
**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

STANDARDSISO.COM : Click to view the full PDF of ISO 4802-2:2023



STANDARDSISO.COM : Click to view the full PDF of ISO 4802-2:2023



COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	4
5 Reagents	4
6 Apparatus	6
7 Sample preparation	7
7.1 Sample size.....	7
7.2 Determination of the filling volume.....	7
7.2.1 Flat-bottomed containers ≤ 20 mm outer flange diameter (except ampoules, syringes and cartridges).....	7
7.2.2 Flat-bottomed containers > 20 mm outer flange diameter.....	7
7.2.3 Round-bottomed containers.....	8
7.2.4 Lipped containers.....	8
7.2.5 Ampoules.....	8
7.2.6 Syringes and cartridges.....	8
8 Procedure	9
8.1 General.....	9
8.2 Cleaning of samples.....	9
8.3 Filling and heating.....	9
8.4 Analysis of the extraction solutions.....	10
8.4.1 Containers of hydrolytic resistance container classes HC _F 1, HC _F 2 and HC _F B or those known to be made from borosilicate glass.....	10
8.4.2 Containers of hydrolytic resistance container classes HC _F 3 and HC _F D, or those known to be made from soda-lime-silica glass.....	10
8.5 Test to determine whether the containers have been surface-treated.....	11
9 Expression of results	12
9.1 Determination.....	12
9.2 Classification.....	12
9.3 Distinction between containers of hydrolytic resistance container class HC _F 1 and hydrolytic resistance container class HC _F 2.....	13
9.4 Designation.....	13
10 Test report	13
11 Reproducibility	13
Bibliography	15

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 76, *Transfusion, infusion and injection, and blood processing equipment for medical and pharmaceutical use*.

This fourth edition cancels and replaces the third edition (ISO 4802-2:2016), which has been technically revised.

The main changes are as follows:

- the explanation of the hydrolytic classes HC_F1, HC_F 2, HC_F 3, HC_FB, HC_F D for different glass types;
- clarification of needed properties of purified water and test water;
- harmonization of the samples cleaning and the autoclavation process with Ph.Eur.;
- including containers up to 0,5 ml filling volume.

A list of all parts in the ISO 4802 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document is largely based on a test method 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 document contains a classification which is related to but not equivalent to the classification set up in ISO 4802-1 for the titration method.

The hydrolytic resistance of the inner glass surface is evaluated by determination of the released alkali reacting ions. According to their hydrolytic resistance, glass containers are classified in defined categories.

HC_F1 glass containers are suitable for most preparations whether or not for parenteral administration.

HC_F2 glass containers are suitable for most acidic and neutral, aqueous preparations whether or not for parenteral administration.

HC_F3 glass containers are in general suitable for non-aqueous preparations for parenteral administration, for powders for parenteral administration (except for freeze-dried preparations) and for preparations not for parenteral administration.

HC_FB glass containers are in general suitable for drinking ampoules (Container Class HGB 2 according to ISO 719).

HC_FD glass containers are in general suitable for lower demands on hydrolytic resistance (Container Class HGB 4 and HGB 5 according to ISO 719).

STANDARDSISO.COM : Click to view the full PDF of ISO 4802-2:2023

[STANDARDSISO.COM](https://standardsiso.com) : Click to view the full PDF of ISO 4802-2:2023

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

Part 2: Determination by flame spectrometry and classification

1 Scope

This document specifies:

- a) a method for determining the hydrolytic resistance of the interior surfaces of glass containers when subjected to attack by water at (121 ± 1) °C for (60 ± 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 document.

The test method specified in this document might not be applicable to containers whose surfaces have been treated for functional modifications, e.g. silicone (e.g. containers that are ready for direct filling).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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

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

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

ISO 3819, *Laboratory glassware — Beakers*

ISO 9187-1, *Injection equipment for medical use — Part 1: Ampoules for injectables*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

ampoule

small, normally flat-bottomed container having stems in many different forms

Note 1 to entry: Ampoules are usually thin-walled and have a capacity normally up to 30 ml. They are intended to be closed, after filling, by flame sealing.

3.2

bottle

flat-bottomed container, made from moulded glass

Note 1 to entry: Bottles are normally thick-walled and have a capacity usually of more than 5 ml. They may be of circular or other geometric cross-section. Bottles are sealed with a closure made from a material other than glass, and not by flame-sealing.

