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

*Verre — Résistance hydrolytique du verre en grains à 98 °C —
Méthode d'essai 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.

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 documents 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 third edition cancels and replaces the second edition (ISO 719:1985), which has been technically revised.

The main changes compared to the previous edition are as follows:

- a more precise definition of the field of application by means of glass types was added;
- wherever possible a harmonization with the identical paragraphs in the European Pharmacopoeia, chapter 3.2.1, and the USP, chapter 660, was established to simplify the application in the laboratories globally. This concerns, e.g. sample size, mesh size;
- the usage of acetone was restricted to always fresh, new acetone, since re-usage might lead to deviating test results;
- the maximum temperature and its tolerance field was simplified and oriented on the technical conditions of a water basin.

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.

Glass — Hydrolytic resistance of glass grains at 98 °C — Method of test and classification

1 Scope

This document specifies

- a) a method for determining the hydrolytic resistance of glass grains at 98 °C. The resistance is measured and expressed by the volume of acid required for titration of the alkali extracted from the unit mass of glass, and can also be expressed by the amount of sodium oxide equivalent to this volume of acid, and
- b) a classification of glass according to the hydrolytic resistance determined by the method of this document.

This document is intended for use on the less resistant types of glass, such as soda-lime glass.

NOTE 1 For the more resistant glasses, e.g. borosilicate glass, the method specified in ISO 720 is more suited.

NOTE 2 It is emphasized that there is no exact correlation between the classification laid down in this document and that laid down in ISO 720, and it is, therefore, essential to identify which classification is being used.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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 1773, *Laboratory glassware — Narrow-necked boiling flasks*

ISO 3819, *Laboratory glassware — Beakers*

ISO 13130, *Laboratory glassware — Desiccators*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

4 Principle

The method of testing is a test for glass as a material applied on glass grains. Extraction of 2 g of grains, of particle size between 300 µm and 500 µm, with grade 2 water for 60 min at 98 °C. Measurement of the degree of the hydrolytic attack by analysis of the extraction solutions.

The test method shall not be applied to glasses with extreme low alkaline contents or that are essentially free of alkaline species as this method measures only the alkaline release as the indication for chemical durability.

The density of the glass to be tested should, preferably, be $2,4 \text{ g/cm}^3 \pm 0,2 \text{ g/cm}^3$ at 20 °C.

5 Reagents

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

5.1 Test water, to be prepared as follows: prepare the test water from distilled water (5.6) by multiple distillations. Remove the carbon dioxide by boiling for at least 15 min before use in a boiling flask (6.6) of fused silica or borosilicate glass and cool. Any other suitable method may be used, e.g. preparation of carbon dioxide-free water according to USP 660^[4].

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 $\text{pH } 5,5 \pm 0,1$ when 0,05 ml of methyl red indicator solution (5.4) is added to 50 ml of the water to be examined. This water may also be used as the reference solution (see Clause 8). The conductivity of the water shall not exceed 1 µS/cm, determined at 25 °C by an in-line conductivity meter.

NOTE 1 This description is based on the European Pharmacopoeia 3.2.1^[3]. In the European Pharmacopoeia, water prepared as described above is designated water R1.

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

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

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

5.4 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 grade 2 water (5.1). Alternatively, the indicator solution may be prepared as described in, e.g. USP 42 [6.60] or Ph.Eur. 10 [3.2.1] (this method is based on dissolution of methyl red in 0,1 M sodium hydroxide, ethanol and distilled water).

5.5 Acetone (CH_3COCH_3).

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

NOTE 1 See national or regional regulation for information on water intended for human consumption.

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

NOTE 3 In the European Pharmacopoeia 3.2.1^[3], water as described above is designated water R.

6 Apparatus

Ordinary laboratory apparatus and, in particular, the following:

6.1 Balance, capable of weighing up to 500 g, accurate to ± 5 mg or better.

6.2 Burettes, having a capacity of 5 ml, 2 ml or 1 ml, conforming to the requirements specified for class A burettes in ISO 385 (see also general requirements specified in ISO 385) and made of glass of hydrolytic resistance grain class HGA 1 as specified in ISO 720.

The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid (5.2).

