INTERNATIONAL STANDARD

ISO 10349-5

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Photography — Photographic-grade chemicals — Test methods —

Part 5:
Determination of heavy metals and iron content

Photographie — Produits chimiques de qualité photographique — Méthodes d'essai —

Partie 5: Détermination des teneurs en métaux lourds et en fer

Reference number
ISO 10349-5:1992(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 10349-5 was prepared by Technical Committee ISO/TC 42, *Photography*.

ISO 10349 consists of the following parts, under the general title *Photography — Photographic-grade chemicals — Test methods*:

— *Part 1: General*

— *Part 2: Determination of matter insoluble in water*

— *Part 3: Determination of matter insoluble in ammonium hydroxide solution*

— *Part 4: Determination of residue after ignition*

— *Part 5: Determination of heavy metals and iron content*

— *Part 6: Determination of halide content*

— *Part 7: Determination of alkalinity or acidity*

— *Part 8: Determination of volatile matter*

— *Part 9: Reaction to ammoniacal silver nitrate*

— *Part 10: Determination of sulfide content*

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— *Part 11: Determination of specific gravity*

— *Part 12: Determination of density*

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Photography — Photographic-grade chemicals — Test methods —

Part 5:
Determination of heavy metals and iron content

1 Scope

This part of ISO 10349 specifies a general test method for the determination of the heavy metals and/or iron content of photographic-grade chemicals.

NOTE 1 If the analysis concerns only heavy metals or iron, then only that diluted standard solution (8.1) need be prepared.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10349. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10349 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 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.*

3 Hazards

See ISO 10349-1 for general hazard warnings and for details of the hazard code system used in this part of ISO 10349.

4 Reagents

See ISO 10349-1 for general requirements.

4.1 Heavy-metals standard (0,010 mg Pb/ml).

Dissolve 1,83 g of lead acetate trihydrate [$Pb(CH_3COO)_2 \cdot 3H_2O$] (DANGER: << S >>) in 500 ml of water in a 1 litre one-mark volumetric flask. Make up to the mark with water, stopper the flask and invert it four or five times for proper mixing. Pipette a 10 ml aliquot of this solution into another 1 litre one-mark volumetric flask. Make up to the mark with water and mix well. Label this solution "heavy-metals standard".

4.2 Iron standard (0,010 mg Fe/ml).

Dissolve 0,70 g of ferrous ammonium sulfate hexahydrate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] in 500 ml of water in a 1 litre one-mark volumetric flask. Add 20 ml of dilute sulfuric acid (1 + 15)[1] and then make up to the mark with water. Stopper the flask and mix well. Pipette a 10 ml aliquot of this solution into a 100 ml one-mark volumetric flask, make up to the mark with water, and mix well. Label this solution "iron standard".

1) This can be prepared from sulfuric acid, $\rho \approx 1{,}84$ g/ml (DANGER: << C >>).

4.3 *p*-Nitrophenol indicator

Dissolve 2,5 g of *p*-nitrophenol in 500 ml of water in a 1 litre one-mark volumetric flask. Make up to the mark with water, stopper, and mix well.

4.4 Hydrochloric acid solution (1 + 3).

Slowly add 25 ml of hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: < C > < B >) to 75 ml of water.

4.5 Dilute hydrochloric acid solution (1 + 99).

Slowly add 10 ml of hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: < C > < B >) to 990 ml of water.

4.6 Ammonium hydroxide solution (1 + 9).

Slowly add 10 ml of ammonium hydroxide, $\rho \approx 0{,}91$ g/ml (DANGER: < C > < B >) to 90 ml of water.

4.7 Sodium sulfide solution

Rinse several crystals of sodium sulfide nonahydrate ($Na_2S{\cdot}9H_2O$) (DANGER: < S > < B >) with water and blot dry on a paper towel. Dissolve 5,0 g of these crystals in 100 ml of water.

4.8 Acetate buffer solution, pH 5.

Prepare an acetic acid solution (1 + 8) by slowly adding 10 ml of glacial acetic acid (DANGER: < C > < B >) to 80 ml of water. Dissolve 23 g of anhydrous sodium acetate in 58 ml of this acetic acid solution (1 + 8).

