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Glass and glassware — Analysis of extract solutions —

Part 4:
Determination of aluminium oxide by molecular absorption spectrometry

Verre et verrerie — Analyse des solutions d'attaque —

Partie 4: Dosage de l'oxyde d'aluminium par spectrométrie d'absorption moléculaire

Reference number
ISO 10136-4:1993(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 10136-4 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, Sub-Committee SC 5, *Quality of glassware*.

ISO 10136 consists of the following parts, under the general title *Glass and glassware — Analysis of extract solutions*:

- *Part 1: Determination of silicon dioxide by molecular absorption spectrometry*
- *Part 2: Determination of sodium oxide and potassium oxide by flame spectrometric methods*
- *Part 3: Determination of calcium oxide and magnesium oxide by flame atomic absorption spectrometry*
- *Part 4: Determination of aluminium oxide by molecular absorption spectrometry*
- *Part 5: Determination of iron(III) oxide by molecular absorption spectrometry and flame atomic absorption spectrometry*
- *Part 6: Determination of boron(III) oxide by molecular absorption spectrometry*

Annex A of this part of ISO 10136 is for information only.

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Introduction

The amounts of aluminium oxide (Al_2O_3) extracted from most glasses by methods normally used for measuring the hydrolytic resistance are extremely small, therefore very sensitive analytical methods are needed for its determination. Technical Committee 2, Chemical Durability and Analysis, of the International Commission on Glass (ICG), examined the problem (see [6] in annex A) and decided that the most widely applicable approach would probably be via a molecular absorption spectrometic method, although flame atomic absorption spectrometry was also studied.

A considerable number of reagents are available for producing colour complexes with aluminium but none is particularly outstanding. Three were selected for the round-robin examination in which eleven laboratories collaborated and they were chromazurol S, eriochrome cyanine and 8-hydroxyquinoline. The results obtained suggested that the chromazurol S method was the most satisfactory from an operational point of view and, at the low concentrations of aluminium oxide expected in extract solutions, is probably as accurate as any other generally available method.

In a later investigation, the determination of very low concentrations of aluminium oxide in extract solutions by flame atomic absorption spectrometry was examined. The results clearly indicated that the proposed flame atomic absorption spectrometric method is not suitable for the determination of low concentrations of aluminium oxide in extract solutions.

The results of investigations on turbidities, especially in grain test solutions, showed that acidification to dissolve possible hydroxides and/or carbonates is necessary prior to the analytical determination. This is achieved by using spectroscopic buffer solutions, which are normally strongly acidic, or by addition of acids.

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Glass and glassware — Analysis of extract solutions —

Part 4:
Determination of aluminium oxide by molecular absorption spectrometry

1 Scope

This part of ISO 10136 specifies an analytical procedure, using molecular absorption spectrometry, for measuring the concentrations of aluminium, expressed as its oxide (Al_2O_3), released into extract solutions during hydrochloric resistance test procedures.

This part of ISO 10136 applies to the analysis of extract solutions obtained from any kind of glass or glassware, including laboratory and pharmaceutical ware made, for example, from borosilicate glass (such as borosilicate glass 3.3 according to ISO 3585), neutral glass, or soda-lime-silica glass as defined in ISO 4802[3][4], food and drink packaging ware, tableware and kitchenware. The extract solution may be obtained from glass articles, for example according to ISO 4802 or from glass as material, for example when tested according to ISO 719[1] or ISO 720[2]. In addition, it may be applied to the extract solutions produced by any method for measuring the hydrolytic resistance of glass or glassware.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10136. 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 10136 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 385-2:1984, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

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

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

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

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

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3585:1991, *Borosilicate glass 3.3 — Properties.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3819:1985, *Laboratory glassware — Beakers.*

ISO 6955:1982, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary.*

3 Definitions

For the purposes of this part of ISO 10136, the following definitions apply.

3.1 extract solution: The aqueous solution obtained from the reaction of glass with water under specific conditions.

3.2 sample measuring solution: The solution actually used for measuring the concentration of the analyte. It may be the undiluted, diluted or modified extract solution.

3.3 analyte: The element or constituent to be determined.

3.4 stock solution: A solution of appropriate composition containing the analyte, expressed as its oxide, in a known but high concentration.

3.5 standard solution: A solution containing the analyte, expressed as its oxide, in a known concentration suitable for the preparation of reference or calibration solutions.

