

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 719

DETERMINATION OF THE HYDROLYTIC RESISTANCE
OF GLASS GRAINS AT 98 °C

1st EDITION

May 1968

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BRIEF HISTORY

The ISO Recommendation R 719, *Determination of the hydrolytic resistance of glass grains at 98 °C*, was drawn up by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1950 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In December 1963, this Draft ISO Recommendation (No. 718) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Greece	Spain
Australia	Hungary	Sweden
Austria	India	Switzerland
Brazil	Israel	Turkey
Canada	Italy	U.A.R.
Chile	Japan	United Kingdom
Colombia	Korea, Rep. of	U.S.S.R.
Czechoslovakia	Netherlands	Yugoslavia
France	New Zealand	
Germany	Poland	

Two Member Bodies opposed the approval of the Draft :

Belgium
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in May 1968, to accept it as an ISO RECOMMENDATION.

DETERMINATION OF THE HYDROLYTIC RESISTANCE OF GLASS GRAINS AT 98 °C

1. SCOPE

The purpose of this ISO Recommendation is to describe a method for determining the hydrolytic resistance of glass grains at 98 °C. The resistance is measured inversely by the volume of acid required for titration of the alkali extracted from unit mass of glass, and is expressed by the amount of sodium oxide equivalent to this volume of acid.

This method is recommended for use on the less resistant types of glass. For the more resistant glasses, the method described in ISO Recommendation R 720, *Determination of the hydrolytic resistance of glass grains at 121 °C*, is preferable.

2. APPARATUS

- 2.1 *Balance*, accuracy ± 5 mg or better.
 - 2.2 *Burettes*, 10 ml, graduated in 0.05 ml, and 1 or 2 ml, graduated in 0.01 ml.
 - 2.3 *Cooling bath*, of capacity sufficient to contain 1 litre of water for each flask used in the test.
 - 2.4 *Conical flasks*, 100 ml capacity, made of chemically resistant glass and pre-treated by filling to the base of the neck with water and heating as described in clause 2.5 below.
 - 2.5 *Volumetric flasks*, 50 ml capacity, 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 flask should be pre-treated by filling to above the graduation mark with water and heating to 100 °C in the heating bath for three separate periods of 1 hour, 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.
- 2.6 *Hammer*, weighing about 1 kg.
 - 2.7 *Mortar and pestle*, made of hardened steel and of the design and approximate dimensions shown in the Figure, page 4.
 - 2.8 *Pipette*, 25 ml.

