INTERNATIONAL STANDARD

ISO 423

Second edition
1994-05-01

Photography — Processing chemicals — Specifications for hydroquinone

Photographie — Produits chimiques pour traitement — Spécifications relatives à l'hydroquinone

Reference number
ISO 423:1994(E)

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.

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.

International Standard ISO 423 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 423:1976), which has been technically revised.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

0.1 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 parts 1, 4 and 5 of ISO 10349.

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, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Normal precautions required in the performance of any chemical procedure are to be exercised at all times but care has been taken to provide warnings 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.

0.2 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 the minimum requirements necessary to assure 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 which 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.

0.3 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.

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 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.

Photography — Processing chemicals — Specifications for hydroquinone

1 Scope

This International Standard establishes criteria for the purity of photographic-grade hydroquinone and describes the tests to be used to determine the purity.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

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

ISO 10349-4:1992, *Photography — Photographic-grade chemicals — Test methods — Part 4: Determination of residue after ignition.*

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

3 General

3.1 Physical properties

Hydroquinone, $C_6H_4(OH)_2$, is in the form of white or almost white needle-like crystals or a free-flowing crystalline powder. It has a relative molecular mass of 110,10.

3.2 Hazardous properties

Hydroquinone can cause eye injuries and dermatitis upon contact with eyes and skin, respectively. Avoid all skin contact, do not breathe the dust or vapour. Wear eye protection and gloves when handling. Contact the manufacturer for further information.

4 Requirements

A summary of the requirements is shown in table 1.

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.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	99,0 % (*m/m*) min.	7.1	ISO 423
Identity			ISO 423
Melting point	171 °C to 175 °C	7.2.1	
Mixed melting point	Not less than sample or standard	7.2.1	
Infrared spectrum	Equivalent to figure 1	7.2.2	
Residue after ignition	0,10 % (*m/m*) max.	7.3	ISO 10349-4
Heavy metals (as Pb)	0,002 % (*m/m*) max.	7.4	ISO 10349-5
Iron (Fe)	0,002 % (*m/m*) max.	7.5	ISO 10349-5
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.6	ISO 423

NOTE — *m*/*m* = mass/mass

7 Test methods

7.1 Assay

7.1.1 Specification

Content of hydroquinone [$C_6H_4(OH)_2$] shall be 99,0 % (*m/m*) min.

7.1.2 Reagents

7.1.2.1 Ammonium cerium(IV) hexanitrate, $(NH_4)_2Ce(NO_3)_6$, standard volumetric solution of 0,10 mol/l[1)2)].

7.1.2.2 Diphenylamine indicator solution.

Dissolve 1 g of diphenylamine in 100 ml of sulfuric acid (7.1.2.3) (<< C >>)[3)].

7.1.2.3 Sulfuric acid, H_2SO_4, standard volumetric solution of 0,05 mol/l[1)4)].

7.1.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

7.1.3.1 Watch glass.

7.1.3.2 Burette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of approximately 0,25 g of the sample on to a watch glass (7.1.3.1). Transfer the watch glass and test portion to a 250 ml wide-mouth conical flask containing 100 ml of water and 10 ml of sulfuric acid (7.1.2.3). Dissolve the sample, add 3 drops of the diphenylamine indicator solution (7.1.2.2) and titrate with the standardized ammonium cerate solution (7.1.2.1) to a red-violet endpoint.

7.1.5 Expression of results

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

$$5{,}51 \cdot c \cdot V / m$$

where

c is the actual concentration, expressed in moles per litre, of the ammonium cerate solution (7.1.2.1);

V is the volume, in millilitres, of the ammonium cerate solution (7.1.2.1) used for the titration;

---

1) Commercially available analysed reagent is recommended.

2) A procedure to prepare ammonium cerium(IV) hexanitrate solution (0,10 mol/l) is given in annex A.

3) Hazard warning codes are defined in ISO 10349-1:1992, clause 4.

4) This may be prepared from sulfuric acid, $\rho = 1{,}81$ g/ml (approximately) (DANGER: << C >>).

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

5,51 is the conversion factor of the equivalent mass of hydroquinone per mole of ammonium cerate (i.e. 110,1/2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Identity tests

7.2.1 Melting point

7.2.1.1 Specifications

The melting point shall be from 171 °C to 175 °C.

A mixed melting point shall be from 171 °C to 175 °C and shall not be less than either the sample or the standard.