6.3 Pipette, having a capacity of 25 ml and conforming to the requirements specified for class A pipettes in ISO 648.

6.4 One-mark volumetric flasks, having a capacity of 50 ml, conforming to the requirements specified for class A one-mark volumetric flasks in ISO 1042, made of glass of hydrolytic resistance grain class HGA 1 as specified in ISO 720, and with glass stoppers. Flasks with the graduation mark in the lower half of the neck should be selected.

Before use, each new flask shall be pretreated in the following manner: The flasks shall be filled to above the graduation mark with hydrochloric acid (5.3) and heated at just above the test temperature for 2 h in the heating bath (6.19). The flasks shall then be rinsed with water (5.6), filled to above the graduation mark with water and heated as above for two periods of 1 h, using fresh water each time. Flasks made from vitreous silica may also be used, in which case pretreatment is not required. When flasks have been used to measure the alkali-release from grain of low-resistance glass, they should be pretreated again, as above, before using them for further tests in order to eliminate cross-contamination.

6.5 Conical flasks, having a capacity of 100 ml and conforming to the requirements of ISO 1773.

6.6 Boiling flasks, having a capacity of 1 000 ml, conforming to the requirements of ISO 1773 and made of vitreous silica or borosilicate glass.

Before use, each new flask shall be pretreated as described in 6.4.

6.7 Beakers, having a capacity of 100 ml and conforming to the requirements of ISO 3819.

6.8 Weighing bottles, having a capacity of about 20 ml.

6.9 Desiccator, conforming to the requirements of ISO 13130.

6.10 Hammer, having a mass of about 0,5 kg, made of tempered, magnetic steel.

6.11 Mortar and pestle, made of tempered magnetic steel, and of the design and approximate dimensions shown in Figure 1.

6.12 Permanent magnet.

6.13 Sieves, conforming to the requirements of ISO 565 and comprising a set of 200 mm diameter square-aperture sieves, with stainless steel mesh, including:

- a sieve A of 500 μm aperture;
- a sieve B of 300 μm aperture;
- a sieve O of a 710 μm aperture.

The cover, pan and, especially, the rings shall be of stainless steel or lacquered wood. The use of sieve O is recommended to retain larger pieces of glass and to avoid heavy wear a sieve A.

6.14 Ball-mill.

The mill shall be made of agate, zirconia or stainless steel with a volume of 250 ml. Two balls with a diameter of 40 mm or three balls with a diameter of 30 mm are suitable.

6.15 Sieving-machine.

A mechanical sieve-shaker or sieving-machine may be used to sieve the grains.

6.16 Ultrasonic cleaner (laboratory type).

6.17 Drying oven, capable for maintaining a temperature of 140 ± 5 °C.

6.18 Thermometer, covering the range from 90 °C to 110 °C, capable of being read to an accuracy of $\pm 0,2$ °C.

6.19 Heating bath, gas or electrically heated, thermostatically controlled, having capacity sufficient to contain at least 1 l of liquid for each flask used in the test and capable of carrying out the heating cycle specified in [Clause 8](#).

6.20 Warm plate, to remove the excess of acetone.

Dimensions in millimetres

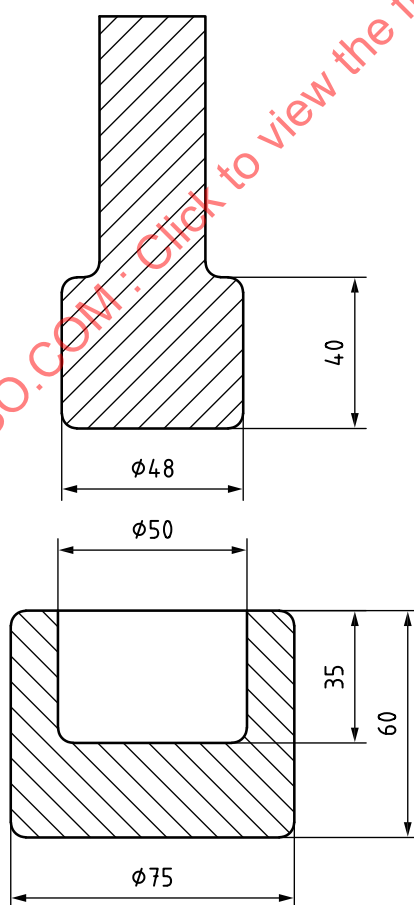


Figure 1 — Mortar and pestle

7 Preparation of sample

7.1 Crushing

Check that the articles as received have been annealed to a commercially acceptable quality.

