International Standard 720

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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

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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 720 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*.

ISO 720 was first published in 1981. This second edition cancels and replaces the first edition, of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

This International Standard 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 may also be expressed by the amount of sodium oxide equivalent to this volume of acid;

b) a classification of glass according to the hydrolytic resistance determined by the method of this International Standard.

This International Standard is intended for use on the more resistant types of glass. For the less resistant glasses, the method specified in ISO 719 is preferable.

NOTE — It is emphasized that there is no exact correlation between the classification laid down in this International Standard and that laid down in ISO 719, and it is therefore essential to identify which classification is being used.

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 385/2, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

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

ISO 648, *Laboratory glassware — One-mark pipettes.*

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

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

ISO 3696, *Water for laboratory use — Specifications.* [1]

ISO 3819, *Laboratory glassware — Beakers.* [1]

3 Principle

The method of test is a test for glass as a material applied on glass grains. *Extraction of 10 g of grains,* of particle size between 300 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.

4 Reagents

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

4.1 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 (5.6).*

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

When tested immediately before use the water shall be neutral to methyl red, i.e. it shall produce an orange-red (not a violet-red or yellow) colour corresponding to pH 5,5 ± 0,1 when four drops of the methyl red indicator solution (4.3) are added to 50 ml of the water.

NOTE — The water, so coloured, may also be used as a reference solution (see clause 7).

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

4.3 Methyl red, indicator solution.

Dissolve 25 mg of the sodium salt of methyl red ($C_{15}H_{14}N_3NaO_2$) in 100 ml of the grade 2 water (4.1).

4.4 Acetone (CH_3COCH_3).

1) At present at the stage of draft.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Balance, accurate to ± 5 mg or better.

5.2 Burettes, having a capacity of 25 ml, 10 ml or 2 ml, complying with the requirements specified for class A burettes in ISO 385/2 (see also general requirements specified in ISO 385/1) and made of glass of hydrolytic resistance grain class HGA 1 as specified in this International Standard.[1)]

The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid (4.2).

5.3 Pipettes, having a capacity of 50 ml and complying with the requirements specified for class A pipettes in ISO 648.

5.4 Conical flasks, having a capacity of 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 clause 7.

NOTE — Flasks made from vitreous silica may also be used, in which case pretreatment is not required.

5.5 Beakers, having a capacity of 50 ml and complying with the requirements of ISO 3819. Before use, each new beaker shall be pretreated by subjecting it to the autoclaving conditions described in clause 7.

5.6 Boiling flasks, having a capacity of 1 000 ml, complying with 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 7.

5.7 Beakers, having a capacity of 100 ml and complying with the requirements of ISO 3819.

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

5.9 Desiccator.

5.10 Hammer, having a mass of about 0,5 kg.

5.11 Mortar and pestle, made of hardened magnetic steel, and of the design and approximate dimensions shown in the figure.