3.3

brimful capacity

volume of water required to fill a container, placed on a flat, horizontal surface

3.4

container

article made from glass to be used as primary packaging material intended to come into direct contact with a pharmaceutical preparation

EXAMPLE Bottles, vials, syringes, ampoules and cartridges. See also [Figure 1](#).

Note 1 to entry: These containers are made from borosilicate or soda-lime-silica glass.

3.5

filling volume

defined volume of water to fill the test specimen

Note 1 to entry: For the determination of the filling volume, see [7.2](#). The filling volume is a test specific quantity that is used to compare container sets from different sources or lots. It has no relation to the nominal product volume.

3.6

borosilicate glass

silicate glass having a very high hydrolytic resistance due to its composition containing significant amounts of boric oxide

Note 1 to entry: Borosilicate glass contains a mass fraction of boric oxide between 5 % and 13 %. This glass type may also contain aluminium oxide and/or alkaline earth oxides.

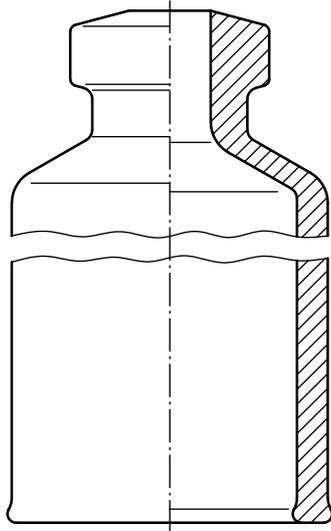
Note 2 to entry: Neutral glass is a borosilicate glass having a very high hydrolytic resistance and a high thermal shock resistance. When tested according to ISO 720, it meets the requirements of class HGA 1. Containers properly made from this glass conform with hydrolytic resistance container class HC_F 1 of this document.

3.7

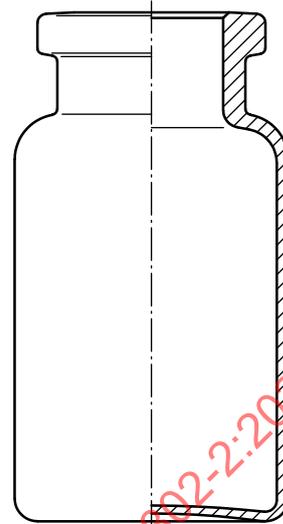
soda-lime-silica glass

silicate glass containing a mass fraction up to approximately 15 % of alkali metal oxides, mainly sodium oxide, and a mass fraction up to about 15 % of alkaline earth oxides, mainly calcium oxide

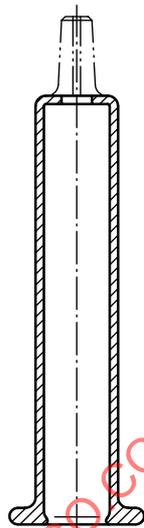
Note 1 to entry: Containers made from this glass will have a moderate hydrolytic resistance due to the chemical composition of the glass, and conform with hydrolytic resistance container class HC_F 3.



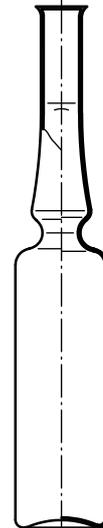
a) Example of a glass cylinder for a pen-injector/cartridge (see ISO 13926-1)



b) Example of an injection vial made of glass tubing (see ISO 8362-1)



c) Example of a syringe glass barrel (see ISO 11040-4)



d) Example of a stem cut ampoule with constriction (see ISO 9187-1)

Figure 1 — Examples of containers

3.8 surface treatment

treatment of the internal surface of 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)

Note 1 to entry: Surface treatment is used, for example, in order to change a soda-lime-silica glass container of hydrolytic resistance class HC_F 3 to a container of hydrolytic resistance class HC_F 2 container. Treated containers are rinsed before use.

3.9 vial

small, flat-bottomed container, made from tubing or from moulded glass

Note 1 to entry: Vials are normally thick-walled and have a capacity up to 100 ml. They are normally sealed with a closure made from a material other than glass, and not by flame-sealing.

4 Principle

This test method is a surface test applied to glass containers as produced and/or as delivered.

The containers to be tested are filled with specified water to a specified capacity. They are loosely capped and then heated under specified conditions. The degree of the hydrolytic attack is measured by flame spectrometric analysis of the extraction solutions. The measurement data shall be classified in accordance with the limits defined in appropriate container specific limit values in accordance with [Table 2](#).

