International Standard 7760

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Photographic processing waste — Determination of hydroquinone content — Spectrophotometric method

Déchets des traitements photographiques — Dosage de l'hydroquinone — Méthode spectrophotométrique

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Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Photographic processing waste — Determination of hydroquinone content — Spectrophotometric method

1 Scope and field of application

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

This method can be applied to samples containing hydroquinone in the concentration range of 200 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 References

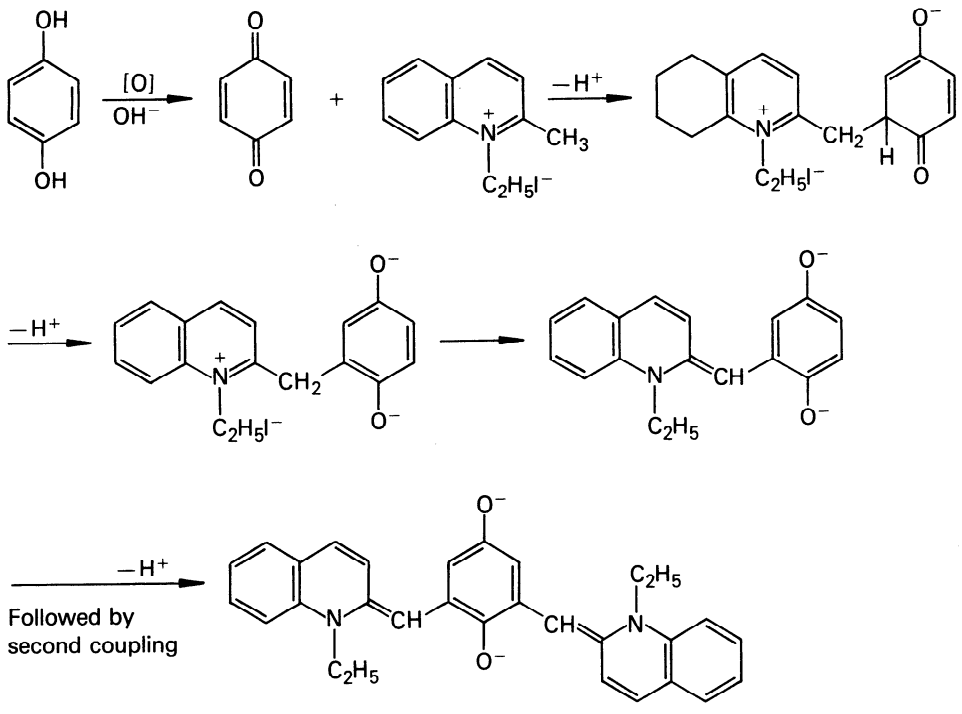
ISO 648, *Laboratory glassware — One-mark pipettes.*

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

3 Principle

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

4 Reactions



5 Reagents

WARNING — Reagents shall be handled in accordance with the health and safety precautions as shown on containers or as given in other sources of such information. The discharge of reagents shall conform to applicable environmental regulations.

All pipette operations shall be carried out with a pipette bulb or plunger pipette. Never pipette by mouth.

Reagents used in the tests shall be certified reagent grade chemicals of a purity acceptable for the analysis.

The acids and ammonia solution referred to in all the instructions shall be of full strength unless dilution is specified. Dilution is specified in terms of molar concentration (molarity) when standardization is required. When dilution is indicated as $(1 + x)$ it means that 1 volume of the reagent or original strength 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.

5.1 Sodium hydroxide, 1 mol/l solution.

DANGER: CORROSIVE, causes burns. Avoid contact with eyes, skin and clothing.

Dissolve 20,0 g of sodium hydroxide in 400 ml of water, cool then transfer to a 500 ml one-mark volumetric flask and dilute to the mark with water.

5.2 Sodium hydroxide, 0,25 mol/l solution.

DANGER: CORROSIVE, causes burns. Avoid contact with eyes, skin and clothing.

Dilute 50 ml of the 1 mol/l sodium hydroxide solution (5.1) to 200 ml in a 200 ml one-mark volumetric flask.

5.3 Citric acid, buffer solution.

Dissolve 53 g of citric acid monohydrate ($C_6H_8O_2 \cdot H_2O$) in about 70 ml of the 0,25 mol/l sodium hydroxide solution (5.2). Transfer to a 100 ml one-mark volumetric flask and dilute to the mark with the 0,25 mol/l sodium hydroxide solution.

5.4 Water/methanol mixture.

DANGER: MAY BE FATAL if swallowed. Harmful if inhaled.

Mix 60 ml of water with 90 ml of methanol (5.11) and cool.

5.5 1-Ethylquinaldinium iodide,[1] 2 g/l solution.

Dissolve 0,2 g of 1-ethylquinaldinium iodide in 100 ml of the water/methanol mixture.