3.6 set of calibration solutions; set of reference solutions: A set of simple or synthetic reference solutions having different analyte concentrations. The zero member is, in principle, the solutions having zero concentration of the analyte. [ISO 6955]

3.7 molecular absorption spectrometry (MAS): A technique for determining the concentration of an analyte in solution by measuring the optical density of a colour complex of the analyte.

3.8 blank test solution: A solution prepared in the same way as the sample measuring solution but so that it does not contain the analyte to be determined.

4 Principle

Complexing of the extract solution to be analysed with chromazurol S. Measurement of the optical density of the resulting solution by means of a molecular absorption spectrometer at 545 nm using 10 mm optical cells.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and grade 1 or grade 2 water specified in ISO 3696.

When acids and ammonium hydroxide are specified only by name or chemical formula, the concentrated reagent is intended. The concentrations of diluted acids or ammonium hydroxide are specified as a ratio, stating the number of volumes of the concentrated reagent to be added to a given number of volumes or water. For example, 1 + 3 means that 1 volume of the concentrated reagent shall be diluted with 3 volumes of water.

Commercially available standard solutions for colorimetry may be used for the preparation of stock or standard solutions.

5.1 Aluminium (Al), foil.

5.2 Aluminium oxide, stock solution.

Transfer 264,7 mg of aluminium (5.1), cut into small pieces, to a 250 ml beaker (6.4) containing 5 ml of hydrochloric acid (5.9) and 5 ml of water. Add 1 drop of copper solution (5.7) to aid dissolution and allow to dissolve with the beaker partially immersed in a cold water bath. Heat to boiling and boil for 1 min. Cool, transfer the solution to a 500 ml one-mark volumetric flask (6.5), make up to the mark with water and mix.

1 ml of this stock solution contains 1,0 mg of Al_2O_3.

5.3 Aluminium oxide, standard solution.

Using a one-mark pipette (6.7), transfer 5,00 ml of the aluminium oxide stock solution (5.2) to a 1 000 ml one-mark volumetric flask (6.5). Add 10 ml of hydrochloric acid (5.9.1), make up to the mark with water and mix.

1 ml of this standard solution contains 5 µg of Al_2O_3.

5.4 Ascorbic acid ($C_6H_8O_6$), 16 g/l solution freshly prepared immediately before use.

5.5 Chromazurol S ($C_{23}H_{13}Cl_2Na_3O_9S$), solution.

Dissolve 0,10 g of chromazurol S in ethanol (5.8), transfer to a 250 ml one-mark volumetric flask (6.5), make up to the mark with ethanol and mix.

5.6 Copper (Cu), foil.

5.7 Copper, (Cu^{2+}), 1 g/l solution.

Transfer 100 mg of copper (5.6), cut into small pieces, to a 50 ml beaker (6.4), add 10 ml of hydrochloric acid (5.9.1) and heat to dissolve. Cool, transfer to a 100 ml one-mark volumetric flask (6.5), make up to the mark with water and mix.

5.8 Ethanol (C_2H_5OH).

5.9 Hydrochloric acid (HCl), $\rho = 1{,}19$ g/ml.

5.9.1 Hydrochloric acid, diluted 1 + 1.

5.9.2 Hydrochloric acid, diluted 1 + 24.

5.10 Sodium acetate trihydrate ($CH_3COONa.3H_2O$), buffer solution.

Dissolve 150 g of sodium acetate trihydrate in water, transfer to a 1 000 ml one-mark volumetric flask (6.5), make up to the mark and mix.

Transfer the solution to a stoppered plastics bottle (6.3) for storage.

5.11 Sodium hydroxide, solution, $c(\text{NaOH}) \approx 0{,}1$ mol/l, stored in a plastics bottle (6.3).

6 Apparatus

All laboratory glassware, except pipettes and burettes, shall be made of borosilicate glass, preferably of type 3.3 complying with the requirements in ISO 3585.

Ordinary laboratory apparatus, and

6.1 Molecular absorption spectrophotometer, capable of measuring optical density at 545 nm.

6.2 Optical cells, 10 mm path length.

6.3 Plastics bottles, stoppered, of a suitable capacity e.g. about 1 000 ml.

6.4 Beakers, of a suitable capacity e.g. 100 ml, and complying with the requirements in ISO 3819.

6.5 One-mark volumetric flasks, of a suitable capacity, and complying with the requirements for class A one-mark volumetric flasks in ISO 1042.