Dependent on different glass types, specific limit values are defined in the following:

The hydrolytic resistance is evaluated by determination of the released alkali reacting ions. According to their hydrolytic resistance, glass containers are classified as follows:

- HC_F 1 glass containers: neutral glass, borosilicate glass with a high hydrolytic resistance due to the chemical composition of the glass itself;

NOTE 1 In the Ph.Eur. 3.2.1^[12] and the USP <660>^[15] the hydrolytic resistance classification are designated Type I.

- HC_F 2 glass containers: usually of soda-lime-silica glass with a high hydrolytic resistance resulting from suitable treatment of the inner surface;

NOTE 2 In the Ph.Eur. 3.2.1^[12] and the USP <660>^[15] the hydrolytic resistance classification are designated Type II.

- HC_F 3 glass containers: usually of soda-lime-silica glass with only moderate hydrolytic resistance;

NOTE 3 In the Ph.Eur. 3.2.1^[12] and the USP <660>^[15] the hydrolytic resistance classification are designated Type III.

- HC_F B glass containers: usually made of borosilicate or soda-lime-silica glass composition with higher hydrolytic resistance (container class: HGB 2 according to ISO 719);
- HC_FD glass containers: usually made of soda-lime-silica glass with low hydrolytic resistance (container class: HGB 4 or HGB 5 according to ISO 719).

The index "F" indicates that the measures for the classification is based on Flame Spectrometry.

5 Reagents

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

5.1 Test water, to be prepared as follows:

Prepare the test water from purified water ([5.5](#)) by multiple distillations. Remove the carbon dioxide by boiling for at least 15 min before use in a boiling flask ([6.3](#)) of fused silica or borosilicate glass and cool.

NOTE 1 Any other suitable method can be used, e.g. preparation of carbon dioxide-free water according to Ph.Eur. 3.2.1^[12], USP 660^[15].

When tested immediately before use, water prepared as described above shall produce an orange-red (not violet-red or yellow) colour corresponding to the neutral point of methyl red indicator of pH $5,5 \pm 0,1$ when 0,05 ml of methyl red indicator solution ([5.5](#)) is added to 50 ml of the water to be examined.

This water may also be used as the reference solution (see [8.4](#)).

The conductivity of the water shall not exceed 1 $\mu\text{S}/\text{cm}$, determined at 25 °C by an in-line conductivity meter.

Where the use of test water is specified in the following analytical procedures the requirements for bacterial endotoxins and microbial contamination are not relevant.

NOTE 2 This description is based on the Ph.Eur. 3.2.1^[12]. In the Ph.Eur., water prepared as described above is designated water R1.

NOTE 3 Water of Grade 2 according to ISO 3696^[2] is suitable for this test.

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

5.3 Hydrochloric acid, solution, $c(\text{HCl}) \approx 6 \text{ mol/l}$ ($\approx 1 + 1$).

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

CAUTION — Hydrofluoric acid is very toxic and highly corrosive. Consider using a material safety data sheet.

5.5 Purified water, prepared by distillation, by ion exchange, by reverse osmosis or by any other suitable method from water having drinking water quality.

Where the use of purified water is specified in the following analytical procedures, the requirements for bacterial endotoxins and microbial contamination are not relevant.

NOTE 1 National or regional regulation on water intended for human consumption can apply.

NOTE 2 Water that corresponds to Grade 3 according to ISO 3696 is suitable.

NOTE 3 In the Ph.Eur. 3.2.1^[12], water as described above is designated water R.

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

Dissolve 80 g of caesium chloride in approximately 300 ml of test water (5.1), add 10 ml of hydrochloric acid (5.3) and transfer to a 1 000 ml volumetric flask (6.3). Dilute to the mark with the test water (5.1) and mix.

5.7 Stock solutions.

5.7.1 Dry sodium chloride, potassium chloride and calcium carbonate at $(110 \pm 5) \text{ }^\circ\text{C}$ for 2 h. Prepare aqueous stock solutions, using the test water (5.1), directly from the chlorides and from the calcium carbonate, after dissolving in the minimum amount of hydrochloric acid so that all solutions have concentrations of 1 mg/ml, calculated as sodium oxide, potassium oxide and calcium oxide.

5.7.2 Commercially available standard solutions may also be used.

5.8 Standard solutions.

5.8.1 Prepare standard solutions by diluting the stock solutions (5.7) with the test water (5.1) to obtain concentrations suitable for establishing the reference solutions in an appropriate manner, e.g. with concentrations of 20 $\mu\text{g/ml}$ of sodium oxide, potassium oxide and calcium oxide respectively.