Store in a dark glass bottle, and discard after 24 h.

5.6 1-Pentanol ($CH_3CH_2CH_2CH_2CH_2OH$).

DANGER: FLAMMABLE. Keep away from heat, sparks, and open flame. Use with adequate ventilation.

5.7 Hydrochloric acid, diluted 1 + 4.

DANGER: CORROSIVE, causes burns. Avoid contact with eyes, skin and clothing.

Dilute 40 ml of concentrated hydrochloric acid to 200 ml.

5.8 Hydrochloric acid, 0,01 mol/l solution.

DANGER: CORROSIVE, causes burns. Avoid contact with eyes, skin and clothing.

Add 1,65 ml of concentrated hydrochloric acid to 2 litres of water and mix.

5.9 Hydroquinone [$C_6H_4(OH)_2$], stock solution.

Dissolve 100 mg of hydroquinone in about 800 ml of 0,01 mol/l hydrochloric acid (5.8). Transfer to a 1 000 ml one-mark volumetric flask and dilute to the mark with the same hydrochloric acid.

Store in a dark glass bottle. Prepare a fresh solution each day.

1 ml of this stock solution contains 100 µg of hydroquinone.

5.10 Hydroquinone, standard solution.

Pipette 10 ml of the hydroquinone stock solution (5.9) into a 200 ml one-mark volumetric flask and dilute to the mark with 0,01 mol/l hydrochloric acid (5.8). Store in a dark glass bottle.

Prepare a fresh solution each day.

1 ml of this standard solution contains 5 µg of hydroquinone.

5.11 Methanol (CH_3OH).

DANGER:

— MAY BE FATAL if swallowed. Harmful if inhaled.

— FLAMMABLE. Keep away from heat, sparks, and open flame. Use with adequate ventilation.

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

6 Apparatus

All glassware should be cleaned with hot 1 mol/l hydrochloric acid and rinsed thoroughly before use.

DANGER: CORROSIVE, causes burns. Avoid contact with eyes, skin and clothing.

6.1 Separatory funnels, of capacity 125 ml.

1) Details on the availability of this solution may be obtained from the ISO Central Secretariat.

6.2 One-mark volumetric flasks, of capacity 25 ml, 100 ml, 200 ml, 500 ml and 1 000 ml, conforming to class A of ISO 1042 where applicable.

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

6.4 Micro pipette, of capacity 0,1 ml.

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

7 Sampling and sample preservation

Hydroquinone 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.

8 Procedure

8.1 Extraction

Place 50 ml of sample, or an aliquot diluted to 50 ml, into a 150 ml beaker and adjust the pH to between 4 and 5 with diluted 1 + 4 hydrochloric acid (5.7) or 1 mol/l sodium hydroxide solution (5.1). The mass of hydroquinone should not exceed 200 µg per analysis.

Transfer to a separatory funnel (6.1) and by means of a graduated pipette (6.3) add 1,0 ml of the citric acid buffer solution (5.3), followed by 12,0 ml of the 1-pentanol (5.6). Shake the funnel 50 times then wait 5 min for the liquids to separate. Draw off the aqueous layer (the lower one) and collect in a second 125 ml separatory funnel (6.1). Add to this second funnel 12,0 ml of 1-pentanol, shake the funnel 50 times then wait 5 min for the liquids to separate. Discard the lower aqueous layer.

Add to the first funnel 10,0 ml of water, shake 20 times, allow to separate for 5 min 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 layer from the first funnel to a dry 25 ml one-mark volumetric flask (6.2). Rinse the second funnel into the first with 1,5 ml of 1-pentanol 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.

8.2 Colour development and spectrophotometric measurements

Pipette 10,0 ml of the combined 1-pentanol extract into a 25 ml one-mark volumetric flask (6.2). Add 10,0 ml of the 1-ethylquinaldinium iodide solution (5.5) and 0,10 ml of the 1 mol/l sodium hydroxide solution (5.1). Dilute to the mark with the water/methanol reagent (5.4) and mix well. Prepare a blank solution using 10 ml of 1-pentanol 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 and then measure their absorbances against distilled water at a wavelength of 675 nm, using two matched cells of optical path length 1 cm and a spectrophotometer (6.5) with a tungsten light source. Samples having absorbances above 0,9 shall be diluted with a mixture of 50 % 1-pentanol, 30 % methanol and 20 % water by volume. Dilution of the sample should not be attempted if the initial absorbance was greater than 2,5. Carry out instead another extraction using a smaller sample.

8.3 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 (5.10). 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.

9 Expression of results

The hydroquinone content, expressed in milligrams per litre, is given by the formula

$$\frac{2{,}5\,(A_s - A_b)\,F}{V}$$

where

A_s is the absorbance of the sample at 675 nm;

A_b is the absorbance of 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 effluent sample taken (usually 50 ml).

10 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.

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