4.9 1,10-Phenanthroline reagent

Thoroughly mix equal volumes of an aqueous solution of 1,10-phenanthroline (1,0 g/l) and an aqueous solution of hydroxylamine hydrochloride (100 g/l) and acetate buffer (4.8).

5 Apparatus

See ISO 10349-1 for requirements for glassware.

5.1 Two matched Nessler colour-comparison cylinders, each with a capacity of 50 ml.

6 Sampling

See ISO 10349-1.

7 Preparation of test samples

Samples shall be prepared by one of the methods given in 7.1 to 7.3.

7.1 Dilution of the residue after ignition

Take the residue remaining after ignition (see ISO 10349-4) and dissolve it in 2 ml of hydrochloric acid (1 + 3) (4.4). Rinse with water into a 100 ml beaker and dilute to 25 ml.

7.2 Dilution of sample with water

Weigh the test portion specified in the appropriate International Standard, transfer it to a 100 ml beaker and dissolve it in 25 ml of water.

7.3 Dilution of sample with water after treatment with acid

Weigh the test portion specified in the appropriate International Standard, transfer it to a 100 ml beaker and dissolve it in 25 ml of water. Slowly add 15 ml of hydrochloric acid, $\rho \approx 1{,}18$ g/ml (< C > < B >), and evaporate to dryness on a steam bath. Cool, then redissolve the residue in 25 ml of water.

8 Procedure

8.1 Preparation of standard solutions

Prepare standard solutions by the method given in 8.1.1 or 8.1.2.

8.1.1 Dilution with water

Pipette the volume of heavy metals standard (4.1) specified in the appropriate International Standard into a 100 ml beaker and bring the volume to 25 ml with water. Into a second 100 ml beaker, pipette the volume of iron standard (4.2) specified in the appropriate International Standard, and bring the volume to 25 ml with water.

8.1.2 Treatment with acid

Pipette the volume of heavy-metals standard (4.1) specified in the appropriate International Standard into a 100 ml beaker. Into a second 100 ml beaker, pipette the volume of iron standard (4.2) specified in the appropriate International Standard. Add 15 ml of hydrochloric acid, $\rho \approx 1{,}18$ g/ml (< C > < B >), to each beaker and evaporate to dryness on a steam bath. Redissolve each residue in 25 ml of water.

8.2 Preconditioning of solutions

Treat the test solution prepared in accordance with clause 7 and the two standard solutions prepared in 8.1 as follows. Add 2 drops of *p*-nitrophenol indicator (4.3). If the solution is colourless, add dilute ammonium hydroxide (1 + 9) (4.6) until the solution first turns yellow. Add dilute hydrochloric acid (1 + 99) (4.5) until the solution becomes colourless, then add 1 ml ex-

cess. Dilute the test solution and standard solutions each to 50 ml with water and mix well.

8.3 Determination

8.3.1 Test for heavy metals (expressed as Pb)

To each of 20 ml of the preconditioned test solution and 20 ml of the diluted heavy metals standard (4.1) prepared in accordance with 8.1, add 10 ml of sodium sulfide solution (4.7), dilute to 50 ml with water, mix well, and transfer to the Nessler colour-comparison cylinders (5.1).

Any colour produced in the test solution shall not exceed that produced in the heavy-metals standard.

8.3.2 Test for iron

To each of 20 ml of the preconditioned test solution (8.2) and 20 ml of the iron standard (4.2) prepared in accordance with 8.1, add 5 ml of 1,10-phenanthroline reagent (4.9), dilute to 50 ml with water, mix well, and transfer to the Nessler colour-comparison cylinders[2].

Any colour produced in the test solution shall not exceed that produced in the iron standard.

9 Test report

The test report shall specify the method used and the test result obtained.

It shall also mention all operating details not specified in this part of ISO 10349, or regarded as optional, together with details of any incidents which may have influenced the test result.

The test report shall include all information necessary for the complete identification of the sample.

2) Colour comparisons may also be made with a spectrometer.

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Descriptors: photography, photographic materials, photographic chemicals, tests, chemical analysis, determination of content, heavy metals, iron, comparison analysis.

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