5.8.2 Commercially available standard solutions may also be used.

5.9 Reference solutions.

The reference solutions for establishing the calibration graph (set of calibration solutions) shall be prepared by diluting suitable concentrated standard solutions (5.8) with the test water (5.1). They shall

cover 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 of containers of hydrolytic resistance container of all classes, the reference solutions shall contain a volume fraction of 5 % of the spectrochemical buffer solution (5.6).

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Autoclave or steam sterilizer, capable of withstanding a pressure of at least 250 kPa and of carrying out the heating cycle specified in 8.3. It shall be capable of maintaining a temperature of (121 ± 1) °C, equipped with a calibrated thermometer or a calibrated thermocouple recorder, a pressure gauge and a vent-cock.

When necessary and appropriate, the autoclave vessel and ancillary equipment shall be thoroughly cleaned before use using the purified water (5.5) in order to avoid contamination that can influence the test results.

Most recent autoclave models are not provided with a vent cock that can be operated manually. The operator is referred to the user instructions and/or technical description provided by the manufacturer of the autoclave with regard to the functioning of the venting operations. A time/temperature printout/recording can be used as evidence of a proper venting stage.

6.2 Burettes, having a suitable capacity according to the analytical procedure to be used and conforming with the requirements specified for class A burettes in ISO 385 and made of glass of hydrolytic resistance grain class HGA 1 as specified in ISO 719 or ISO 720 (Glass of hydrolytic resistance grain class ISO 719-HGB 1 adequately meets the requirements of class HGA 1 specified in ISO 720.).

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

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

6.5 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.

As an alternative to flame spectroscopy, ICP-OES (Inductively coupled Plasma-Optical Emission Spectroscopy) or ICP-MS (Inductively coupled Plasma-Mass Spectrometry) may be used for the measurements. The equipment shall be qualified and validated for the appropriate use.

6.6 Beakers, having a suitable capacity and conforming with the requirements specified in ISO 3819.

Before its first use, each beaker shall be pretreated by filling with purified water (5.5) and autoclaving at 121 °C for at least 1 h (see 8.3).

6.7 Metal foil, e.g. made from aluminium or stainless steel.

7 Sample preparation

7.1 Sample size

For each container capacity to be tested, the number of containers 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 of container [volume corresponding to filling volume (see 7.2)] ml	Number of containers to be measured separately	Additional containers for desired preliminary analysis (8.4.2.1)
≤2	20	2
>2 ≤5	15	2
>5 ≤30	10	2
>30 ≤100	5	1
>100	3	1

7.2 Determination of the filling volume

7.2.1 Flat-bottomed containers ≤20 mm outer flange diameter (except ampoules, syringes and cartridges)

Select six containers (having a capacity ≤100 ml) or three containers (having a capacity >100 ml) at random from the sample lot and remove any debris or dust. Allow the dry containers to reach room temperature. Weigh each of the empty containers to the nearest 0,01 g for containers having a nominal volume ≤30 ml, and to the nearest 0,1 g for containers having a nominal volume >30 ml. Place the containers on a horizontal surface and fill them nearly to the top with purified water (5.5), avoiding overflow and introduction of air bubbles. Adjust the liquid levels to the brimful line using purified water (5.5). The meniscus shall be equal to the upper edge of the bore.

Weigh the filled container to the nearest 0,01 g for containers having a nominal volume ≤30 ml, and to the nearest 0,1 g for containers having a nominal volume >30 ml. Calculate the mass of water, in grams, contained within the container.

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

Determine 90 % of this mean brimful capacity to one decimal place. This amount of water is used for the particular sample lot.

7.2.2 Flat-bottomed containers >20 mm outer flange diameter

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

7.2.3 Round-bottomed containers

Select six containers (having a capacity ≤ 100 ml) or three containers (having a capacity >100 ml) at random from the sample lot and remove any debris or dust. Allow the dry containers to reach room temperature. Fix each container vertically in an appropriate device and determine the brimful capacity in accordance with [7.2.1](#).

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

7.2.4 Lipped containers

Wrap adhesive plastic tape around the rim of the containers such that the tape around the lip is level with the rim. Weigh the container, then fill them with purified water ([5.5](#)) and reweigh as described in [7.2.1](#).