6.6 Graduated pipette, of a suitable capacity e.g. 20 ml, and complying with the requirements for class A graduated pipettes in ISO 835-1, ISO 835-2 or ISO 835-3.

6.7 One-mark pipettes, of capacity 10 ml and 50 ml, and complying with the requirements for class A one-mark pipettes in ISO 648.

6.8 Burettes, of a suitable capacity e.g. 10 ml, and complying with the requirements for class A burettes in ISO 385-2.

6.9 Balance, with a discrimination of 0,1 mg.

6.10 pH-meter, with glass and calomel electrodes, or combined electrodes.

6.11 Filter papers, of the ashless type washed twice with acid, and designated as follows:

"open" or "coarse" will have a porosity generally used for filtering aluminium hydroxide;

"medium" will have a porosity generally used for filtering calcium oxalate;

"close" or "fine" will have a porosity generally used for filtering barium sulfate.

7 Sampling and samples

The sample for analysis shall be the extract solution produced in any hydrolytic resistance test procedure.

8 Procedure

8.1 Preparation and measurement of the sample measuring solution and the blank test solution

8.1.1 Sample measuring solution

Using a one-mark pipette (6.7), transfer a 25 ml aliquot of the extract solution to a beaker (6.4) and acidify, under control of a pH-meter (6.10), with hydrochloric acid (5.9.2) to a pH-value of about 3. Then heat till boiling. Using an appropriate filter (6.11), filter the solution into another beaker (6.4). Wash the filter carefully with small portions of warm water (maximum 4 ml). Cool and then adjust the pH-value to about 5 with sodium hydroxide solution (5.11). Transfer the solution to a 50 ml one-mark volumetric flask (6.5). Rinse with 5 ml of ascorbic acid solution (5.4) in total, then mix and allow to stand for 10 min. Add 7 ml of acetate buffer solution (5.10) and 3 ml of chromazurol S solution (5.5), make up to the mark with water, mix and allow to stand for 10 min.

8.1.2 Blank test solution

Prepare the blank test solution using a volume of water equal to the volume of extract solution used to prepare the sample measuring solution.

8.1.3 Measurement of optical density

Measure the optical density of the sample measuring solution at 545 mm (see 6.1) using 10 mm optical cells (6.2), and also of the blank test solution with the zero member (see 3.6) as reference.

8.2 Preparation of the calibration graph

Using a burette (6.8), transfer accurately measured volumes of the aluminium oxide standard solution (5.3) to separate 100 ml one-mark volumetric flasks (6.5), to cover the range 0 to 25 µg Al_2O_3. Dilute, where necessary, to about 5 ml with water, add 10 ml of ascorbic acid solution (5.4) and proceed as described in 8.1.1 using 14 ml of acetate buffer solution (5.10) and 6 ml of chromazurol S solution (5.5).

Plot the optical densities against mass of Al_2O_3 to produce the calibration graph.

9 Expression of results

Determine the mass of aluminium oxide (Al_2O_3) in the sample measuring solution (8.1.1) and in the blank test solution (8.1.2) from the calibration graph (8.2). Subtract, and calculate the concentration of aluminium oxide in the extract solution and express as micrograms of Al_2O_3 per millilitre of extract solution.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 10136;

b) an identification of the extracted samples;

c) a reference to the hydrolytic resistance test method used (see annex A) to produce the extract solution;

d) the results obtained, expressed as micrograms of Al_2O_3 per millilitre of extract solution;

e) any unusual features noted during the determination.

Annex A
(informative)

Bibliography

[1] ISO 719:1985, *Glass — Hydrolytic resistance of glass grains at 98 °C — Method of test and classification.*

[2] ISO 720:1985, *Glass — Hydrolytic resistance of glass grains at 121 °C — Method of test and classification.*

[3] ISO 4802-1:1988, *Glassware — Hydrolytic resistance of the interior surfaces of glass containers — Part 1: Determination by titration method and classification.*

[4] ISO 4802-2:1988, *Glassware — Hydrolytic resistance of the interior surfaces of glass containers — Part 2: Determination by flame spectrometry and classification.*

[5] ISO 6286:1982, *Molecular absorption spectrometry — Vocabulary — General — Apparatus.*

[6] The chemical durability of glass: determination of Aluminium oxide in extract solutions. (A report by Sub-Committee 2 of the International Commission on Glass). Glastechn. Ber., (1978), Vol. **51**, No. 4, pp.75-78.