International Standard 719

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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

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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 719 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	Poland
Canada	India	Romania
Egypt, Arab Rep. of	Italy	Spain
France	Korea, Rep. of	United Kingdom
Germany, F. R.	Netherlands	USSR

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 719-1968 of which it constitutes a technical revision.

Printed in Switzerland

Glass — Hydrolytic resistance of glass grains at 98 °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 98 °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 the less resistant types of glass. For the more resistant glasses, the method specified in ISO 720 is preferable.

2 It must be emphasised that there is no exact correlation between the classification of this International Standard and the classification of ISO 720, and it is therefore essential to identify which classification is being used.

2 References

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

ISO 648, *Laboratory glassware — One-mark 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 — Boiling flasks (narrow-necked).*

3 Reagents

During the analysis, 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 1×10^{-4} S/m at 20 °C; and it shall be neutral to methyl red.

3.2 Hydrochloric acid, standard volumetric solution [c(HCl) = 0,01 mol/l].

3.3 Buffer solution, pH = 5,2.

Add 92,8 ml of a citric acid solution [$c(C_6H_8O_7)$ = 0,1 mol/l] to 107,2 ml of a *di*sodium hydrogen phosphate solution [$c(Na_2HPO_4)$ = 0,2 mol/l], or alternatively, dissolve 1,02 g of potassium hydrogen phthalate ($C_8H_5KO_4$) in 30 ml of sodium hydroxide solution [c (NaOH) = 0,1 mol/l] and add carbonate-free water to 100 ml.

3.4 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 Balance, accurate to ± 5 mg or better.

4.2 Burettes, of suitable capacity, as follows :

— 5 ml, graduated in 0,02 ml;

— 2 ml or 1 ml, graduated in 0,01 ml.

The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid (3.2).

4.3 Pipette, 25 ml, complying with the requirements of class A of ISO 648.

4.4 One-mark volumetric flasks, 50 ml, complying with the requirements of class A of ISO 1042, made of chemically resistant glass, and with glass stoppers. It is advisable to select flasks with the graduation line in the lower half of the neck. Before use, each new flask shall be pre-treated by filling to above the graduation mark with water and treating three times in accordance with the heating procedure specified in clause 6, using a fresh quantity of water in the flask each time.

NOTE — Flasks made from vitreous silica may also be used, in which case the pre-treatment is not required.

4.5 Conical flasks, 100 ml, complying with the requirements of ISO 1773. New flasks shall be pre-treated by filling to the base of the neck with water and treating as specified in 4.4.

4.6 Stoppered storage vessel (desiccator).

4.7 Hammer, mass about 1 kg.

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

4.9 Magnet.

4.10 Cooling bath, of capacity sufficient to contain 1 litre of water for each flask used in the test.

4.11 Sieves : 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 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 of sieve A.

4.12 Thermometer, covering the range of 90 to 110 °C, capable of being read to an accuracy of ± 0,2 °C.

4.13 Heating bath, gas or electrically heated, thermostatically controlled, of capacity sufficient to contain 1 litre of liquid for each flask used in the test and capable of carrying out the heating cycle specified in clause 6.

