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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Glassware — Hydrolytic resistance of the interior surfaces of glass containers —

Part 2 :
Determination by flame spectrometry and classification

Verrerie — Résistance hydrolytique des surfaces internes des récipients en verre —

Partie 2 : Détermination par spectrométrie de flamme et classification

Reference number
ISO 4802-2 : 1988 (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 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.

International Standard ISO 4802-2 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus.*

International Standards ISO 4802-1 and ISO 4802-2 cancel and replace International Standard ISO 4802 : 1982, of which they constitute a technical revision.

ISO 4802 consists of the following parts, under the general title : *Glassware — Hydrolytic resistance of the interior surfaces of glass containers* :

— *Part 1 : Determination by titration method and classification*

— *Part 2 : Determination by flame spectrometry and classification*

Printed in Switzerland

Introduction

This part of ISO 4802 is largely based on methods of test approved by the International Commission on Glass (ICG), Technical Committee 2, *Chemical Durability and Analysis*, for measuring the hydrolytic resistance of the interior surfaces of glass containers.

This part of ISO 4802 contains a classification related to the classification which is set up in ISO 4802-1 for the titration method.

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Glassware — Hydrolytic resistance of the interior surfaces of glass containers —

Part 2 :
Determination by flame spectrometry and classification

1 Scope

This part of ISO 4802 specifies

a) methods for determining the hydrolytic resistance of the interior surfaces of glass containers when subjected to attack by water at 121 °C ± 1 °C for 60 min ± 1 min. The resistance is measured by determining the amount of sodium and other alkali metal or alkaline earth oxides in the extraction solution using flame atomic emission or absorption spectrometry (flame spectrometry);

b) a classification of glass containers according to the hydrolytic resistance of the interior surfaces determined by the methods specified in this part of ISO 4802.

NOTE — The hydrolytic resistance container class HC obtained by the flame spectrometry is comparable with the class HC obtained according to ISO 4802-1, although the individual test values are not equal.

2 Applicability

This part of ISO 4802 applies to containers, such as bottles, vials, ampoules, flasks, beakers, etc., made for instance from soda-lime-silica glass, whether surface-treated or not, or from borosilicate glass or neutral glass.

This part of ISO 4802 does not apply to double-ended ampoules or to the classification of closed ampoules.

3 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4802. 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 4802 are encouraged to investigate the possibility of applying the most recent editions of the standards shown below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1 : General requirements.*

ISO 385-2 : 1984, *Laboratory glassware — Burettes — Part 2 : Burettes for which no waiting time is specified.*

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

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

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

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

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

4 Definitions

For the purposes of this part of ISO 4802, the following definitions shall apply.

4.1 container : Any article made from borosilicate, neutral or soda-lime-silica glass, such as bottles, vials, ampoules and articles especially intended for laboratory or pharmaceutical use, which is capable of being filled.

4.2 borosilicate glass : A silicate glass containing between 5 % and 13 % (m/m) of boric oxide, having a high thermal shock resistance and a very high hydrolytic resistance due to its composition.

Containers properly made from this glass comply with hydrolytic resistance container class HC 1 of this International Standard.

4.3 neutral glass : A silicate glass containing significant amounts of boric oxide, usually between 5 % and 13 % (m/m), aluminium and/or alkaline earth oxides, and having a very high hydrolytic resistance due to its composition.

Containers properly made from this glass comply with hydrolytic resistance container class HC 1 of this International Standard.

4.4 soda-lime-silica glass : A silicate glass containing up to approximately 15 % (m/m) of alkali metal oxides — mainly sodium oxide — and up to about 15 % (m/m) of alkaline earth oxides, mainly calcium oxide.

Containers made from this glass will have a moderate hydrolytic resistance due to the chemical composition of the glass, and comply with hydrolytic resistance container class HC 3 or hydrolytic resistance container class HC D. After surface treatment (see 4.5), soda-lime-silica glass containers of hydrolytic resistance container class HC 3 will have a very high hydrolytic resistance, due to the treatment, and comply with hydrolytic resistance container class HC 2.

4.5 surface treatment : Treatment of the internal surface of soda-lime-silica glass containers with reagents in order to achieve a de-alkalized surface and to produce a significantly lower release of alkali metal ions (and alkali earth metal ions).

4.6 brimful capacity : The volume of water required to fill a container, placed on a flat, horizontal surface, until the meniscus just touches the strike-plate (see 7.7).

4.7 filling volume : The volume of water to be filled into the test specimen. For vials, bottles and lipped containers, it is defined as 90 % of the brimful capacity. For ampoules, it is defined as the volume up to the height where the body of the ampoule declines to the shoulder (see figure 2).

