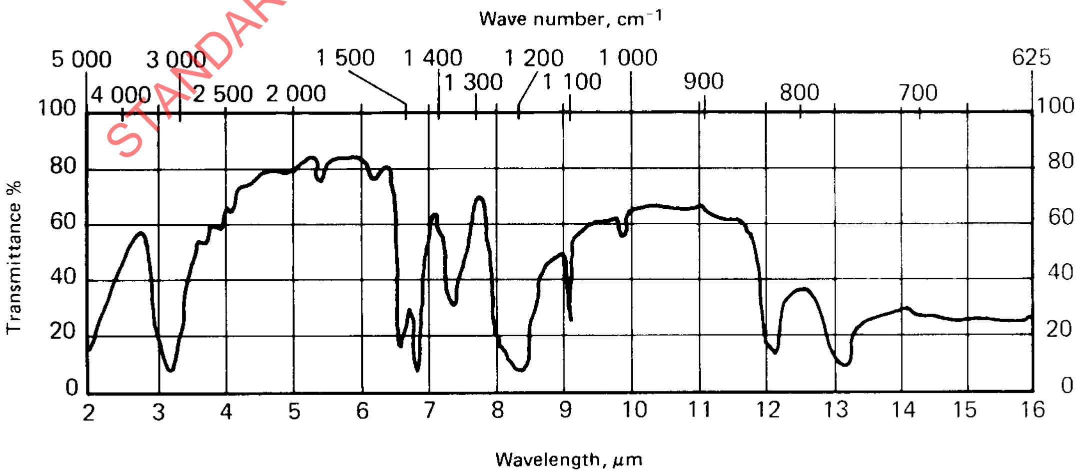

FIGURE — **Reference infra-red spectrum of hydroquinone**
(KBr plate)

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 Assay

4.1.1 *Reagents*

4.1.1.1 Arsenic trioxide (As_2O_3), primary standard.

4.1.1.2 Osmium tetroxide solution.

Dissolve 0,25 g of osmium tetroxide in 100 ml of the 0,05 M sulphuric acid solution (4.1.1.6).

CAUTION — Osmium tetroxide is poisonous — avoid contact.

4.1.1.3 Sulphuric acid solution, ρ approximately 1,84 g/ml.

4.1.1.4 Sulphuric acid solution, dilute (1 + 5).

4.1.1.5 Sodium hydroxide solution, 100 g/l.

4.1.1.6 Sulphuric acid, 0,05 M standard volumetric solution.

4.1.1.7 Cerate, 0,1 M standard volumetric solution.

4.1.1.7.1 PREPARATION

Weigh 50 to 54 g of ammonium cerium(IV) hexanitrate $[(NH_4)_2Ce(NO_3)_6]$ and mix with 27 ml of the sulphuric acid solution (4.1.1.3) in a 600 ml beaker with mechanical stirring. **Using extreme caution,** add water in 100 ml portions[2], with mechanical stirring, allowing 2 to 3 min between each portion. Continue the additon of water until the cerate is completely dissolved. Dilute to 1 000 ml with water and mix well.

NOTE — A primary standard cerium(IV) compound may be used as an alternative to standardization with arsenic trioxide as described in 4.1.1.7.2.

4.1.1.7.2 STANDARDIZATION

Weigh, to the nearest 0,000 1 g, approximately 0,2 g of the primary standard dry arsenic trioxide (4.1.1.1) on the watch glass (4.1.2.1). Transfer the watch glass and contents to a 250 ml wide-mouth conical flask. Add 15 ml of the sodium hydroxide solution (4.1.1.5) and warm the mixture gently. When dissolution is complete, cool to 20 ± 5 °C and add 25 ml of the sulphuric acid solution (4.1.1.4). Dilute to 100 ml with water, add 3 drops (approximately 0,15 ml) of the osmium tetroxide solution (4.1.1.2) and 1 drop of the ferroin indicator solution (4.1.1.8). Titrate this solution with the cerate solution (4.1.1.7) until the reddish-orange colour changes to colourless or very pale blue. A sluggish end-point indicates insufficient osmium tetroxide; up to 10 to 15 drops (approximately 0,7 ml) may be required as the solution ages.

4.1.1.7.3 CALCULATION

The molarity, T, of the cerate solution is given by the formula

$$T = \frac{20{,}22\, m}{V}$$

where

m is the mass, in grams, of the arsenic trioxide (4.1.1.1);

V is the volume, in millilitres, of the cerate solution (4.1.1.7) used for the standardization.