Approximate dimensions in millimetres

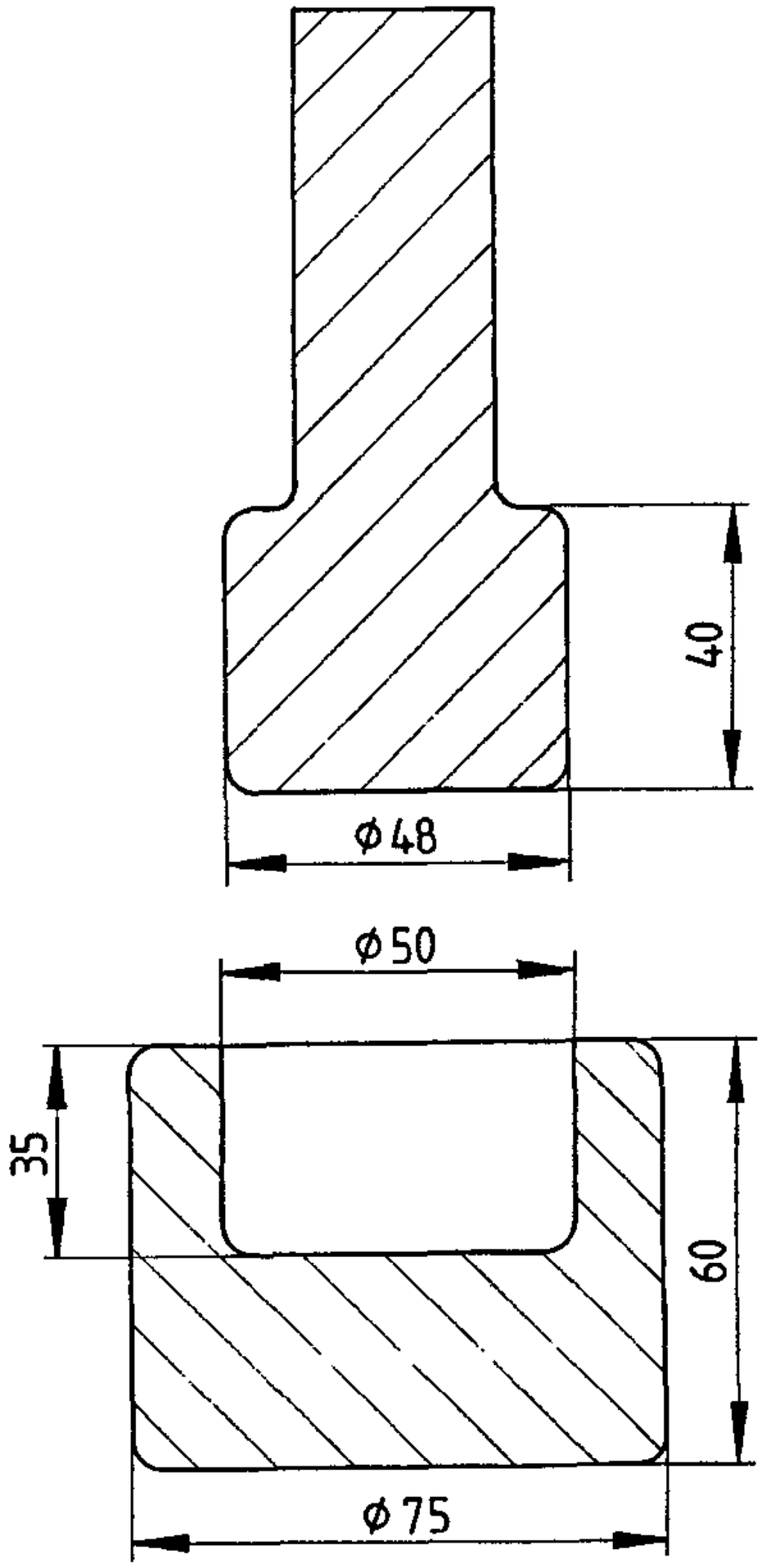

Figure — Mortar and pestle

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

5.12 Magnet.

5.13 Sieves, complying with 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 convenient aperture between 600 and 1 000 µm.

The cover, pan and, especially, the rings shall be of stainless steel or lacquered wood.

NOTE — The use of sieve O is recommended to retain larger pieces of glass and to avoid heavy wear on sieve A.

5.14 Ball-mill

The mill shall be made of agate 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.

5.15 Sieving-machine

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

5.16 Ultrasonic cleaner (laboratory type).

5.17 Drying oven, suitable for operation up to 150 °C.

5.18 Autoclave or **steam sterilizer,** capable of withstanding a pressure of at least $2{,}5 \times 10^5$ N/m²* and of carrying out the heating cycle specified in clause 7. It should preferably be equipped with a constant-pressure regulator or other means of maintaining the temperature at 121 ± 1 °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 flasks.

The autoclave vessel and ancillary equipment shall be thoroughly cleaned before use.

6 Preparation of sample

6.1 Density of the glass

The density of the glass to be tested should, preferably, be 2,4 ± 0,2 g/cm³ at 20 °C.

6.2 Crushing

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

NOTE — 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, may 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, which should, preferably, have a wall thickness greater than 1,5 mm, in clean paper and crush to produce three 100 g samples of pieces not more than 30 mm across.

6.3 Manual preparation

Place 30 to 40 g of pieces between 10 and 30 mm across, taken from a 100 g sample (see 6.2), in the mortar (5.11), insert the pestle (5.11), and strike it sharply, once only, with the hammer (5.10).

NOTE — 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 (5.13). 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 O and from the receiving pan.

Reassemble the set of sieves and shake by hand for 5 min. Transfer to the weighing bottle (5.8) those glass grains which pass through sieve A, but which are retained on sieve B.

Repeat the crushing and sieving procedure with the other two 100 g samples and thus three 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 (5.12). Transfer each sample into a beaker (5.7) for cleaning.

