

INTERNATIONAL
STANDARD

ISO
10136-2

First edition
1993-07-01

Glass and glassware — Analysis of extract solutions —

Part 2:

Determination of sodium oxide and potassium oxide by flame spectrometric methods

iTeh STANDARD PREVIEW
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ISO 10136-2:1993
*Verre et verrerie — Analyse des solutions d'attaque —
Partie 2: Dosage de l'oxyde de sodium et de l'oxyde de potassium par
spectrométrie d'émission de flamme*



Reference number
ISO 10136-2:1993(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 10136-2 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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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

Classifications of glass or glassware, in National or International Standards and in the various pharmacopoeia, have relied for many years on a titration of extract solutions with a diluted mineral acid. Such solutions may contain not only the alkali metal oxides (sodium and potassium), but also the alkaline earth oxides (calcium and magnesium), which are also titrated by acid. Thus, the determination is actually of the total alkalinity of the extract solution and this is calculated as the equivalent mass of sodium oxide. In recent years, with the advent of more modern techniques, such as flame spectrometric methods, these techniques have been applied more and more. Therefore, the specific determination of sodium and potassium in extract solutions is possible, and this is of interest for many general investigations on the durability of glasses.

The procedure was established and applied to simulated or actual durability extract solutions of glasses in an international collaborative study conducted by Technical Committee 2, Chemical Durability and Analysis, of the International Commission on Glass (ICG). In these round robins, up to 21 analysts from 15 laboratories collaborated. The final results of the round robin were that for the very low concentrations in extract solutions, e.g. from borosilicate glasses, no spectrochemical buffer needs to be added, and that for other aqueous extract solutions the addition of only caesium chloride is sufficient (see [5] in annex A).

The flame spectrometric methods described in this part of ISO 10136 are claimed to be satisfactory for determining both alkali metal oxides in durability 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 2:

Determination of sodium oxide and potassium oxide by flame spectrometric methods

1 Scope

This part of ISO 10136 specifies the analytical methods of flame spectrometry, using either atomic emission (also in the filter flame spectrometer technique) or atomic absorption spectrometry, for measuring the concentrations of sodium and potassium, expressed as sodium oxide (Na₂O) and potassium oxide (K₂O), released into extract solutions during hydrolytic 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 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 solution having zero concentration of the analyte. [ISO 6955]

3.7 flame atomic absorption spectrometry (FAAS): A technique for determining the concentration of chemical elements based on the measurement of the absorption of characteristic electromagnetic radiation in a vapour phase in a flame.

3.8 flame emission spectrometry (FES): A method of determining chemical elements based on the measurement of the intensity of characteristic electromagnetic radiation emitted by atoms or molecules in a flame. [ISO 6955]

3.9 filter flame spectrometry (Filter FES): A method equivalent to flame emission spectrometry (FES) (3.8) using filters for selection of the detection lines.

3.10 spectrochemical buffer solution: A solution of a substance or substances added to the sample measuring solution and to the reference solutions in order to reduce interferences during flame spectrometric measurements.

3.11 optimum working range: The range of concentrations of an analyte in solution over which the relationship between absorption (or emission) and concentration is linear.

4 Principle

Spraying of the extract solution to be analysed into the flame of the burner of an emission or absorption spectrometer, if necessary with the addition of a spectrochemical buffer solution, or into the flame of a filter flame spectrometer without this addition. For

FES and FAAS, for the determination of sodium and potassium using their lines at 589,0 nm and 766,5 nm respectively, using a set of reference solutions. For a flame filter spectrometer, using the special filters for the determination of sodium and potassium.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and grade 1 or grade 2 water as 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 of 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 spectrometry may be used for the preparation of stock or standard solutions.

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

5.1.1 Hydrochloric acid, diluted 1 + 1.

5.1.2 Hydrochloric acid, diluted 1 + 12.

5.2 Caesium chloride (CsCl).

5.3 Spectrochemical buffer solution, caesium chloride solution.

Dissolve 40 g of caesium chloride (5.2) in water, add 100 ml of the hydrochloric acid (5.1) and transfer to a 1 000 ml one-mark volumetric flask (6.4). Dilute to the mark with water and mix. Store the spectrochemical buffer solution in a pre-treated borosilicate glass bottle (6.3).

5.4 Sodium chloride (NaCl).

Dry for at least 1 h at 110 °C, allow to cool and store in a desiccator.

5.5 Potassium chloride (KCl).

Dry for at least 1 h at 110 °C, allow to cool and store in a desiccator.

5.6 Sodium oxide, stock solution

Use commercially available standard solution or prepare as follows.

Dissolve 1,885 8 g of the sodium chloride (5.4) in water in a 1 000 ml one-mark volumetric flask (6.4),

make up to the mark and mix. Store this solution in a pre-treated borosilicate glass bottle (6.3).

1 ml of this stock solution contains 1 mg of Na_2O .

5.7 Potassium oxide, stock solution.

Use commercially available standard solutions, or prepare as follows.

Dissolve 1,583 0 g of the potassium chloride (5.5) in water in a 1 000 ml one-mark volumetric flask (6.4), make up to the mark and mix. Store this solution in a pre-treated borosilicate glass bottle (6.3).

1 mg of this solution contains 1mg of K_2O .

5.8 Sodium oxide, and potassium oxide, standard solutions.

Preliminary measurements of the extract solution shall show which range of concentration of both oxides is to be expected. According to the analytical task, prepare standard solutions by diluting appropriate volumes of the stock solutions (5.6 and 5.7) such that, for instance, concentrations of 100 μg Na_2O and 100 μg K_2O per millilitre are obtained, or, since K_2O is very often released in only small amounts, 100 μg Na_2O and 10 μg K_2O per millilitre are obtained.