If an article is not annealed to a commercially acceptable quality, this fact should be noted because the results can be affected. Such articles, if very badly annealed, might also break very easily and extra care should be taken when handling them. Further annealing should not be carried out before the test.

Wrap the glass articles in clear paper and crush to produce two 100 g samples of pieces not more than 30 mm across.

7.2 Manual preparation

Place 30 g to 40 g of pieces (see 7.1) between 10 mm and 30 mm across in the mortar (6.11), insert the pestle (6.11), and strike it sharply, once only, with the hammer (6.10).

If more than one hammer blow is used in crushing the glass, the very fine particles produced may be compacted into aggregates which may or may not be subsequently broken down and which can, therefore, introduce further variables into the test.

Transfer the glass from the mortar to the upper sieve O of the assembled set of sieves (6.13) and shake the set of sieves briefly to separate the finer particles. Return the glass remaining on sieves A and O to the mortar. Repeat crushing and sieving until only about 10 g of glass remain on sieve O. Discard the glass from sieve A and O and from the receiving pan. Shake the set of sieves by hand for 5 min. Reserve for the test those grains that pass through sieve A and that are retained on sieve B.

At least 10 g of sample are required for the test. If it is necessary to crush and sieve more sample, the sample already obtained shall be removed from sieve B and stored in a weighing bottle (6.8).

After completion of all crushing and sieving, combine the samples, spread the grains on clean glazed paper and remove any iron particles using the magnet (6.12). Transfer the grains into a beaker (6.7) for cleaning.

7.3 Mechanical preparation

Transfer about 50 g of the coarsely broken glass (see 7.1) into the mill-beaker (6.14), add the balls and crush thin-walled glass (wall thickness $\leq 1,5$ mm) for 2 min, thick-walled glass ($>1,5$ mm) for 5 min.

Transfer the grains to the upper sieve O of the assembled set (6.13) of the sieving-machine (6.15), sieve for about 30 s and collect the grains retained on sieve B in the beaker (6.7), which shall be kept in the desiccator (6.9). Transfer the glass from sieves O and A back into the ball-mill and crush again for the time given above. Repeat sieving and crushing until about 10 g of grains have been collected from sieve B. Continue as specified in 7.3, last paragraph.

7.4 Cleaning

Add to the grains in each beaker (6.7) 30 ml of the acetone (5.5) and scour the grains by a suitable means, such as a rubber or plastic-coated glass rod.

After scouring, swirl the grains, allow it to settle and decant as much acetone as possible. Add another 30 ml of the acetone, swirl, allow it to settle and decant again and add a new portion of 30 ml of the acetone. Fill the bath of the ultrasonic cleaner (6.16) with water at room temperature, then place the beaker in the rack and immerse it until the level of the acetone is at the level of the water; apply the ultrasonic for 1 min. To avoid deviations of the measuring results, don't reuse acetone used previously.

Swirl the beaker and decant the acetone as completely as possible and then repeat the ultrasonic cleaning operation. If a fine turbidity persists, repeat the ultrasonic cleaning and acetone washing until

the solution remains clear. Swirl and decant the acetone, then dry the grains, first by putting the beaker with the grains on a warm plate (6.20) to remove excess acetone and then by heating at 140 °C for 20 min in the drying oven (6.17).

Transfer the dried grains from the oven into a weighing bottle (6.8), insert the stopper and cool in the desiccator (6.9).

8 Procedure

Weigh 2,00 g of the cleaned and dried grains into each of three of the one-mark volumetric flasks (6.4). Fill the flasks to the mark with the grade 2 water (5.1) and fill two more flasks with the water, one to serve as a reference solution and the other to serve as temperature control.

Distribute the glass grains evenly over the flat bases of the sample flasks by gently shaking them, then place all of the flasks, without stoppers, in the heating bath (6.19), so that they are immersed to half-way up the necks (a rack to hold the flasks may be used). Increase the rate of heating such that boiling temperature is achieved in the control flasks as soon as possible; when reached, apply the stoppers immediately. Continue heating for 60 min $\pm$ 1 min from the time of immersion, maintaining boiling temperature in the flasks.