6.4 Mechanical preparation

Transfer about 50 g of the coarsely broken glass (see 6.2) into the mill-beaker (5.14), add the balls and crush thin-walled glass (wall thickness < 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 (5.13) of the sieving machine (5.15), sieve for about 30 s and

* $2{,}5 \times 10^5$ N/m² = 0,25 MPa = 2,5 bar

collect the grains retained on sieve B in the beaker (5.7), which shall be kept in the desiccator (5.9). 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 11 g of grains have been collected from sieve B. Continue as specified in 6.3, last paragraph.

6.5 Cleaning

Add to the grains in each beaker (5.7) 30 ml of the acetone (4.4) and scour the grains by a suitable means, such as a rubber- or plastics-coated glass rod.

NOTE — The method of scouring involves holding the beaker at an angle of about 30° to 45° firmly against the bench and pressing firmly the covered end of a glass rod of about 10 mm in diameter into the bottom corner and against the sides, so that the grains are trapped between it and the sides and the bottom of the beaker as the rod is rotated around the beaker. Continue the rotation for about 20 revolutions.

After scouring, swirl the grains and decant as much acetone as possible. Add another 30 ml of the acetone, swirl and decant again and add a new portion of the acetone. Fill the bath of the ultrasonic cleaner (5.16) 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 ultrasonics for 1 min.

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 to remove excess acetone and then by heating at 140 °C for 20 min in the drying oven (5.17).

Transfer the dried grains from each beaker to separate weighing bottles (5.8), insert the stoppers and cool in the desiccator (5.9).

7 Procedure

Weigh 10,00 g of the cleaned and dried grains of each sample into separate conical flasks (5.4). Add 50 ml of the grade 2 water (4.1) into each by means of a pipette (5.3). 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 inverted beakers (5.5) so that the inner bases of the beakers fit snugly down onto the top rims of the flasks. Place all four flasks in the rack in the autoclave (5.18), containing water at ambient temperature, and ensure that they are held above the level of the water in the vessel. Close the autoclave lid securely, but leave the vent-cock open. Heat at a regular rate such that steam issues vigorously from the vent-cock after 20 to 30 min, and maintain a vigorous evolution of steam for a further 10 min. Close the vent-cock and increase the temperature at the rate of 1 °C/min to 121 °C. Maintain the temperature at 121 ± 1 °C for 30 ± 1 min from the time when the holding temperature is reached, then cool at a rate of 0,5 °C/min to 100 °C, venting to prevent formation of a vacuum.

Remove the flasks from the autoclave, cool the flasks in running water and complete the titration within 1 h.

Add four drops of methyl red indicator solution (4.3) to each flask and titrate immediately with the hydrochloric acid solution (4.2) until the colour matches exactly that of the 50 ml of the water of the reference solution plus four drops of indicator contained in a similar conical flask.

NOTE — 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 (4.3). 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.

8 Expression of results

8.1 Calculation

Calculate the mean value of the results, in millilitres of hydrochloric acid solution (4.2) 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(HCl) = 0,02 mol/l] ≙ 620 µg 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.

8.2 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 International Standard.

8.3 Designation

For convenience of reference to the hydrolytic resistance of glass as a material complying with the classification of this International Standard, 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(HCl) = 0,02 mol/l] per gram of glass grains equivalent to 49,6 µg of sodium oxide per gram of glass grains (class HGA 1) shall be:

Glass, hydrolytic resistance grain class ISO 720 - HGA 1

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) an identification of the sample;

c) the consumption of hydrochloric acid solution [c(HCl) = 0,02 mol/l], in millilitres per gram of glass grains, mean value;

d) in addition, if required, the equivalent of alkali, in micrograms of sodium oxide per gram of glass grains, mean value;

e) the hydrolytic resistance grain class HGA (designation of the glass tested);

f) the wall thickness of the articles used for the test if it was < 1,5 mm;

g) the density of the glass if it was outside the range of 2,4 ± 0,2 g/cm³ at 20 °C;

h) 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(HCl) = 0,02 mol/l] (4.2) per gram of glass grains ml/g	Permissible range of the values obtained
up to and including 0,10	25 % 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 grain test (autoclave test)

Class[1)]	Consumption of hydrochloric acid solution [c(HCl) = 0,02 mol/l] (4.2) per gram of glass grains ml/g	Equivalent of alkali expressed as mass of sodium oxide (Na_2O) per gram of glass grains µg/g
HGA 1	up to and including 0,10	up to and including 62
HGA 2	from 0,10 up to and including 0,85	from 62 up to and including 527
HGA 3	from 0,85 up to and including 1,50	from 527 up to and including 930

1) "HGA" stands for the hydrolytic resistance of glass grains according to the autoclave test method.