

SLOVENSKI STANDARD

SIST ISO 4802-1:1995

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**Steklovina - Hidrolitska odpornost notranjih površin steklenih posod - 1. del:
Določanje s titracijsko metodo in klasifikacija**

Glassware -- Hydrolytic resistance of the interior surfaces of glass containers -- Part 1:
Determination by titration method and classification

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Verrerie -- Résistance hydrolytique des surfaces internes des récipients en verre -- Partie
1: Détermination par analyse titrimétrique et classification

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Ta slovenski standard je istoveten z: ISO 4802-1:1988

ICS:

81.040.30	Izdelki iz stekla	Glass products
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INTERNATIONAL STANDARD

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Glassware — Hydrolytic resistance of the interior surfaces of glass containers —**Part 1 :****Determination by titration method and classification****(standards.iteh.ai)***Verrerie — Résistance hydrolytique des surfaces internes des récipients en verre —**Partie 1 : Détermination par analyse titrimétrique et classification*

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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 4802-1 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 a method 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.

The European Pharmacopœia Commission has adopted the principle of the determination by titration and has set up a classification for glass containers for injectable preparations which is now included in this part of ISO 4802. In addition, this part of ISO 4802 contains a classification of containers other than for injectable preparations.

According to many results of international interlaboratory tests this part of ISO 4802 specifies the test conditions in more detail than the European pharmacopœia in order to increase the reproducibility of the test results.

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

Part 1 : Determination by titration method and classification

1 Scope

This part of ISO 4802 specifies

a) a method for determining the hydrolytic resistance of the interior surfaces of glass containers when subjected to attack by water at $121\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ for $60\text{ min} \pm 1\text{ min}$. The resistance is measured by titration of a known aliquot portion of the extraction solution produced with hydrochloric acid solution, in which case the resistance is inversely proportional to the volume of acid required;

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

NOTE — The hydrolytic resistance container class HC obtained by titration is comparable with the class HC obtained according to ISO 4802-2, 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.

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

ISO 719 : 1985, *Glass — Hydrolytic resistance of glass grains at $98\text{ }^{\circ}\text{C}$ — Method of test and classification.*

ISO 720 : 1985, *Glass — Hydrolytic resistance of glass grains at $121\text{ }^{\circ}\text{C}$ — Method of test and classification.*

ISO 1773 : 1976, *Laboratory glassware — Boiling flasks (narrow-necked).*

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.

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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

This method of test is a surface test 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 titration 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 2 water, which complies with the requirements specified in ISO 3696 and which has been freed from dissolved gases, such as carbon dioxide, by boiling for at least 15 min in a boiling flask of fused silica or borosilicate glass. The boiling flask shall be pretreated once as specified in 9.2 before it is used for the first time.

When tested immediately before use, this test water shall be neutral to methyl red, i.e. it shall produce an orange-red (not a violet-red or yellow) colour corresponding to $\text{pH } 5,5 \pm 0,1$ when four drops of the methyl red indicator solution (6.5) are added to 50 ml of the test water.

NOTE — The water, so coloured, may also be used as the reference solution (see 9.3).

Such test water can normally be stored for 24 h in a stoppered flask without change of the pH value.

ATTENTION — Grade 1 water, complying with the requirements specified in ISO 3696, will not comply with the above specified pH requirement and shall therefore not be used for the determination by titration.

6.2 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,01 \text{ mol/l}$.

6.3 Hydrochloric acid, solution, $c(\text{HCl}) \approx 2 \text{ mol/l}$.

6.4 Hydrofluoric acid, $c(\text{HF}) \approx 22 \text{ mol/l}$ (i.e. $\approx 400 \text{ g HF/l}$ solution).

6.5 Methyl red, indicator solution.

Dissolve 25 mg of the sodium salt of methyl red ($\text{C}_{15}\text{H}_{14}\text{N}_3\text{NaO}_2$) in 100 ml of the test water (6.1).

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

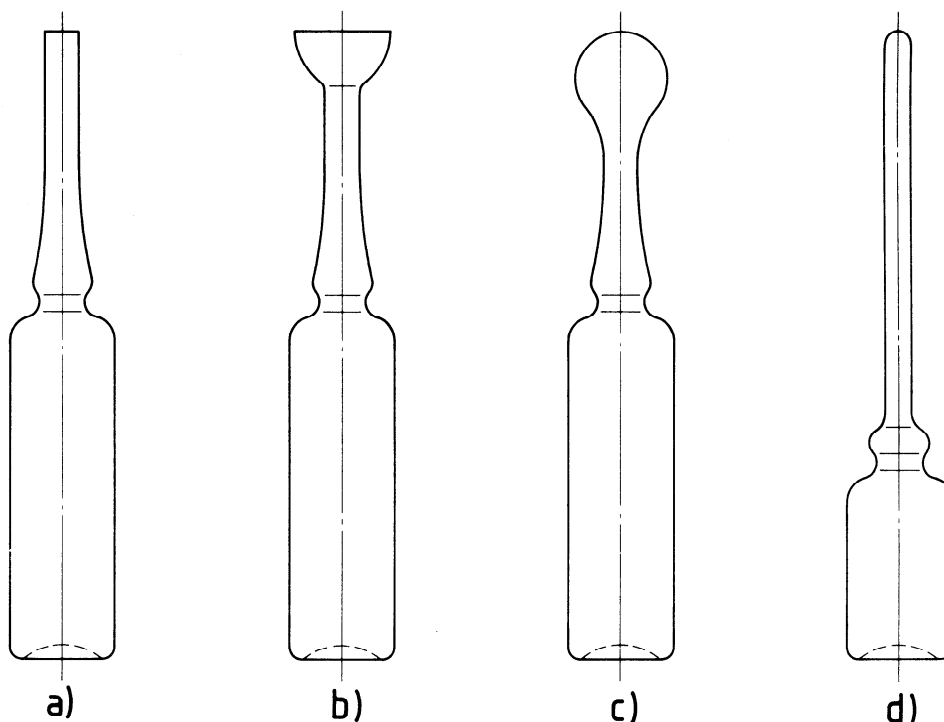


Figure 1 — Examples of typical open [a) and b)] and closed [c) and d)] ampoules

7 Apparatus

Ordinary laboratory apparatus, and

7.1 Autoclave or steam sterilizer, capable of withstanding a pressure of at least $2,5 \times 10^5 \text{ N/m}^2$ * 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 $121^\circ\text{C} \pm 1^\circ\text{C}$. 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 of 50 ml, 25 ml, 10 ml or 2 ml, 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 720¹⁾.

The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid (6.2).

7.3 Conical flasks, having a capacity of 100 ml and 250 ml and complying with the requirements of ISO 1773.

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

7.4 Pipettes, having a suitable capacity and complying with the requirements specified for class A pipettes in ISO 648.

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

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.

* $2,5 \times 10^5 \text{ N/m}^2 = 0,25 \text{ MPa} = 2,5 \text{ bar}$

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