INTERNATIONAL STANDARD

ISO 7760

Second edition
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Photography — Processing waste — Determination of hydroquinone

Photographie — Effluents de traitements — Dosage de l'hydroquinone

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 7760 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 7760:1986), of which it constitutes a technical revision.

Introduction

This International Standard is one of a series devoted to the analysis of photographic wastes; it encompasses the field of analysis of the hydroquinone content in a photographic effluent.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques. Some of the procedures use caustic, toxic, or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks, or aprons. Normal precautions for the safe performance of any chemical procedure must be exercised at all times, but specific details have been provided for hazardous materials. Hazard warnings are designated by a letter enclosed in angle brackets "〈〉". These are defined in clause 5 and then used throughout the text. More detailed information on hazards, handling, and use of these chemicals may be available from the manufacturer.

Photographic laboratories can establish conformity to effluent regulations only by chemical analysis. If this cannot be done in-house, an outside laboratory should be used.

Photography — Processing waste — Determination of hydroquinone

1 Scope

This International Standard specifies a spectrophotometric method for the determination of hydroquinone in photographic processing waste.

This method can be applied to samples containing hydroquinone in the concentration range of 200 μg/l to 4 000 μg/l; aminophenols and phenylenediamines should also be determined by this method. However, sulfonated hydroquinones or products from the further oxidation of benzoquinone will not be determined.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettetes.*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

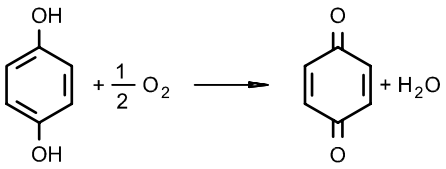
ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series.*

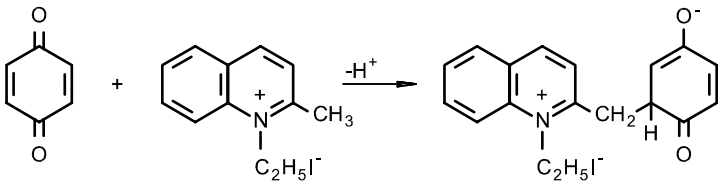
ISO 10349-1:1992, *Photography — Photographic-grade chemicals — Test methods — Part 1: General.*

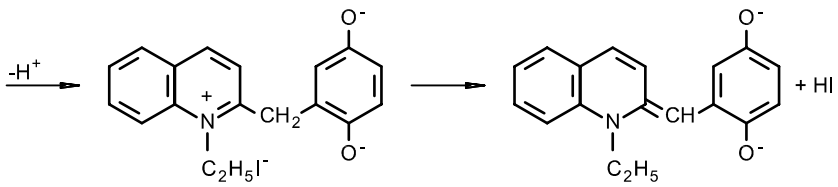
3 Principle

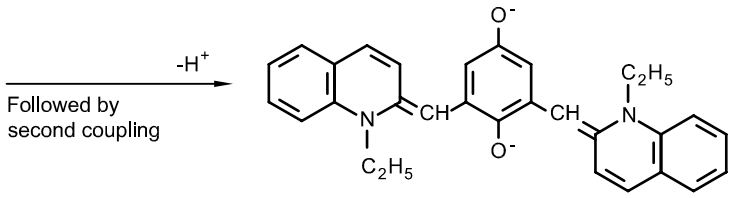
The hydroquinone (together with some other organic compounds) is extracted at a slightly acidic pH from the aqueous sample with 1-pentanol. The extracted hydroquinone is then reacted under alkaline conditions with 1-ethylquinaldinium iodide solution. The absorbance of the coloured complex that is formed is then measured.

4 Reactions









5 Safety and operational precautions

5.1 Hazard warnings

Some of the chemicals specified in the test procedures are caustic, toxic or otherwise hazardous. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks, and aprons. Specific danger notices are given in the text and footnotes for particularly dangerous materials, but normal precautions are required during the performance of any chemical procedure at all times.

The first time that a hazardous material is noted in the test procedures, the hazard will be indicated by the word **"DANGER"** followed by a symbol consisting of angle brackets "〈 〉" containing a letter that designates the specific hazard. A double bracket "〈〈 〉〉" will be used for particularly perilous situations. In subsequent statements involving handling of these hazardous materials, only the hazard symbol consisting of the brackets and letter(s) will be displayed. Furthermore, for a given material, the hazard symbols will be used only once in a single paragraph.

