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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

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INTERNATIONAL STANDARD

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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*

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, $c(HCl) \approx 2$ mol/l.

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

6.4 Hydrofluoric acid, $c(HF) \approx 22$ 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.

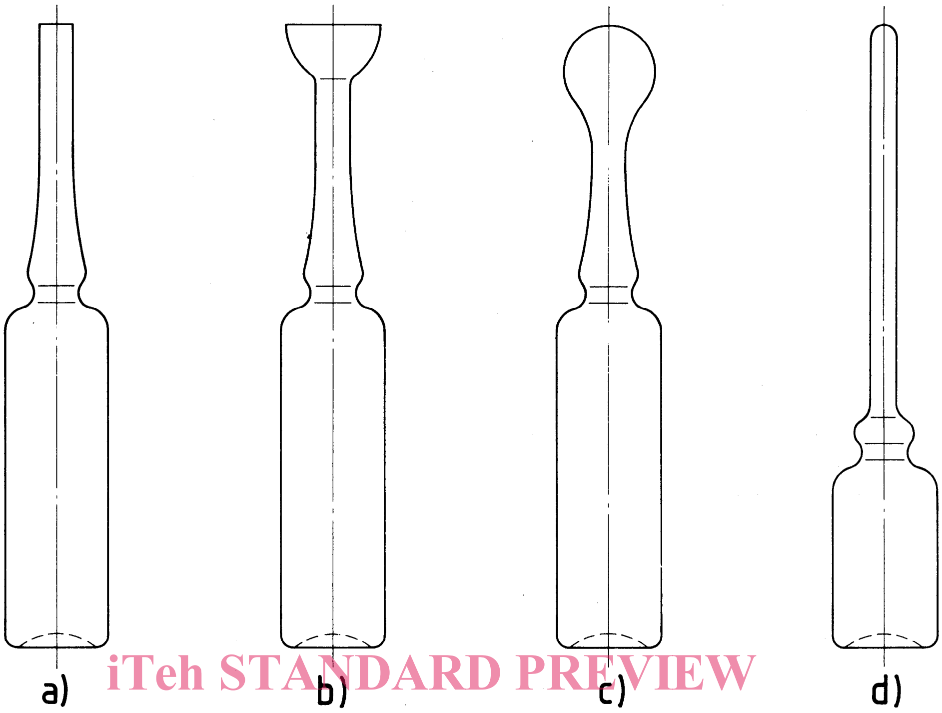

Figure 1 — Examples of typical open [a) and b)] and closed [c) and d)] ampoules

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).