4.8 vial; phial : Small, flat-bottomed container, made from tubing or from moulded glass; normally thick-walled and with a capacity up to about 50 ml.

NOTE — Vials are normally sealed with a closure made from a material other than glass, and not by flame-sealing.

4.9 bottle : Flat-bottomed container, made from moulded glass; normally thick-walled and with a capacity usually of more than 50 ml.

Bottles may be of circular or other geometric cross-section.

NOTE — Bottles are normally sealed with a closure made from a material other than glass, and not by flame-sealing.

4.10 ampoule : Normally flat-bottomed container, made from thin-walled tubing, and having stems in many different forms.

Ampoules are intended to be closed after filling by flame-sealing. Capacity normally up to 25 ml.

Types : open and closed ampoules (see figure 1).

5 Principle

The methods of test are surface tests normally applied to glass containers as delivered.

Filling of the containers to be tested with specified water to a specified capacity and heating of the containers loosely capped under specified conditions. Measurement of the degree of the hydrolytic attack by flame spectrometric analysis of the extraction solutions.

6 Reagents

During the test, unless otherwise stated, use only reagents of recognized analytical grade.

6.1 Test water, consisting of grade 1 water or grade 2 water, which complies with the requirements specified in ISO 3696.

6.2 Hydrochloric acid, solution, c(HCl) ≈ 2 mol/l.

6.3 Hydrochloric acid, solution, c(HCl) ≈ 6 mol/l (≈ 1 + 1).

6.4 Hydrofluoric acid, c(HF) ≈ 22 mol/l (i.e. ≈ 400 g HF/l solution).

6.5 Distilled water or **water of equivalent purity** (grade 3 water complying with the requirements specified in ISO 3696).

6.6 Spectrochemical buffer solution (caesium chloride solution, CsCl).

Dissolve 80 g of caesium chloride in approximately 300 ml of test water (6.1), add 10 ml of hydrochloric acid (6.3) and transfer to a 1 000 ml volumetric flask (7.3). Dilute to the mark with the test water (6.1) and mix.

6.7 Stock solutions

6.7.1 Dry sodium chloride, potassium chloride and calcium carbonate at 110 °C ± 5 °C for 2 h. Prepare aqueous stock solutions, using the test water (6.1), directly from the chlorides and from the calcium carbonate, after dissolving in just sufficient excess of hydrochloric acid so that all solutions have concentrations of 1 mg/ml, calculated as sodium oxide, potassium oxide and calcium oxide, respectively.

6.7.2 Commercially available standard solutions may also be used.

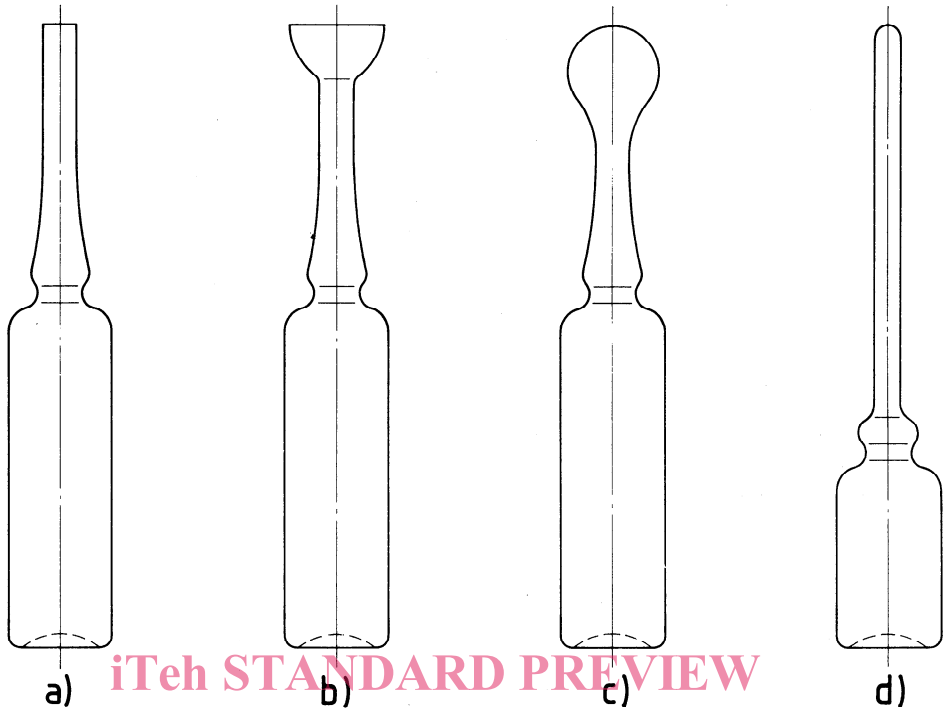

Figure 1 — Examples of typical open [a) and b)] and closed [c) and d)] ampoules

6.8 Standard solutions

6.8.1 Prepare standard solutions by diluting the stock solutions (6.7) with the test water (6.1) to obtain concentrations suitable for establishing the reference solutions in an appropriate manner, e.g. with concentrations of 20 µg/ml of sodium oxide, potassium oxide and calcium oxide, respectively.