Store these standard solutions in pre-treated borosilicate glass bottles (6.3).

1 000 ml of each standard solutions shall contain 5 ml of hydrochloric acid (5.1.1).

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 Flame atomic emission or flame atomic absorption spectrometer, equipped with line sources for sodium and potassium, and with gas supplies and burners for air/acetylene or air/propane mixtures.

Or **flame emission filter spectrometer**, equipped with filters for the sodium and potassium lines.

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

6.3 Pre-treated borosilicate glass bottles, of a suitable capacity

Before the reagent solutions are transferred to the bottles, each new bottle shall be pre-treated in the following manner. It shall be filled with hydrochloric acid (5.1.2) and heated almost until boiling in a heating bath for 2 h. The bottle shall then be rinsed with wa-

ter, filled with water and heated as before for two periods of 1 h, using fresh water each time.

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

6.5 One-mark pipettes, of a suitable capacity, and complying with the requirements for class A one-mark pipettes in ISO 648.

6.6 Graduated pipette, of capacity 10 ml, and complying with the requirements for class A graduated pipettes in ISO 835-1, ISO 835-2 or ISO 835-3.

6.7 Burette, of capacity 10 ml, and complying with the requirements for class A burettes in ISO 385-2.

6.8 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 of the sample measuring solution

Choose, according to the volume of the received extract solution, appropriate cleaned dry one-mark volumetric flasks (6.4). Using suitable pipette (6.5 and 6.6), transfer a volume (corresponding to 5 % of the volume of the one-mark volumetric flasks) of the spectrochemical buffer solution (5.3) to the flasks. Make up to the mark with the extract solution, mix well, heat gently and leave to stand for 2 min. Cool with tap water to room temperature and, in every case, filter through a dry filter paper (6.8) that is suitable for retaining turbidity. The filtrate is the sample measuring solution.

If the extract solutions are turbid, homogenize by vigorous shaking. As soon as possible after the larger particles have settled, make up to the marks of the prepared one-mark volumetric flasks with the extract solution and continue as described in the previous paragraph.

8.2 Preparation of sodium oxide and potassium oxide reference solutions

Prepare the reference solutions for establishing the calibration graph by measuring different volumes of a suitable standard solution (5.8), using a suitable burette (6.7), into 100 ml one-mark volumetric flasks (6.4). Add 5 ml of spectrochemical buffer solution (5.3) and make up to the mark with water.

The set of reference solutions should normally cover the optimum working ranges of the specific elements according to the instrument used for the measurement. Typical concentration ranges of the reference solutions and the conditions for use of the instruments are given in table 1.

8.3 Preparation of the calibration graphs

Adjust the instrument (6.1) to the optimum measuring conditions, according to the operating manuals, using the parameters shown in table 1.

Spray the reference solutions (8.2), chosen according to the composition of the sample measuring solutions, into the flame of the instrument in order of increasing concentrations and obtain a set of emission or absorption readings for Na₂O and K₂O, respectively. Spray water to clean the spray chamber, then repeat the spraying procedure to obtain five readings for each concentration of the reference solutions. From the mean values of the readings, plot the graphs of the Na₂O-concentration and the K₂O-concentration respectively against the instrument readings.

8.4 Measurement of the sample measuring solution

Spray the sample measuring solution into the flame of the instrument and obtain the sodium and/or potassium signals respectively. Repeat the spraying procedures, using water to clean the spray chamber between the measurements of different concen-

trations, to obtain five readings for the analytes (Na, K).

NOTE 1 It is advisable to control the constancy of the instrument by spraying one of the reference solutions previously used, preferably one with a similar alkali concentration.

9 Expression of results

9.1 Calculate the mean values, obtained according to 8.4, for the sample measuring solution. Read off the concentration for Na₂O or K₂O respectively for each measured solution from the appropriate calibration graph (8.3). Multiply these results by 1,05 and express the results in micrograms of the individual oxide per millilitre of the extract solution.

9.2 If surfaces of larger glass pieces were extracted, the results can be related to the surface. In this case, measure the total surface area of the glass sample, surface plus edges, to an accuracy of $\pm 2\%$ and calculate the surface related release, B , of the alkali oxide(s), in micrograms per square decimetre, using the equation

$$B = \frac{\rho_{AO} \cdot V}{A}$$

where

ρ_{AO} is the mean value, in micrograms per millilitre, of the measured alkali oxide concentration;

V is the total volume, in millilitres, of the extract solution;

NOTE 2 The total volume of the extract solution is not equal to the volume of the sample measuring solution.

A is the total surface area, in square decimetres, of the extracted glass sample.

Express the results in micrograms of the individual oxide per square decimetre.

Table 1 — Conditions for use of the instruments for measuring sodium (Na) and potassium (K)

Element	Instrument	Usual optimum working range µg/ml	Gas mixture	Detection line nm
Na, K	Filter FES	≤ 10	air/propane	Filters
Na	FES or FAAS	≤ 3	air/acetylene or air/propane	589,0
K	FES or FAAS	≤ 3		766,5

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) a reference to the method of measurement used (flame atomic emission or flame atomic absorption spectrometry, filter flame spectrometry);
- e) a statement whether turbid solutions were measured;
- f) the results obtained, expressed as micrograms of Na₂O and/or K₂O per millilitre of extract solution;
- g) any unusual features noted during the determination.

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