# 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.



International Standard ISO 720 was developed by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, and was circulated to the member bodies in November 1979.



It has been approved by the member bodies of the following countries:



| | | |
|---|---|---|
| Australia | Hungary | Romania |
| Canada | India | Spain |
| Egypt, Arab Rep. of | Korea, Rep. of | United Kingdom |
| France | Netherlands | USSR |
| Germany, F. R. | Poland | |

The member body of the following country expressed disapproval of the document on technical grounds :

Czechoslovakia

This International Standard cancels and replaces ISO Recommendation R 720-1968 of which it constitutes a technical revision.

# 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 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.

NOTES

1 The test method of this International Standard is recommended for use on all types of glass. For the less resistant glasses, the method specified in ISO 719 is also suitable.

2 It must be emphasised that there is no exact correlation between the classification of this International Standard and the classification of 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.* [1)]

ISO 385/2, *Laboratory glassware — Burettes — Part 2 : Burettes for which no waiting time is specified.* [1)]

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal size of apertures.*

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).*

## 3 Reagents

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

**3.1 Distilled or deionized water,** of high purity, complying with the following requirements when tested immediately before use : it shall be free from dissolved gases and heavy metals, particularly copper, as shown by the dithizone test; it shall have a specific conductivity not exceeding $1 \times 10^{-4}$ S/m at 20 °C; and it shall be neutral to methyl red.

**3.2 Acetone,** $CH_3COCH_3$, pure.

**3.3 Sodium hydroxide,** standard volumetric solution, [$c$(NaOH) = 0,02 mol/l].

The solution shall be carbonate-free, and shall be standardized immediately before use against potassium hydrogen phthalate ($C_8H_5O_4K$), using a phenolphthalein indicator solution prepared by dissolving 0,5 g of phenolphthalein, $C_{20}H_{14}O_4$, in 60 ml of ethyl alcohol, [$C_2H_5OH$, 95 % ($V/V$)], and diluting with water to 100 ml.

**3.4 Hydrochloric acid,** standard volumetric solution, [$c$(HCl) = 0,02 mol/l].

**3.5 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 water (3.1).

## 4 Apparatus

Ordinary laboratory apparatus and

**4.1 Autoclave** or **steam sterilizer,** capable of withstanding a pressure of $1,7 \times 10^5$ N/m$^2$ * and of carrying out the heating cycle specified in clause 6. It should preferably be equipped with a constant-pressure regulator or other means for maintaining the temperature at 121 ± 1 °C. The vessel shall be capable of containing at least six 250 ml conical flasks, and shall be equipped with a rack for supporting the flasks, a thermometer, a pressure gauge and a vent cock.

**4.2 Balance,** accurate to ± 5 mg or better.

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

1) At present at the stage of draft. (Revision, in part, of ISO/R 385.)

* $1,7 \times 10^5$ N/m$^2$ = 0,17 MPa = 1,7 bar

**4.4 Hammer,** mass about 1 kg.

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

**4.6 Magnet.**

**4.7 Sieves :** a set of 200 mm diameter square-aperture sieves, with stainless steel mesh, including :

— a sieve (A)[1] of 420 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.

**4.8 Beakers,** 50 ml, made of chemically resistant glass. New beakers shall be aged by treating three times in accordance with the heating procedure specified in clause 6.

**4.9 Burettes,** of suitable capacity as follows :

— 25 ml, complying with the requirements of class A of ISO 385/2;

— 10 ml, graduated in 0,02 ml;

— 2 ml, graduated in 0,01 ml.

The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid (3.4).

**4.10 Pipette,** 50 ml, complying with the requirements of class A of ISO 648.

**4.11 Conical flasks,** 250 ml, complying with the requirements of ISO 1773.

New flasks shall be aged by treating three times in accordance with the heating procedure specified in clause 6.