6.8.2 Commercially available standard solutions may also be used.

6.9 Reference solutions

The reference solutions for establishing the calibration graph (set of calibration solutions) shall be prepared by diluting suitable concentrated standard solutions (6.8) with the test water (6.1). They should cover normally the optimum working ranges of the specific elements according to the instrument used for the measurement. Typical concentration ranges of the reference solutions are

— for determination by flame atomic emission spectroscopy (FAES) of sodium oxide and potassium oxide : up to 10 µg/ml

— for determination by flame atomic absorption spectrometry (FAAS) of sodium oxide and potassium oxide : up to 3 µg/ml

— for determination by flame atomic absorption spectrometry (FAAS) of calcium oxide : up to 7 µg/ml

For the measurement on containers of hydrolytic resistance container classes HC 1, HC 2 or HC B (borosilicate or highly resistant glasses), the reference solutions shall be used without addition of the spectrochemical buffer solution (6.6).

For the measurement of containers of hydrolytic resistance container classes HC 3 or HC D (soda-lime-silica glasses), the reference solutions shall contain 5 % (V/V) of the spectrochemical buffer solution (6.6).

7 Apparatus

Ordinary laboratory apparatus, and

7.1 Autoclave or **steam sterilizer,** capable of withstanding a pressure of at least 2,5 × 10^5 N/m²* and of carrying out the heating cycle specified in 9.2. It should preferably be equipped with a constant-pressure regulator or other means of maintaining the temperature at 121 °C ± 1 °C. The vessel shall have an internal diameter of at least 300 mm and shall be equipped with a heating device, a thermometer or a calibrated thermocouple, a pressure gauge, a pressure-release safety device, a vent-cock, and a rack for supporting the samples.

The autoclave vessel and ancillary equipment shall be thoroughly cleaned before use.

7.2 Burettes, having a suitable capacity according to the analytical procedure to be used and complying with the requirements specified for class A burettes in ISO 385-2 and made of glass of hydrolytic resistance grain class HGA 1 as specified in ISO 720[1)].

7.3 One-mark volumetric flasks, having a capacity of 1 000 ml and complying with the requirements specified for class A one-mark volumetric flasks in ISO 1042.

7.4 Water bath, capable of being heated to approximately 80 °C.

7.5 Flame atomic absorption (FAAS) or **flame atomic emission (FAES) instrument**

FAAS instruments shall be equipped with line sources for sodium, potassium and calcium; they shall be equipped with air/propane or air/acetylene gas supplies and burners for measuring sodium and potassium, and with a nitrous oxide/acetylene gas supply and burner for measuring calcium.

FAES instruments shall be equipped with air/propane or air/acetylene gas supplies and burners for measuring sodium and potassium.

7.6 Beakers, having a suitable capacity and complying with the requirements specified in ISO 3819.

Before use, each new beaker shall be pretreated by subjecting it to the autoclaving conditions described in 9.2.

7.7 Strike-plates (for measuring the brimful capacity of small bottles and bottles), made of rigid, inert, transparent material of any convenient shape, but with a central hole approximately 5 mm in diameter. The strike-plate shall be large enough to fit snugly on and completely cover the sealing surface of the container the brimful capacity of which is to be measured.

8 Sample preparation

8.1 Sample size

For each container capacity to be tested, the number of containers which are to be measured separately is specified in table 1.

Table 1 — Number of containers for the determination of the hydrolytic resistance by flame spectrometry methods

Capacity [volume corresponding to filling volume (see 8.2)] ml	**Number of containers to be measured separately**	**Additional containers for desired preliminary measurements**
Up to and including 2	20	2
From 2 up to and including 5	15	2
From 5 up to and including 30	10	2
From 30 up to and including 100	5	1
From 100 upwards	3	1

8.2 Determination of the filling volume

8.2.1 Flat-bottomed containers up to 30 ml capacity (except ampoules)

Select six containers at random from the sample lot and remove any dirt or packaging debris by shaking the container. Place each dry container on a flat, horizontal surface and allow to reach a temperature of 22 °C ± 2 °C. Cover each container with a strike-plate (7.7) with the hole positioned approximately central to the mouth of the container. Fill each container with distilled water (6.5) at 22 °C ± 2 °C from a burette (7.2), through the hole in the strike-plate, until the meniscus is just level with the bottom of the hole. Ensure that no air bubbles are trapped at the water/strike-plate interface. Then read the volume of water filled in from the burette to two decimal places. This volume is the brimful capacity of the container.

