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ISO RECOMMENDATION

R 423

SPECIFICATION FOR PHOTOGRAPHIC GRADE HYDROQUINONE

1st EDITION

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Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 423, *Specification for Photographic Grade Hydroquinone*, was drawn up by Technical Committee ISO/TC 42, *Photography*, the Secretariat of which is held by the American Standards Association, Inc. (ASA).

Work on this question by the Technical Committee began in 1956 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In August 1961, this Draft ISO Recommendation (No. 426) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 423 March 1965

SPECIFICATION FOR PHOTOGRAPHIC GRADE HYDROQUINONE

1. SCOPE

This ISO Recommendation is one of a series to establish criteria of purity of chemicals suitable for processing photographic materials. A " photographic grade " chemical is one which meets purity requirements as described.

This specification states the purity requirements and test methods for photographic grade hydroquinone ($C_6H_4(OH)_2$) (p-dihydroxybenzene).

2. PHYSICAL APPEARANCE

Hydroquinone is in the form of white crystals or crystalline powder.

3. SUMMARY OF REQUIREMENTS

Assay: 99.0 per cent minimum, 101.0 per cent maximum.
Ash: 0.08 per cent maximum.
Heavy metals (as Pb): 0.002 per cent maximum.
Iron (Fe): 0.002 per cent maximum.
Resorcinol: 0.1 per cent maximum.
Identity: To pass melting point test. The infra-red identity test may be used as a supplemental method.
Solubility in dilute acetic acid: To pass test.

4. ASSAY

(99.0 per cent minimum, 101.0 per cent maximum)

4.1 **Reagent: Standard cerate solution.** Mix 52 ± 2 g of ammonium ceric nitrate * with 27 ml of sulphuric acid in a 600 ml beaker with mechanical stirring. Cautiously add distilled water in 100 ml portions, with mechanical stirring, allowing 2 to 3 min between each portion. Continue the addition of water until the cerate is completely dissolved. Dilute to 1 litre with distilled water and mix well.

4.1.1 *Standardization of cerate solution.* Place about 0.2 g of primary standard dry arsenic trioxide on a 25 mm diameter watch glass and weigh accurately. Transfer the watch glass and contents to a 250 ml conical flask. Add 15 ml of 10 per cent sodium hydroxide solution, and warm the mixture gently. When solution is complete, cool to room temperature and add 25 ml of dilute sulphuric acid (1 + 5). Dilute to 100 ml with distilled water. As catalyst, add 0.15 ml of 0.01 M osmium tetroxide (prepared by dissolving 0.25 g of osmium tetroxide in 100 ml of approximately 0.1 N sulphuric acid) and add 1 drop of ferroin indicator.

(CAUTION: Osmium tetroxide is poisonous—avoid contact).

Titrate the arsenic trioxide solution with the cerate solution to be standardized, until the reddish-orange colour changes to colourless or very pale blue. A sluggish end-point indicates insufficient osmium tetroxide; up to 0.7 ml may be required as the solution ages.

$$\frac{\text{mass of } As_2O_3 \times 1000}{\text{millilitres of cerate solution} \times 49.45} = \text{normality of cerate solution}$$

* Reagents used in making the tests should be recognized reagent grade chemicals normally used for careful analytical work. In all the directions, the acids and ammonium hydroxide referred to should be of full strength, unless dilution is specified. Dilution is specified in terms of normality, when standardization of the reagent is required. When dilution is indicated as (1+x), it means 1 volume of the reagent or strong solution diluted with x volumes of distilled water.

4.2 Procedure

Place about 0.25 g of the sample on a 25 mm diameter watch glass and weigh accurately. Transfer the watch glass and sample to a 250 ml conical flask containing 100 ml of distilled water and 10 ml of approximately 0.1 N sulphuric acid. Dissolve the sample, add 3 drops of diphenylamine indicator (prepared by dissolving 1 g of diphenylamine in 100 ml of sulphuric acid), and titrate with the standard cerate solution to a red-violet end-point.

1 ml 0.1 N cerate = 0.0055 g hydroquinone

5. ASH

(0.08 per cent maximum)

Incinerate 5 ± 0.1 g of the sample in a tared platinum crucible and then ignite the residue at 600 ± 25 °C for 4 hours. Cool in a desiccator and weigh. The residue mass should be not more than 0.004 g.

NOTE.—Save the residue for the heavy metals and iron tests.

6. HEAVY METALS (as Pb)

(0.002 per cent maximum)

Prepare a 25 ml heavy metals test control containing 0.10 mg of lead ion and a 25 ml iron test control containing a soluble iron salt equivalent to 0.10 mg of iron (see section 7). Dissolve the residue from the ash test (see section 5) in 0.5 ml of hydrochloric acid, warming if necessary, and transfer the solution (with washing) to a 100 ml beaker. Treat both test controls and the sample solution in the same manner. Add 2 drops of p-nitrophenol indicator (0.25 per cent aqueous solution) and then add dilute ammonium hydroxide (1 + 9), dropwise, until the solution turns yellow. Add dilute hydrochloric acid (1 + 99), dropwise, until the solution becomes colourless and then add 2.5 ml excess. Dilute to 50 ml with distilled water. To 20 ml aliquots of both the heavy metals test control and the sample solution (save the iron test control and the balance of the sample solution for the iron test in clause 7.2), add 5 ml of hydrogen sulphide water, dilute to 50 ml with distilled water and mix well. Any colour produced in the sample solution should be not stronger than that produced in the heavy metals test control. Use Nessler tubes for comparison.

7. IRON (Fe)

(0.002 per cent maximum)

7.1 Reagents

7.1.1 *pH5 acetate buffer*. Add 23 g of anhydrous sodium acetate to 58 ml of 2 M acetic acid and dilute to 1 litre with distilled water. Adjust the final pH of the solution to 5.0 ± 0.1 with glacial acetic acid or 10 per cent sodium hydroxide solution.

7.1.2 *o-phenanthroline (1,10-phenanthroline) mixture*. Thoroughly mix equal parts of 0.1 per cent o-phenanthroline (aqueous solution), 10 per cent hydroxylamine hydrochloride solution and pH 5 acetate buffer.

7.2 Procedure

To 20 ml aliquots of the iron test control and the sample solution as prepared in section 6, add 5 ml of the o-phenanthroline mixture, mix well and let stand for 10 min. Dilute to 50 ml with distilled water and mix well. Any colour produced in the sample solution should be not stronger than that produced in the iron test control. Use Nessler tubes for comparison.