Hazard warning symbols will not be used for common organic solvents when used in quantities of less than 1 litre, unless they are particularly hazardous.

Detailed warnings for handling chemicals and their diluted solutions are beyond the scope of this International Standard.

Employers shall provide training and health and safety information in conformance with legal requirements.

The hazard code system used in this International Standard is intended to provide information to the users and is not meant for compliance with any legal requirements for labelling, as these vary from country to country.

It is strongly recommended that anyone using these chemicals obtain pertinent information from the manufacturer about the hazards, handling, use and disposal of these chemicals.

5.2 Hazard information code system

⟨B⟩ Harmful if inhaled. Avoid breathing dust, vapour, mist or gas. Use only with adequate ventilation.

⟨C⟩ Harmful if contact occurs. Avoid contact with eyes, skin or clothing. Wash thoroughly after handling.

⟨F⟩ Will burn. Keep away from heat, sparks and open flame. Use with adequate ventilation.

⟨O⟩ Oxidizer. Contact with other material may cause fire. Do not store near combustible materials.

⟨S⟩ Harmful if swallowed. Wash thoroughly after handling. If swallowed, obtain medical attention immediately.

⟨⟨S⟩⟩ May be fatal if swallowed. If swallowed, obtain medical attention immediately.

5.3 Safety precautions

ALL PIPETTE OPERATIONS SHALL BE PERFORMED WITH A PIPETTE BULB OR PLUNGER PIPETTE. THIS IS A CRITICAL SAFETY WARNING!

Safety glasses shall be worn for all laboratory work.

6 Materials and reagents

6.1 General

6.1.1 Handling and labelling

Reagents shall be handled in conformity with health and safety precautions as shown on containers or as given in other sources of such information. Proper labelling of prepared reagents includes chemical name, date of preparation, expiration date, restandardization date, name of preparer, and adequate health and safety precautions. The discharge of reagents shall conform to applicable environmental regulations.

6.1.2 Purity

Reagents used in the test procedures shall be certified reagent-grade chemicals and shall meet appropriate standards or be chemicals of a purity acceptable for the analysis. For details, see ISO 6353-1, ISO 6353-2 and ISO 6353-3.

6.1.3 Water

Whenever water is specified without other qualifiers in the test procedures, only distilled water or water of equivalent purity shall be used.

6.1.4 Strength of solutions

6.1.4.1 Acids and ammonium hydroxide are full strength unless otherwise specified.

6.1.4.2 When a standardized solution is required, its amount-of-substance concentration is expressed in moles per litre. The number of significant figures to which the molarity is known shall be sufficient to ensure that the reagent does not limit the reliability of the test method.

6.1.4.3 When a standardized solution is not required, its concentration is expressed in grams per litre (g/l) to the appropriate number of significant figures.

6.1.4.4 When a solution is to be diluted, its dilution is indicated by [x + y], meaning that x volumes of reagent, or concentrated solution, are to be diluted with y volumes of water (6.1.3).

6.2 Reagents

6.2.1 Sodium hydroxide solution, c(NaOH) ≈ 1 mol/l.

Dissolve 20,0 g of sodium hydroxide (DANGER: ⟨⟨C⟩⟩) in 400 ml of water. Cool, then transfer to a 500 ml one-mark volumetric flask and dilute to the mark with water.

6.2.2 Sodium hydroxide solution, c(NaOH) ≈ 0,25 mol/l.

Dilute 50 ml of the 1 mol/l sodium hydroxide (6.2.1) to 200 ml in a 200 ml one-mark volumetric flask.

6.2.3 Citric acid buffer solution

Dissolve 53 g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in about 70 ml of 0,25 mol/l sodium hydroxide (6.2.2). Transfer to a 100 ml one-mark volumetric flask and dilute to the mark with 0,25 mol/l sodium hydroxide.

6.2.4 Water/methanol mixture (DANGER: ⟨S⟩).

Mix 60 ml of water with 90 ml of methanol (DANGER: ⟨S⟩) and cool.

