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

*Verre — Résistance hydrolytique du verre en grains à 121 °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 720:1985), 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 process of autoclaving was adapted to the requirements of the European Pharmacopoeia, chapter 3.2.1, which will ease the application of this document;
- the usage of acetone was restricted to always fresh, new acetone, since re-usage might lead to deviating test results;

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 121 °C — Method of test and classification

1 Scope

This document specifies

- a) a method for determining the hydrolytic resistance of glass grains at 121 °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 more resistant types of glass, e.g. borosilicate glass.

NOTE 1 For the less resistant glasses, e.g. soda-lime, the method specified in ISO 719 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 719, 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 1773, *Laboratory glassware — Narrow-necked boiling flasks*

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

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 10 g of grains, of particle size between 300 µm and 425 µm, with grade 2 water for 30 min at 121 °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 (5.5) by multiple distillations. Any other suitable method can 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 pH $5,5 \pm 0,1$ when 0,05 ml of methyl red indicator solution (5.3) 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 is suitable for this test.

5.2 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,02 \text{ mol/l}$.

5.3 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 can 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).

5.4 Acetone (CH_3COCH_3).

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.

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 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 $\pm 5 \text{ mg}$ or better.

6.2 Burettes, having a capacity of 25 ml, 10 ml or 2 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 this document.

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

6.3 Pipettes, having a capacity of 50 ml and conforming to the requirements specified for class A pipettes in ISO 648.

6.4 Conical flasks, having a capacity of 250 ml and conforming to the requirements of ISO 1773. Before use, each new flask shall be pretreated by subjecting it to the autoclaving conditions described in [Clause 8](#). Flasks made from vitreous silica may also be used, in which case pretreatment is not required.

6.5 Beakers, having a capacity of 50 ml and conforming to the requirements of ISO 3819. Before use, each new beaker shall be pretreated by subjecting it to the autoclaving conditions described in [Clause 8](#).

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 by subjecting it to the autoclaving conditions described in [Clause 8](#).

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](#).

Approximate dimensions in millimetres

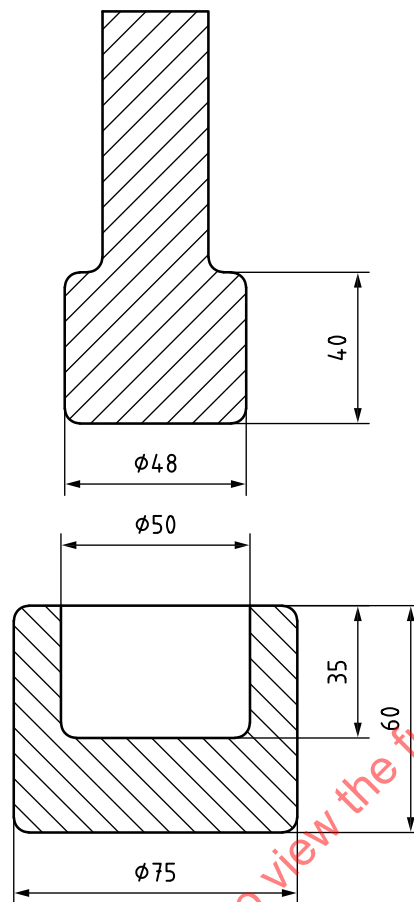


Figure 1 — Mortar and pestle

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 425 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 on 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 3 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 Autoclave or steam sterilizer, capable of withstanding a pressure of at least $(2,5 \times 10^5)$ N/m²¹⁾ and of carrying out the heating cycle specified in [Clause 8](#). It should preferably be equipped with a constant-pressure regulator or other means of maintaining the temperature at 121 °C $\pm$ 1 °C. The vessel shall be equipped with a heating device, a thermometer integrated in the autoclave, a pressure gauge, a vent cock (for manually operated autoclaves only), and a rack of sufficient capacity to accommodate above the water level, the number of flasks needed to carry out the test.

The autoclave has the possibility to connect a calibrated resistance thermometer or a calibrated thermocouple from the inner chamber to an external measuring device to allow a temperature measurement independent from the autoclave system.

The autoclave vessel and ancillary equipment shall be thoroughly cleaned with Grade 2 water as specified in ISO 3696 before use.

6.19 Warm plate, to remove the excess of acetone.

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 clean 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 between 10 mm and 30 mm across, taken from a 100 g sample (see [7.1](#)), 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](#)). Repeat the crushing procedure until the whole of the 100 g sample has been added to the sieve O. Shake the set of sieves for a short time by hand and then remove the glass from sieves A and O. Repeat crushing and sieving on this glass until only about 10 g of glass remain on sieve O. Discard the glass from sieve A and O and from the receiving pan.