7.2.1.2 Apparatus

7.2.1.2.1 Capillary-tube melting point apparatus, complete with thermometer for the range 100 °C to 200 °C.

7.2.1.3 Procedure

Prepare three capillary tubes containing:

a) the sample to be tested;

b) a sample known to be hydroquinone;

c) a finely ground mixture of a) and b) mixed in equal proportions.

Identify the tubes and attach them to the thermometer. Heat the apparatus (7.2.1.2.1) to about 155 °C. Insert the thermometer with the samples attached, and thereafter heat so that the temperature rises at a constant rate of 1 °C/min. Note the melting point of each sample as indicated by the first appearance of liquefaction.

7.2.2 Infrared spectrum

7.2.2.1 Specification

The infrared absorption curve shall be essentially the same as that of the reference spectrum (figure 1). This optional recommendation is supplementary to the requirements of 7.2.1.

7.2.2.2 Apparatus

7.2.2.2.1 Test sieve, 63 µm aperture size, conforming to ISO 565.

7.2.2.2.2 Infrared spectrometer, equipped for the 2 µm to 16 µm regions, and accessory equipment for using potassium bromide plates or mineral oil mull.

7.2.2.3 Procedure

Grind about 1 g of the sample to a homogeneous fine powder and prepare a 0,5 % (*m/m*) mixture of the sample in finely ground potassium bromide. Grind together thoroughly to pass through the test sieve (7.2.2.2.1). Prepare a pressed plate of the mixture containing 0,13 g to 0,16 g of the mixture per square centimetre. Record the infrared spectrum from 2 µm to 16 µm. Compare with the reference spectrum given in figure 1.

NOTE 1 As an alternative procedure, the sample may be ground and dispersed in mineral oil. It will then be necessary to take into account the absorption bands of the oil.

7.3 Residue after ignition

7.3.1 Specification

Maximum residue after ignition shall be 0,10 % (*m/m*).

7.3.2 Procedure

Determine the residue after ignition in accordance with ISO 10349-4. Weigh, to the nearest 0,01 g, a test portion of about 5,0 g of the sample and incinerate at 600 °C ± 50 °C for 4 h. Cool in a desiccator and then weigh to the nearest 0,001 g. Retain this residue for the heavy metals (7.4) and iron (7.5) tests.

7.4 Heavy metals content

7.4.1 Specification

Maximum heavy metals content shall be 0,002 % (*m/m*).

7.4.2 Procedure

NOTE 2 The standard for the iron test (7.5) 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 the residue after ignition (7.3) corresponding to 1 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 5 ml of the 25 ml residue solution). Use 2 ml of heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.5 Iron content

7.5.1 Specification

Maximum iron content shall be 0,002 % (*m/m*).

7.5.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of the residue after ignition (7.3) corresponding to 1 g of the test sample prepared in accordance with ISO 10349-5:1992, 7.1. (i.e. 5 ml of the 25 ml residue solution). Use 2 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.6 Appearance of solution

7.6.1 Specification

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

7.6.2 Reagent

7.6.2.1 Acetic acid (1 + 9), prepared from glacial acetic acid (DANGER: < C >< B >).

7.6.3 Procedure

Prepare 50 g/l solution of the sample in the acetic acid solution (7.6.2.1). Examine for clarity and colour by both reflected and transmitted light.