6.2.5 1-Ethylquinaldinium iodide solution, 2 g/l.

Dissolve 0,2 g of 1-ethylquinaldinium iodide in 100 ml of the water/methanol mixture (6.2.4) (⟨S⟩). Store in a dark glass bottle and discard after 24 h.

6.2.6 1-Pentanol, $CH_3CH_2CH_2CH_2CH_2OH$ (DANGER: ⟨F⟩).

6.2.7 Hydrochloric acid solution, HCl [1 + 4] (DANGER: ⟨C⟩).

Dilute 40 ml of concentrated hydrochloric acid (DANGER: ⟨B⟩ ⟨C⟩) to 200 ml.

6.2.8 Hydrochloric acid solution, c(HCl) ≈ 0,01 mol/l.

Add 1,65 ml of concentrated hydrochloric acid (⟨B⟩ ⟨C⟩) to 2 litres of water and mix.

6.2.9 Hydroquinone stock solution, $C_6H_4(OH)_2$ (1 ml contains 100 μg of hydroquinone).

Dissolve 100 mg ± 1 mg of hydroquinone (DANGER: ⟨S⟩) in about 800 ml of 0,01 mol/l hydrochloric acid (6.2.8). Transfer to a 1 litre one-mark volumetric flask and dilute to the mark with 0,01 mol/l hydrochloric acid. Store in a dark glass bottle. Prepare a fresh solution each day.

6.2.10 Hydroquinone standard solution (1 ml contains 5 μg of hydroquinone).

Pipette 10 ml of the hydroquinone stock solution (6.2.9) into a 200 ml one-mark volumetric flask and dilute to the mark with 0,01 mol/l hydrochloric acid (6.2.8). Store in a dark glass bottle. Prepare a fresh solution each day.

6.2.11 pH indicator paper, with a pH range of 4 to 5.

7 Apparatus

7.1 General

All glassware subject to heating shall be of heat-resistant borosilicate glass[1)].

All glassware should be cleaned with hot 1 mol/l hydrochloric acid (⟨C⟩) and rinsed thoroughly before use.

Pipettes and other volumetric glassware shall meet the requirements specified in ISO 10349-1.

7.2 Spectrophotometer, for measurements at a wavelength of 675 nm and fitted with two matched silica cells of 1 cm optical path length.

7.3 Micro-pipette, of capacity 0,1 ml.

7.4 Separatory funnels, of capacity 125 ml.

7.5 One-mark volumetric flasks, of capacity 25 ml, 100 ml, 200 ml, 500 ml and 1 litre, conforming to Class A of ISO 1042 where applicable.

7.6 Pipettes, of capacity 10 ml and 50 ml, conforming to Class A of ISO 648, and 0 ml to 20 ml **graduated pipettes**.

8 Sampling and sample preparation

Hydroquinone ⟨S⟩ readily undergoes aerial oxidation. In the presence of sulfite or bisulfite (which are common photographic processing effluents), the initial oxidation products are sulfonates which will not respond to this test method. The sample container should therefore be filled to the top to minimize dead air space and should be refrigerated if the analysis cannot be run immediately. Filtration of the sample may be necessary.

It is necessary that the analysis be done on a representative sample. The sampling of a process effluent or a plant effluent can encompass many difficulties and due care shall be exercised. See ISO 5667-1, ISO 5667-2 and ISO 5667-3.

Sampling shall be carried out in conformance with regulatory requirements. Sampling should be done under typical operating conditions and normally should be representative of the overall plant effluent. Daily samples that are truly representative of the effluents require sampling over 24 h and sampling that is proportional to the flow rate.

Samples taken during a sudden discharge or during another non-routine operation will not yield results representative of the normal operation.

1) Pyrex ® is an example of suitable glassware available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

9 Procedure

9.1 Extraction

Place 50 ml of the sample, or an aliquot diluted to 50 ml, in a 150 ml beaker and adjust the pH to between 4 and 5 with [1 + 4] hydrochloric acid (6.2.7) (⟨C⟩) or 1 mol/l sodium hydroxide (6.2.1) (⟨⟨C⟩⟩). The pH can be checked with indicator paper (6.2.11). The mass of hydroquinone should not exceed 200 μg per analysis.