Reassemble the set of sieves and shake for 5 min. Transfer to the weighing bottle ([6.8](#)) those glass grains that pass through sieve A and that are retained on sieve B.

Repeat the crushing and sieving procedure with the other 100 g sample and thus 2 samples of grains, each of which shall be in excess of 10 g, are obtained. Spread each sample on a piece of clean glazed paper and remove any iron particles using the magnet ([6.12](#)). Transfer each sample into a beaker ([6.7](#)) for cleaning.

1) $(2,5 \times 10^5)$ N/m² = 0,25 MPa = 2,5 bar.

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 (5.4) 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 (6.19) 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 each beaker to separate weighing bottles (6.8), insert the stoppers and cool in the desiccator (6.9).

8 Procedure

Weigh 10,00 g of the cleaned and dried grains of each sample into separate conical flasks (6.4). Add 50 ml of the grade 2 water (5.1) into each by means of a pipette (6.3). Pipette 50 ml of the grade 2 water into another conical flask to serve as a reference solution. Distribute the grains evenly over the flat bases of the flasks by gently shaking them.

Cap the flasks with neutral glass or aluminium foil rinsed with water grade 2 or inverted beakers (6.5) so that the inner bases of the beakers fit snugly down onto the top rims of the flasks. Place all three flasks in the rack in the autoclave (6.18), containing water at ambient temperature, and ensure that they are held above the level of the water in the vessel.

The autoclave is run in such a way that the temperature in the flasks follows a thermal cycle with the following characteristics: temperature raised from room temperature to 100 °C within 20 – 30 min; temperature maintained at (100 ± 1) °C for (10 ± 1) min; temperature in the containers raised from 100 °C to 121 °C within 20 min to 22 min; temperature maintained at (121 ± 1) °C for (30 ± 1) min; temperature cooled to 100 °C within 40 min to 44 min.

Close the autoclave door or lid securely and run the autoclave to achieve the target thermal cycle in the containers. Where a manual autoclave is run, leave the vent cock open. Heat the autoclave at a regular rate so that steam issues vigorously from the vent cock after 20 min to 30 min, and maintain a vigorous evolution of steam for a further 10 min.

Close the vent cock, follow the temperature increase on the calibrated thermocouple measuring device by comparison with readings taken from the autoclave thermometer and adjust the autoclave settings accordingly in order to match the target thermal cycle. Keep the temperature ramp as smooth as possible.

Using the calibrated thermocouple measuring device, ensure that deviations from the holding temperature of (121 ± 1) °C are within the tolerance. When cooling down, vent to prevent the formation

of a vacuum. For safety reasons (boiling retardation) do not open the autoclave before the water in the containers has reached a temperature of 95 °C.

Remove the flasks from the autoclave, cool the flasks in running water as soon as possible, avoid thermal shock, and complete the titration within 1 h.

Add 0,05 ml of methyl red indicator solution (5.3) to each flask and titrate immediately with the hydrochloric acid solution (5.2) until the colour matches exactly that of the 50 ml of the water of the reference solution plus 0,05 ml of indicator contained in a similar conical flask.

When necessary, for obtaining a clearer end-point, the clear solution should be decanted into a separate 250 ml flask. Rinse the grains by swirling them in three separate 15 ml portions of the grade 2 water and add the washings to the main solution. Add four more drops of the methyl red indicator solution (5.3). Then titrate and calculate the result as described below. In this case, add also 45 ml of grade 2 water and four more drops of methyl red indicator solution to the reference solution.

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

$[c(\text{HCl}) = 0,02 \text{ mol/l} \triangleq 620 \text{ } \mu\text{g} \text{ of sodium oxide}]$

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 0,08 ml of hydrochloric acid solution [$c(\text{HCl}) = 0,02 \text{ mol/l}$] per gram of glass grains equivalent to 49,6 μg of sodium oxide per gram of glass grains (class HGA 1) would be:

Glass, hydrolytic resistance grain class ISO 720 — HGA 1

10 Test report

The test report shall include the following information:

- a reference to this document, i.e. ISO 720:2020;
- an identification of the sample;
- the consumption of hydrochloric acid solution [$c(\text{HCl}) = 0,02 \text{ mol/l}$], in millilitres per gram of glass grains, mean value;