7.2.5 Ampoules

Place at least six dry ampoules on a flat, horizontal surface and fill them with purified water ([5.5](#)), at room temperature, from a burette ([6.2](#)), until the water level (meniscus) reaches point h_6 of ISO 9187-1, where the body of the ampoules 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 all ampoules of the same lot.

NOTE The filling volume can also be determined by weighing.

7.2.6 Syringes and cartridges

Select six syringes or cartridges. Close the small opening (mouth of cartridges and needle and/or Luer cone of syringes) using an inert material (e.g. tip cap). Determine the mean brimful volume in accordance with [7.2.1](#).

Then determine 90 % of the mean brimful capacity to one decimal place. This volume is the filling volume.

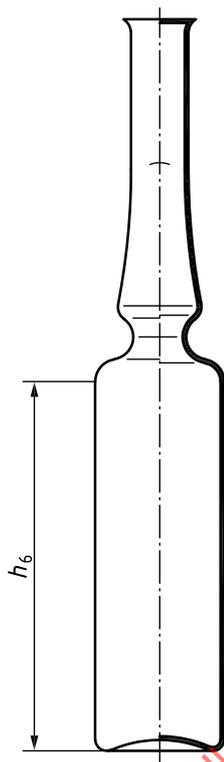


Figure 2 — Filling volume of ampoules (up to h_6)

8 Procedure

8.1 General

This procedure shall be completed within one working day.

8.2 Cleaning of samples

Remove from all open samples any debris or dust. Shortly before each test, fill each container to the brim with purified water (5.5) at ambient temperature and allow to stand, for (20 ± 5) min. Immediately before testing, empty the samples, rinse twice with purified water (5.5) and then once with the test water (5.1) and allow to drain.

Closed ampoules shall not be rinsed before testing.

NOTE For opening by flame, closed ampoules can be warmed, e.g. in a water bath or air-oven at about $40\text{ }^{\circ}\text{C}$ for approximately 2 min before opening, to avoid under pressure when opening, or cut and broken at the height of the sealing point.

8.3 Filling and heating

Fill each container, selected for the sample size in accordance with 7.1 and cleaned in accordance with 8.2, to the filling volume with the test water (5.1) by means of suitable volumetric measuring devices.

Cap each container, including ampoules, loosely with an inert material, for example with inverted beakers (6.6) of such a size that the bottoms of the beakers fit snugly down on the rims of the sample. Cap ampoules with a clean metal foil (6.7), e. g. aluminium foil. Place syringes and cartridges in a beaker and cover the beaker with a clean metal foil (6.7).

Ensure that the metal foil (6.7) does not release measurable ions into the test water.

Containers of a volume of 2 ml or less, in which the water is not sufficiently retained during the autoclaving process, may be closed in a suitable way, e.g. with a stopper or plug of inert material, such as silicone, and fixed using a plunger or a stable fixing or clamping device.

Place the samples, gathered in groups in glass dishes or in the beaker, on the rack in the autoclave (6.1), containing purified water (5.5) at ambient temperature, and ensure that they are held above the level of the water in the vessel.

Insert the end of a calibrated thermal device in a filled container through a hole of approximately the diameter of the thermocouple and connect it to an external measuring device. If the container is too small to insert a thermocouple, apply a thermocouple in a suitable, similar container. Close the autoclave door or lid securely but leave the vent-cock open. Start automatic recording of the temperature versus time and heat the autoclave with the following characteristics: raise the temperature with a regular rate to 100 °C within 20 min to 30 min and maintain the temperature at (100 ± 1) °C for a further (10 ± 1) min. Raise the temperature from 100 °C to 121 °C within 20 min to 22 min. Maintain the temperature at (121 ± 1) °C for (60 ± 1) min from the time when the holding temperature is reached. Cool down to 100 °C within 40 min to 44 min.

CAUTION — For security reasons (boiling retardation), do not open the autoclave before the water in the containers has reached a temperature of 95 °C. Consider the safety instructions of the user manual.

NOTE Experience has shown that the rate of heating to 121 °C, the holding temperature of (121 ± 1) °C and the rate of cooling to 100 °C are critical. Deviations from the specified conditions can produce variable results even to the extent of invalidating them.

Remove the hot samples from the autoclave and cool to room temperature within 30 min. Start with the determinations after cooling. Special care shall be taken in cooling down large capacity containers as thermal drops larger than 40 °C can cause the fracture of the glass by thermal shock.