4.1.1.8 Ferroin [tris(1,10-phenanthroline)iron(2+) ion] indicator solution.

4.1.1.9 Diphenylamine indicator solution.

Dissolve 1 g of diphenylamine in 100 ml of the sulphuric acid solution (4.1.1.3).

4.1.2 *Apparatus*

Ordinary laboratory apparatus and

4.1.2.1 Watch glass, 25 mm diameter.

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

4.1.3 *Procedure*

Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g of the laboratory sample on the watch glass (4.1.2.1). Transfer the watch glass and test portion to a 250 ml wide-mouth conical flask containing 100 ml of water and 10 ml of the standard volumetric sulphuric acid solution (4.1.1.6). Dissolve the test portion, add 3 drops of the diphenylamine indicator solution (4.1.1.9) and titrate with the standard volumetric cerate solution (4.1.1.7) to a red-violet end-point.

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[1] 1 mol/l = 1 kmol/m³ = 1 mol/dm³ = 1 M

[2] Normal procedure for mixing acid and water is to add acid slowly to the water. The procedure being used here is the reverse of the normal. Therefore, **extreme caution** must be used to avoid the possible splattering due to heat of evolution.

4.1.4 *Calculation*

The assay, expressed as a percentage by mass of hydroquinone [$C_6H_4(OH)_2$], is given by the formula

$$\frac{5{,}5\,VT}{m}$$

where

V is the volume, in millilitres, of the cerate solution (4.1.1.7) used for the titration;

T is the exact molarity of the cerate solution (4.1.1.7),

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

4.2 Appearance of solution

4.2.1 *Reagent*

4.2.1.1 Acetic acid solution, dilute (1 + 19).

4.2.2 *Procedure*

Prepare a 50 g/l solution of the laboratory sample in the acetic acid solution (4.2.1.1) and examine for clarity and colour by both reflected and transmitted light.

4.3 Determination of residue after ignition

4.3.1 *Apparatus*

Ordinary laboratory apparatus and

4.3.1.1 Platinum crucible.

4.3.1.2 Muffle furnace, capable of being controlled at 600 ± 50 °C.

4.3.2 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample into the previously weighed platinum crucible (4.3.1.1) and heat carefully. Finally, ignite the residue in the furnace (4.3.1.2), controlled at 600 ± 50 °C, for 4 h. Cool in a desiccator and weigh the crucible and contents to the nearest 1 mg. Retain this residue for the heavy metals and iron limit tests under 4.4.3 and 4.5.3.

4.3.3 *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.

4.4 Limit test for heavy metals

4.4.1 *Reagents*

4.4.1.1 Hydrochloric acid solution, ρ approximately 1,18 g/ml.

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

4.4.1.3 Ammonia solution, dilute (1 + 9).

4.4.1.4 Heavy metals, standard solution.

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

4.4.1.5 Water, saturated at room temperature with hydrogen sulphide.

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

4.4.2 *Apparatus*

Ordinary laboratory apparatus and

4.4.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.4.3 *Procedure*

Dissolve the residue from the ignition test under 4.3.2 in 0,5 ml of the hydrochloric acid solution (4.4.1.1), warming if necessary, and transfer the solution (with washing) to a beaker using about 40 ml of water. Also take 4 ml of the standard heavy metals solution (4.4.1.4) and treat this and the test solution in the following manner. Add 1 drop of the *p*-nitrophenol indicator solution (4.4.1.6) and then, drop by drop, the ammonia solution (4.4.1.3), until just yellow, followed by the hydrochloric acid solution (4.4.1.2), drop by drop, until colourless again. Add 2,5 ml in excess.

Dilute the test solution to 50 ml with water and transfer a 20 ml aliquot to one of the Nessler cylinders (4.4.2.1), retaining the balance for the iron limit test under 4.5.3. Transfer the treated standard heavy metals solution to the other Nessler cylinder and treat both solutions in the following manner. Add 5 ml of the hydrogen sulphide water (4.4.1.5), dilute to 50 ml and mix well.

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

4.5 Limit test for iron

4.5.1 *Reagents*

As specified in 4.4.1. and

4.5.1.1 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 ± 0,1 with glacial acetic acid or 100 g/l sodium hydroxide solution.