Transfer the pH-adjusted sample to a separatory funnel (7.4) and, by means of a graduated pipette (7.6), add 1,0 ml of the citric acid buffer solution (6.2.3), followed by 12,0 ml of the 1-pentanol (6.2.6) (⟨F⟩). Shake the funnel 50 times, then wait 5 min for the liquids to separate. Draw off the aqueous layer (the lower one) and collect it in a second 125 ml separatory funnel. Add 12,0 ml of 1-pentanol to this second funnel, shake the funnel 50 times, then wait 5 min for the liquids to separate. Discard the lower aqueous layer.

Add 10,0 ml of water to the first funnel and shake 20 times. Allow 5 min for separation and transfer the lower aqueous layer to the second funnel. Shake this second funnel 20 times, allow to separate for 5 min, and discard the lower aqueous layer. Transfer the organic layers from the first funnel and the second funnel to a dry 25 ml one-mark volumetric flask (7.5). Rinse the second funnel into the first funnel with 1,0 ml of 1-pentanol (⟨F⟩), and use this to rinse the first funnel into the one-mark volumetric flask. Dilute to the mark, in the 25 ml volumetric flask, with 1-pentanol and mix well. The extract is stable for 24 h if stored in a dark place.

9.2 Colour development and spectrophotometric measurements

Pipette 10,0 ml of the combined 1-pentanol extract into a 25 ml one-mark volumetric flask (7.5). Add 10,0 ml of the 1-ethylquinaldinium iodide solution (6.2.5) and 0,10 ml of 1 mol/l sodium hydroxide (6.2.1) (⟨⟨C⟩⟩). Dilute to the mark with the water/methanol mixture (6.2.4) (⟨S⟩) and mix well. Prepare a blank solution using 10 ml of 1-pentanol (6.2.6) (⟨F⟩) in place of the sample. A blank solution should be prepared for each new batch of reagents.

Let these solutions stand for exactly 20 min. Then measure their absorbances against distilled water at a wavelength of 675 nm, using two matched cells of 1 cm optical path length and a spectrophotometer (7.2) with a tungsten light source. Samples having absorbances above 0,9 shall be diluted with a mixture of 50 % 1-pentanol (⟨F⟩), 30 % methanol (⟨S⟩), and 20 % water by volume. Dilution of the sample should not be attempted if the initial absorbance was greater than 2,5. Instead, carry out another extraction using a smaller sample.

9.3 Preparation of the calibration graph

Carry out the complete procedure on 2,0 ml, 5,0 ml, 10,0 ml and 15,0 ml portions of the standard hydroquinone solution (6.2.10). Calculate the absorbance factor, F, for each amount used by dividing the micrograms of hydroquinone used in the test by the corresponding absorbance. Calculate an average value, and use this to construct a straight-line calibration graph. The slope of this graph, or "F" value, shall be determined empirically for each spectrophotometer. This shall be done with each new lot of 1-ethylquinaldinium iodide.

10 Expression of results

The hydroquinone concentration ρ_{hq}, expressed in milligrams per litre, is given by the formula

$$\rho_{hq} = \frac{2{,}5\left(A_s - A_b\right)F}{V}$$

where

2,5 is the dilution factor (see 9.2);

A_s is the absorbance of the sample at 675 nm;

A_b is the absorbance of the blank solution at 675 nm;

F is the factor (mean) relating absorbance to the number of micrograms of hydroquinone in the final 25 ml flask of measured solution;

V is the volume of the effluent sample taken (usually 50 ml).

11 Test report

The test report shall include the following information:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.

Bibliography

[1] ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

[2] ISO 385-2:1984, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

[3] ISO 385-3:1984, *Laboratory glassware — Burettes — Part 3: Burettes for which a waiting time of 30 s is specified.*

[4] ISO 835-1:1981, *Laboratory glassware — Graduated pipettes — Part 1: General requirements.*

[5] ISO 835-2:1981, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified.*

[6] ISO 835-3:1981, *Laboratory glassware — Graduated pipettes — Part 3: Pipettes for which a waiting time of 15 s is specified.*

[7] ISO 835-4:1981, *Laboratory glassware — Graduated pipettes — Part 4: Blow-out pipettes.*

[8] ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders.*

[9] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ICS 13.030.30; 37.040.30

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