8.4 Analysis of the extraction solutions

8.4.1 Containers of hydrolytic resistance container classes HC_F 1, HC_F 2 and HC_F B or those known to be made from borosilicate glass

Normally, these containers do not release potassium or calcium in a significant amount and only sodium is to be determined. Carry out preliminary measurements of the potassium and calcium oxide concentrations on one of the extraction solutions. If, for one container type, the concentration of potassium oxide is less than 0,2 µg/ml, and if the concentration of calcium oxide is less than 0,1 µg/ml, the remaining extraction solutions of this container type need not be analysed for these ions.

Add to one container, from each container type, a volume of the spectrochemical buffer solution (5.6) equivalent to 5 % of the filling volume.

Aspirate the extraction solution from each sample directly into the flame of the atomic absorption or atomic emission instrument (6.5), and determine the concentrations of sodium oxide (and potassium oxide and calcium oxide, if present) by reference to calibration graphs produced from the aqueous reference solutions (5.9) of suitable concentration. For very small volume containers a pooling of 5 or 6 containers is necessary to have enough fluid capacity for testing. This shall be done by an appropriate inert container not influencing the chemical composition of the fluid.

8.4.2 Containers of hydrolytic resistance container classes HC_F 3 and HC_F D, or those known to be made from soda-lime-silica glass

8.4.2.1 Preliminary analysis

Add to one container, from each container type, a volume of the spectrochemical buffer solution (5.6) equivalent to 5 % of the filling volume.

Cap narrow-necked containers with a piece of inert plastic film and mix the liquid well by shaking. Mix the liquids in other containers by using stirrers.

Ensure that the plastic film does not release the ions to be determined.

Aspirate the extraction solution into the flame of the instrument (6.5) and determine first the approximate sodium oxide concentration, then the exact potassium oxide and the calcium oxide concentration. When the potassium oxide concentration is less than 0,2 µg/ml and if the concentration of calcium oxide is less than 0,1 µg/ml, the remaining solutions of this container type need not be analysed for these ions.

According to the instrument conditions, the sodium oxide concentration may be above the optimum working range. For FAAS techniques, this normally takes place for concentrations greater than 3 µg/ml of sodium oxide. In these cases, dilute the extraction solution for the final measurements so that the sodium oxide concentration becomes less than 3 µg/ml.

Take care that dilution to concentrations less than 3 µg/ml of sodium oxide is done carefully. The volumes shall be measured to two decimal places and the measurement and dilution shall be done in very carefully cleaned apparatus.

If dilution is necessary, prepare such a diluted solution from an original extraction solution, and add the spectrochemical buffer solution (5.6) [5 % (volume fraction)].

NOTE Experience shows that calcium oxide and potassium oxide can be accurately measured from undiluted solutions.

8.4.2.2 Final analysis

If dilution is unnecessary, add a volume of the spectrochemical buffer solution (5.6) equivalent to 5 % of the filling volume as described in 8.4.2.1, mix well and determine sodium oxide and calcium oxide (and potassium oxide, if present) by reference to calibration graphs. They shall be produced from the aqueous reference solutions (5.9) and shall contain a volume fraction of 5 % of the spectrochemical buffer solution (5.6).

For the determination of the calcium oxide concentration with the FAAS method, the nitrous oxide/acetylene flame shall be used.

If dilution is necessary, determine sodium oxide, calcium oxide and potassium oxide, if present, following the procedures as described before. The measuring solutions shall contain a volume fraction of 5 % of the spectrochemical buffer solution (5.6).

Ensure that any dilution (because of sodium oxide concentration and/or caesium chloride addition) is taken into consideration in the calculations.

Concentration values <1,0 µg/ml should be expressed to two decimal places, values ≥1,0 µg/ml to one decimal place.

8.5 Test to determine whether the containers have been surface-treated

NOTE The hydrolytic resistance of the interior surface of vials and bottles made from soda-lime-silica glass can be considerably increased by treating these surfaces during the course of production.

If there are doubts whether surface treatment or not was performed on a container and/or to distinguish between type I and type II glass containers, unused glass containers or the container samples previously tested shall be used.

Fill the samples with a mixture of one volume of hydrofluoric acid (5.4) and nine volumes of hydrochloric acid (5.2) to the brimful point. Allow the filled samples to stand at ambient temperature for 10 min, then empty the solution very carefully. Rinse the samples five times with purified water (5.5), then at least once again with purified water (5.5). Then test the samples as specified in 8.3, and 8.4.