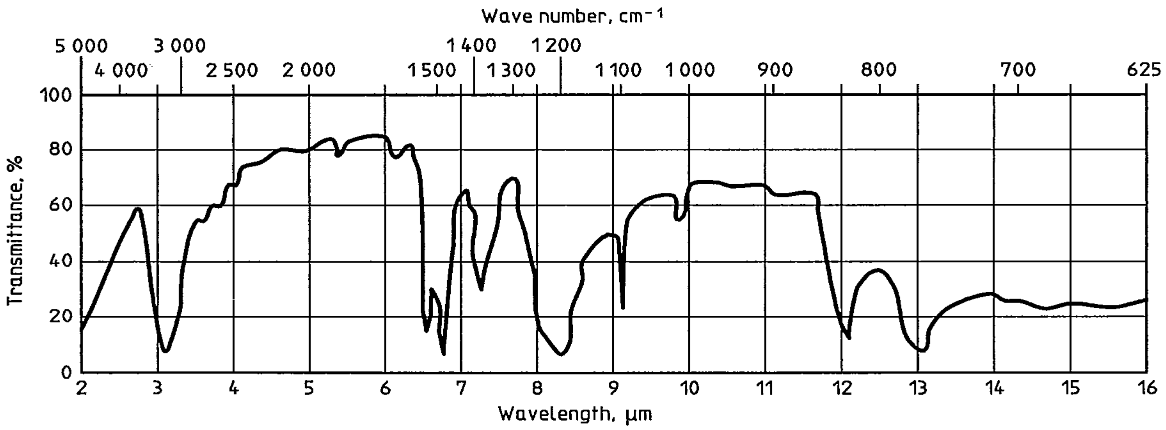

Figure 1 — Reference infrared spectrum of hydroquinone (KBr plate)

Annex A
(informative)

Preparation of standard ammonium cerium(IV) hexanitrate solution, $c[(NH_4)_2Ce(NO_3)_6] = 0,1$ mol/l

A.1 Reagents

A.1.1 Ammonium cerium(IV) hexanitrate, $(NH_4)_2Ce(NO_3)_6$.

A.1.2 Arsenic trioxide (As_2O_3), primary standard grade (DANGER:<< S >>).

A.1.3 Ferroin [tris (1,10-phenanthroline) iron(II) ion] indicator solution.

Dissolve 1,48 g of 1,10-phenanthroline in 100 ml of 0,025 mol/l ferrous sulfate solution.

A.1.4 Osmium tetroxide solution (DANGER: << S >><< B >>< C >).

Dissolve 0,25 g of osmium tetroxide (DANGER: << S >><< B >>< C >) in 100 ml of the 0,05 mol/l sulfuric acid solution (7.1.2.3).

A.1.5 Sodium hydroxide solution, 100 g/l (DANGER: < C >).

A.1.6 Sulfuric acid, $\rho = 1,81$ g/ml (approximately) (DANGER: << C >>).

A.1.7 Sulfuric acid (1 + 5) (DANGER: < C >).

This may be prepared from sulfuric acid (A.1.6) (DANGER: << C >>).

A.2 Preparation

Weigh 50 g to 54 g of ammonium cerium(IV) hexanitrate (A.1.1) and mix with 27 ml sulfuric acid (A.1.6) (<< C >>) in a 600 ml beaker with mechanical stirring. **Using extreme caution**, add water in 100 ml portions, with mechanical stirring, allowing 2 to 3 min between each portion.

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

Continue the addition of water until the ammonium cerium(IV) hexanitrate is completely dissolved. Dilute to 1 000 ml with water and mix well.

NOTE 3 Commercially prepared primary standard cerium(IV) solutions may be used as an alternative to this preparation and the following standardization.

A.3 Standardization

Weigh, to the nearest 0,000 1 g, approximately 0,2 g of primary standard dry arsenic trioxide (A.1.2) (<< S >>) on a watch glass (7.1.3.1). Transfer the watch glass and contents to a 250 ml wide-mouth conical flask. Add 15 ml of sodium hydroxide solution (A.1.5) (< C >) and warm the mixture gently.

When dissolution is complete, cool to 20 °C ± 5 °C and add 25 ml of the sulfuric acid solution (1 + 5) (A.1.7) (< C >). Dilute to 100 ml with water, add 3 drops (approximately 0,15 ml) of the osmium tetroxide solution (A.1.4) (<< S >><< B >>< C >) and 1 drop of the ferroin indicator solution (A.1.3).

Titrate this solution with the cerate solution (A.2) until the reddish orange colour changes to colourless or very pale blue. A sluggish endpoint indicates insufficient osmium tetroxide (A.1.4); up to 10 or 15 drops (approximately 0,7 ml) may be required as the solution ages.

A.4 Expression of results

The actual concentration, in moles per litre, of the ammonium cerate solution is given by

$$20{,}22m/V$$

where

m is the mass, in grams, of the arsenic trioxide (A.1.2);

V is the volume, in millilitres, of the prepared ammonium cerate solution (A.2) being standardized;

20,22 is the conversion factor for the conversion of litres to millilitres (i.e. 1 000) divided by the equivalent weight of arsenic trioxide (197,84/4).

ICS 37.040.30

Descriptors: photography, photographic materials, photographic chemicals, hydroquinoline, specifications, materials specifications, purity criteria, tests, chemical analysis.

Price based on 6 pages