**4.12 Stoppered storage vessels** (desiccators).

**4.13 Weighing bottles,** stoppered, about 20 ml capacity.

**4.14 Cooling bath.**

Approximate dimensions in millimetres

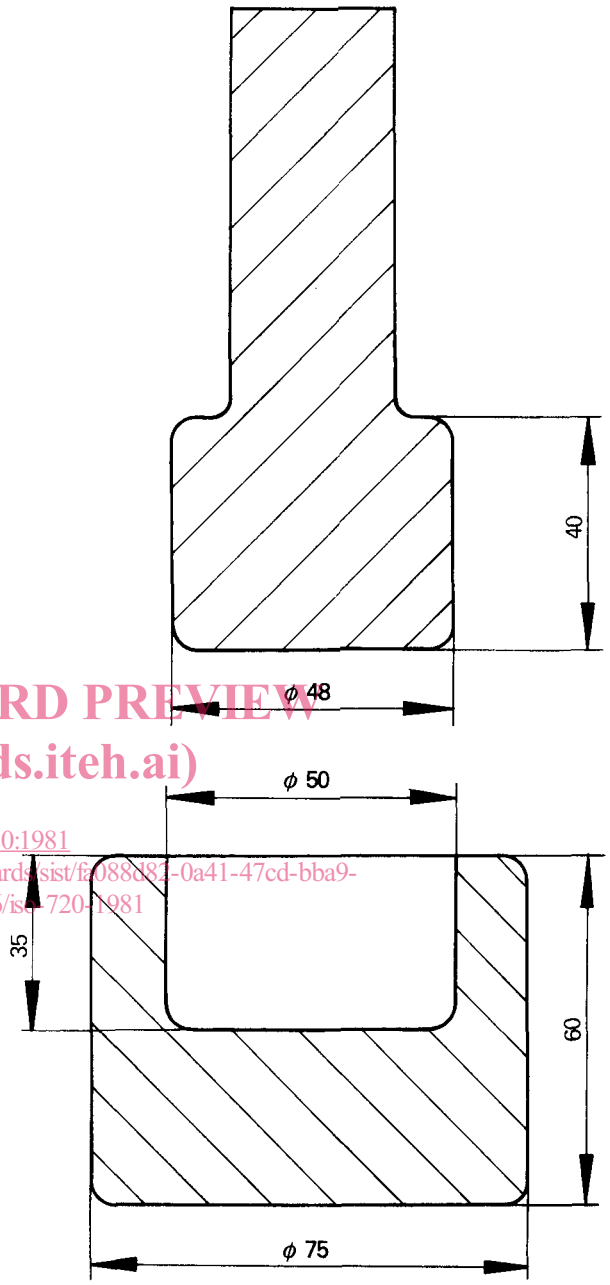


**Figure — Hardened steel mortar and pestle**



## 5 Preparation of sample

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

NOTE — If an article is not annealed to a commercial acceptable quality, this fact should be noted because the results can be affected.

Further annealing should not be carried out before test.

1) The aperture size of sieve A is subject to reconsideration since the size 420 µm has not been included in ISO 565.

Then wrap the glass articles, which should preferably have a wall thickness greater than 1,5 mm, in clean paper and crush to pieces not over 25 mm diameter. Place 30 to 40 g of the coarsely crushed sample in the hardened steel mortar (see 4.5), insert the pestle and strike it sharply, once only, with the hammer (4.4).

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

Empty the contents of the mortar into the assembled set of sieves (4.7). Repeat the crushing procedure until the whole of the 100 g sample has been added to the set of sieves. Shake the sieves for a short time by hand and then remove the glass from sieves A and O and repeat the crushing and sieving operations 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, or on a mechanical sieve-shaker for a time and at an amplitude equivalent to the hand sieving. Reserve for the test the glass grains which pass through sieve A, but are retained on sieve B.

Repeat the crushing and sieving procedure with two additional 100 g samples and thus obtain three samples of grains, each of which shall be in excess of 10 g. Spread each sample on a piece of clean glazed paper and pass the magnet (4.6) through the grains to remove any iron particles. Transfer each sample to a separate storage vessel (4.12), and insert the stopper. The storage time shall not exceed 24 h.



## 6 Procedure

Transfer approximately 11 g of the freshly prepared sample into each of three of the conical flasks (4.11). Remove any adherent fine particles by swirling the grains six times in separate 30 ml portions of the acetone (3.2), decanting as much acetone as possible after each washing. Place the flasks and contents on a warm plate to remove excess acetone and then place in the drying oven (4.3), controlled at 140 °C, for 20 min. Remove the flasks from the oven, transfer the grains from each flask to separate weighing bottles (4.13) and allow the stoppered bottles to cool in a desiccator (4.12).

Transfer a 10,00 g portion of the prepared sample from each weighing bottle to a separate conical flask and add 50 ml of distilled water into each by means of a pipette (4.10). Pipette 50 ml of distilled water into another conical flask to serve as a blank test.

Cap the flasks with inverted beakers (4.8) 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 (4.1), containing about 1 litre of water, and ensure that they are held above the level of the water in the chamber. Close the autoclave lid securely, but leave the vent cock open. Heat until steam issues vigorously from the vent cock for 10 min. Close the vent cock and increase the temperature at 1 °C per minute to 121 °C. Maintain the temperature at 121 ± 1 °C for 30 min from the time when the holding temperature is reached, then cool at 0,5 °C per minute to 100 °C, venting to prevent formation of a vacuum.

Remove the flasks from the autoclave, place in the cooling bath (4.14), and cool in running water.

Add 5 drops of the methyl red indicator solution (3.5) to each flask and titrate immediately with the hydrochloric acid solution (3.4).

NOTE — In order to obtain 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 25 ml portions of distilled water and add the washings to the main solution. Then titrate and calculate the result as described above. In this case, add 75 ml of distilled water to the blank test solution.

## 7 Expression of results

Subtract the blank test value from the three values obtained for the samples and calculate the mean value of the results per gram of sample; report this value 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.

NOTE — If the wall thickness of the articles used for the test is less than 1,5 mm, or if the density of the glass is outside the range 2,4 ± 0,2 g/ml at 20 °C, these values should also be reported.

## 8 Classification and designation

**8.1** Glass shall be classified as shown in the table, according to the consumption of acid and its equivalent of alkali (expressed as $Na_2O$), when tested by the method specified in this International Standard.

**Table — Classification**

| Class | Consumption of HCl solution (0,02 mol/l) (3.4) per gram of glass grains | Equivalent of alkali expressed as mass of $Na_2O$ per gram of glass grains |
|---|---|---|
| | ml/g | µg/g |
| 1 | up to 0,10 | up to 62 |
| 2 | above 0,10 up to 0,85 | above 62 up to 527 |
| 3 | above 0,85 up to 1,50 | above 527 up to 930 |

**8.2** For convenience of reference to the hydrolytic resistance of glass complying with the classification of this International Standard, the use is recommended of a designation as follows :

*Example :* For a glass with a consumption of 0,08 ml of HCl solution [$c$(HCl) = 0,02 mol/l] per gram of glass grains equivalent to 49,6 µg of $Na_2O$ per gram of glass grains (class 1) :

Glass, hydrolytic resistance class ISO 720-1