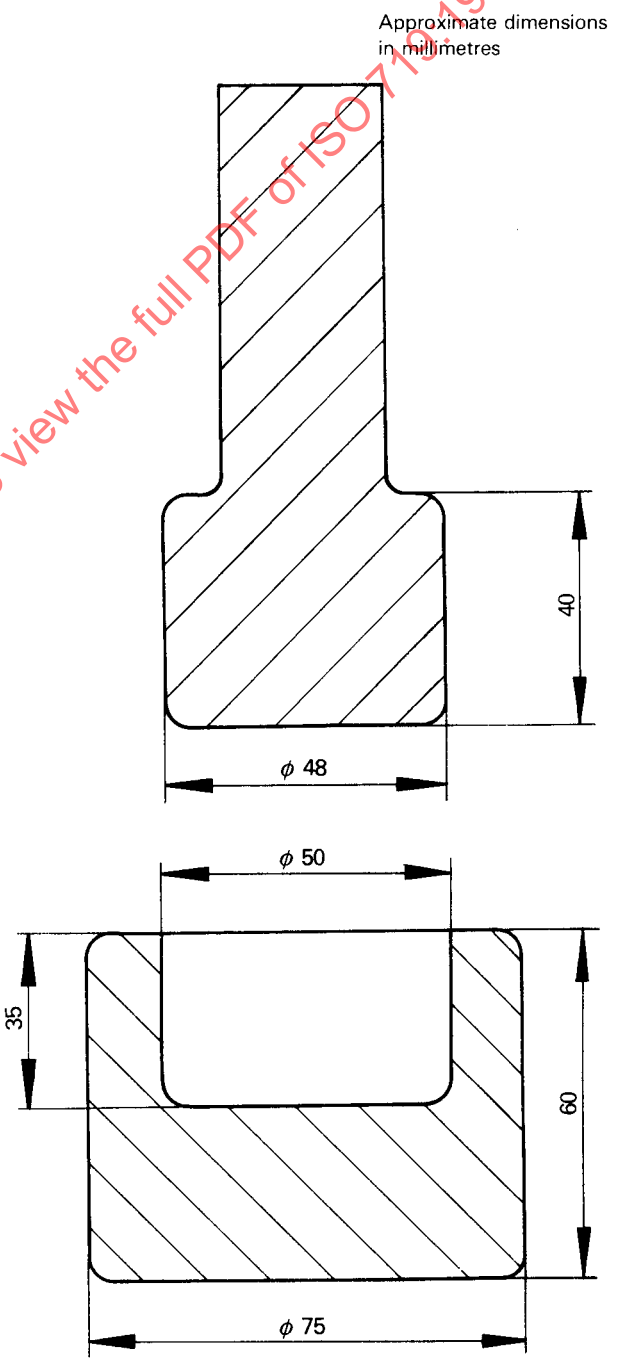

Figure — Hardened steel mortar and pestle

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.

Then wrap the glass articles, which should preferably have a wall thickness greater than 1,5 mm, in clean paper and break them with a few blows of the hammer (4.7). Transfer at least 30 g of pieces between 10 and 30 mm diameter to the hardened steel mortar (see 4.8), insert the pestle and strike it sharply, once only, with the hammer.

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.

Transfer the glass from the mortar to the upper sieve (see 4.11) and shake the set of sieves briefly to separate the finer particles. Return to the mortar the glass remaining on sieves A and O and repeat the crushing and sieving until only about 10 g of glass remain on sieve O. Discard the glass from sieve O and from the receiving pan. Shake the set of sieves by hand for 5 min. Reserve for the test the grains which pass through sieve A, but are retained on sieve B.

At least 10 g of sample is required for the test. If it is necessary to crush and sieve more sample, it is essential that the sample already obtained should be removed from sieve B and not sieved again.

After completion of all crushing and sieving, combine the samples, spread the grains on clean glazed paper and pass the magnet (4.9) through them to remove any iron particles. Transfer the sample to the storage vessel (4.6) and insert the stopper. The storage time shall not exceed 24 h.

6 Procedure

Transfer 2,00 g of the freshly prepared sample into each of three of the volumetric flasks (4.4). Remove any adherent fine particles by swirling the grains six times in separate 30 ml portions of water (3.1), decanting as much water as possible after each washing. Fill the flasks with water to the mark and fill two more flasks with distilled water, one to serve as a blank test 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 four flasks, without stoppers, in the heating bath (4.13), 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 the specified temperature of 98 ± 0,5 °C is achieved in the control flask within 3 min; after 5 min, insert the stoppers. Continue the heating for 60 min from the time of immersion, maintaining the temperature at 98 ± 0,5 °C in the flasks.

Remove the flasks from the bath, take out the stoppers, place the flasks in the cooling bath (4.10), cool in running water and adjust the contents of each flask to the mark with distilled 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 a pipette (4.3), transfer 25 ml of the clear solution from each flask into separate conical flasks (4.5), add to each of these flasks two drops of the methyl red indicator (3.4) and titrate with the hydrochloric acid solution (3.2), matching the end-point to 25 ml of buffer solution plus 2 drops of indicator solution contained in a similar conical flask. Titrate all three sample solutions and the blank test solution in the same way.

7 Expression of results

Subtract the blank test value from each of 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) = 0,01 mol/l] ≙ 310 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 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 (0,01 mol/l) (3.2) 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 31
2	above 0,10 up to 0,20	above 31 up to 62
3	above 0,20 up to 0,85	above 62 up to 264
4	above 0,85 up to 2,0	above 264 up to 620
5	above 2,0 up to 3,5	above 620 up to 1 085

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) = 0,01 mol/l] per gram of glass grains equivalent to 24,8 µg of Na_2O per gram of glass grains (class 1) :

Glass, hydrolytic resistance class ISO 719-1.