Calculate the mean value of the results from the six containers. Then calculate 90 % of this mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.2 Flat-bottomed containers of 30 ml capacity and greater

Select six containers (having a capacity less than or equal to 100 ml) or three containers (having a capacity greater than 100 ml) at random from the sample lot and remove any dirt or packaging debris by shaking the containers. Allow the dry containers to reach a temperature of 22 °C ± 2 °C. Cover each container with a suitable strike-plate (7.7) and weigh each of the empty covered containers to the nearest 0,1 g. Remove the strike-plate and fill the container nearly to the top with distilled water (6.5) at 22 °C ± 2 °C, then cover it again with its strike-

* 2,5 × 10^5 N/m² = 0,25 MPa = 2,5 bar

1) Glass of hydrolytic resistance grain class ISO 719 — HGB 1 adequately meets the requirements of class HGA 1 specified in ISO 720.

plate so that the hole is positioned approximately central to the mouth of the container. Continue filling the container with distilled water at 22 °C ± 2 °C from a burette (7.2), through the hole in the strike-plate as described in 8.2.1.

Weigh the filled container with its strike plate to the nearest 0,1 g and calculate the mass of water, in grams, contained within the container.

Calculate the mean value of the results from the six containers and express the result in millilitres of water; this value is the mean brimful capacity of the containers.

Calculate 90 % of this mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.3 Round-bottomed containers (except ampoules)

Select six containers (having a capacity less than or equal to 100 ml) or three containers (having a capacity greater than 100 ml) at random from the sample lot and remove any dirt or packaging debris by shaking the containers. Allow the dry containers to reach a temperature of 22 °C ± 2 °C. Fix each container vertically in an appropriate device and determine the brimful capacity according to 8.2.1 or 8.2.2 respectively.

Then calculate 90 % of the mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.4 Lipped containers

Wrap adhesive plastics tape around the rim of the containers such that the tape around the lip is level with the rim. Weigh the container with its strike-plate (7.7) in place, then fill and reweigh as described in 8.2.2, without taking the strike-plate off.

8.2.5 Ampoules

Place at least six dry ampoules at 22 °C ± 2 °C on a flat, horizontal surface and fill them with the distilled water (6.5), at the same temperature, from a burette (7.2), until the water reaches point A, where the body of the ampoule declines to the shoulder (see figure 2). Read the capacities to two decimal places and calculate the mean value.

This volume, expressed to one decimal place, is the filling volume and shall be filled in ampoules of the same lot.

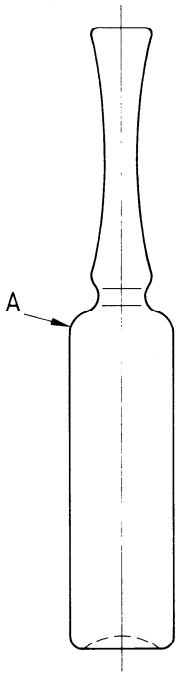

Figure 2 — Filling volume of ampoules (up to point A)

9 Procedure

This procedure shall be completed within one working day.

9.1 Cleaning of samples

This cleaning process shall be completed from the first rinsing in not less than 20 min and not more than 25 min.

Remove from all open samples any packaging debris or dirt which has collected during storage and transport. Rinse each sample thoroughly at least twice with the distilled water (6.5) at ambient temperature, then allow to stand, filled with the distilled water. Immediately before testing, empty the samples, rinse once with the distilled water and then once with the test water (6.1). Allow to drain completely.

Closed ampoules shall be warmed in a water bath or air-oven at about 50 °C for approximately 2 min before opening. They shall not be rinsed before testing.

9.2 Filling and heating

Fill each container, selected for the sample in accordance with 8.1 and cleaned in accordance with 9.1, to the filling volume with the test water (6.1) by means of suitable volumetric measuring devices.

Each container including ampoules shall be loosely capped with an inert material, for example with inverted beakers (7.6) of such a size that the bottoms of the beakers fit snugly down on the rims of the sample, ampoules for example with clean aluminium foil.

NOTE — Ensure that the foil does not release ions to be measured into the test water.