Remove the flasks from the heating bath, take out the stoppers, cool the flasks in running water as soon as possible, avoid thermal shock and complete the titration within 1 h. Adjust the contents of each flask to the mark with the grade 2 water. Replace the stoppers and mix the contents of each flask thoroughly, then allow to stand until the grains settle and a clear supernatant solution is obtained.

By means of the pipette (6.3), transfer 25 ml of the clear solution from each flask into separate conical flasks (6.5), add to each of these flasks two drops of the methyl red indicator solution (5.4), and titrate immediately with the hydrochloric acid solution (5.2), until the colour matches exactly that of the 25 ml of the water of the reference solution plus two drops of the indicator contained in a similar conical flask.

9 Expression of results

9.1 Calculation

Calculate the mean value of the results, in millilitres of hydrochloric acid solution (5.2) per gram of sample, and, if required, its equivalent in alkali extracted, calculated as micrograms of sodium oxide (Na_2O) per gram of glass grains:

1 ml of hydrochloric acid solution

$[c(\text{HCl}) = 0,01 \text{ mol/l}] \triangleq 310 \text{ } \mu\text{g}$ of sodium oxide

If the highest and the lowest observed values differ by more than the permissible range given in Table 1, repeat the test.

9.2 Classification

Glass shall be classified as shown in Table 2, according to the consumption of acid and its equivalent of alkali [expressed as sodium oxide (Na_2O)], when tested by the method specified in this document.

9.3 Designation

For convenience of reference to the hydrolytic resistance of glass as a material conforming to the classification of this document, the use of a designation as follows is recommended:

EXAMPLE The designation for a glass with a consumption of 0,60 ml of hydrochloric acid solution $[c(\text{HCl}) = 0,01 \text{ mol/l}]$ per gram of glass grains equivalent to 186 μg of sodium oxide per gram of glass grains (class HGB 3) shall be:

Glass, hydrolytic resistance grain class ISO 719 — HGB 3

10 Test report

The test report shall include the following information:

- a reference to this document, i.e. ISO 719:2020;
- an identification of the sample;
- the consumption of hydrochloric acid solution [$c(\text{HCl}) = 0,01 \text{ mol/l}$], in millilitres per gram of glass grains, mean value;
- in addition, if required, the equivalent of alkali, in micrograms of sodium oxide per gram of glass grains, mean value;
- the hydrolytic resistance grain class HGB (designation of the glass tested);
- the wall thickness of the articles used for the test if it was $\leq 1,5 \text{ mm}$;
- the density of the glass if it was outside the range of $2,4 \text{ g/cm}^3 \pm 0,2 \text{ g/cm}^3$ at 20°C ;
- a statement, if appropriate, that the glass article used for the test was not annealed to commercially acceptable quality.

Table 1 — Permissible range of the values obtained

Mean of the values obtained for the consumption of hydrochloric acid solution [$c(\text{HCl}) = 0,01 \text{ mol/l}$] (5.2) per gram of glass grains ml/g	Permissible range of the values obtained
up to and including 0,10	30 % of the mean
from 0,10 up to and including 0,20	20 % of the mean
from 0,20 upwards	10 % of the mean

Table 2 — Limit values in the hydrolytic resistance grains test (boiling water test)

Class ^a	Consumption of hydrochloric acid solution [$c(\text{HCl}) = 0,01 \text{ mol/l}$] (5.2) per gram of glass grains ml/g	Equivalent of alkali expressed as mass of sodium oxide (Na_2O) per gram of glass grains $\mu\text{g/g}$
HGB 1	up to and including 0,10	up to and including 31
HGB 2	from 0,10 up to and including 0,20	from 31 up to and including 62
HGB 3	from 0,20 up to and including 0,85	from 62 up to and including 264
HGB 4	from 0,85 up to and including 2,0	from 264 up to and including 620
HGB 5	from 2,0 up to and including 3,5	from 620 up to and including 1 085
^a "HGB" stands for the hydrolytic resistance of glass grains according to the boiling water test method.		