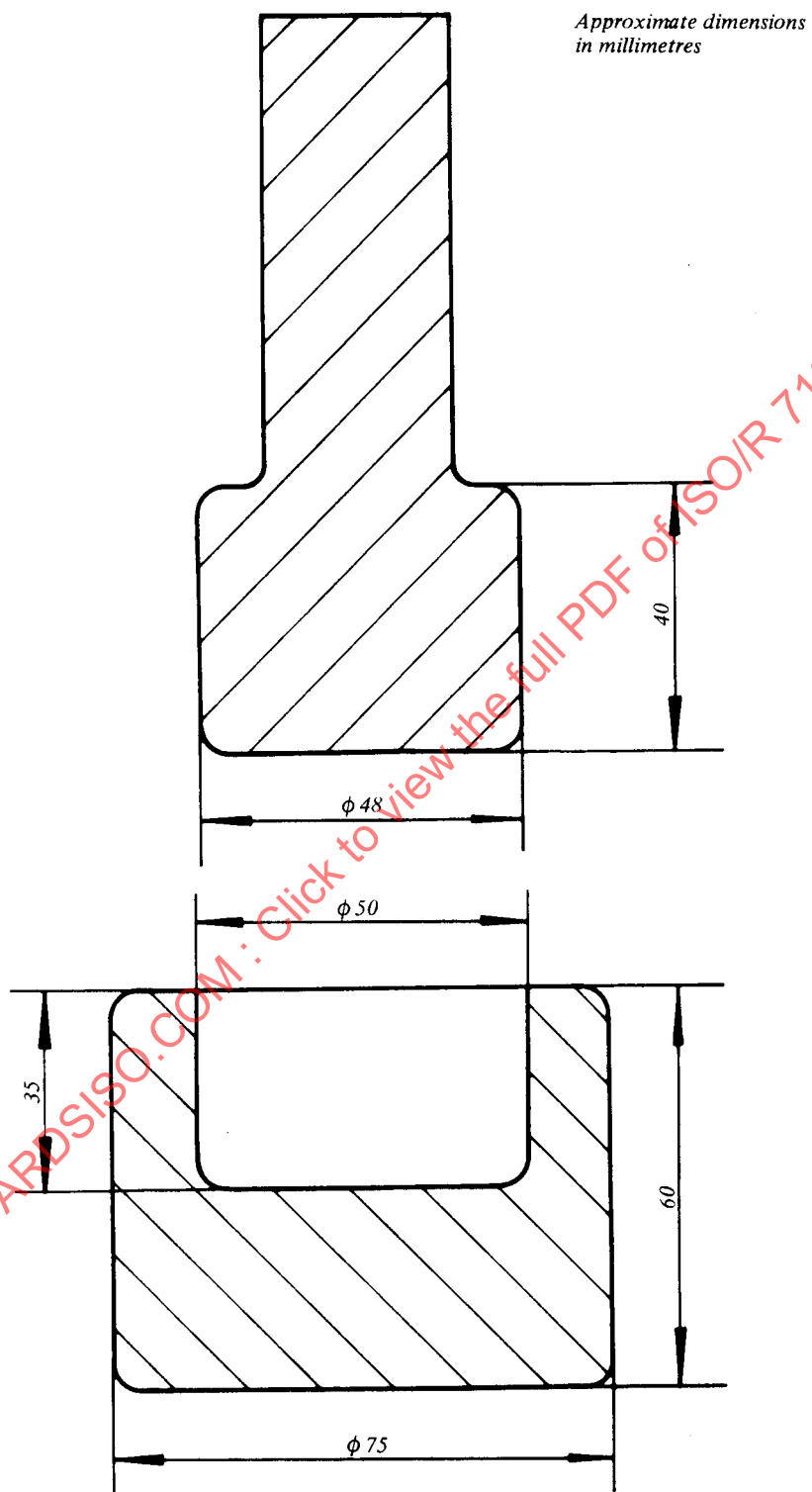


FIGURE — Hardened steel mortar and pestle

2.9 *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 1000 μm .

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

2.10 *Thermometer,* covering the range of 90 to 110 °C, capable of being read to an accuracy of ± 0.2 °C.

2.11 *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 described in section 6.

2.12 *Stoppered storage vessel.*

3. REAGENTS

Analytical grade reagents should be used throughout.

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

3.2 *Citric acid* 0.1 M. Dissolve 21.008 g of solid citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) in water and dilute to 1 litre.

3.3 *Hydrochloric acid*, 0.01 N.

3.4 *Disodium hydrogen phosphate*, 0.2 M. Dissolve 35.60 g of solid disodium hydrogen phosphate ($\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 litre.

3.5 *Buffer solution*, pH = 5.2. Add 92.8 ml of 0.1 M citric acid to 107.2 ml of 0.2 M. disodium hydrogen phosphate.

3.6 *Methyl red indicator.* Dissolve 25 mg of the sodium salt of methyl red in 100 ml of water.

* The aperture size of sieve B is subject to reconsideration, since the size 300 μm has not been included in ISO Recommendation R 565, *Woven wire cloth and perforated plates in test sieves. Nominal sizes of apertures.*

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

4. PREPARATION OF SAMPLE

Wrap the glass articles as received,* which should preferably have a wall thickness greater than 1.5 mm, in clean paper and break them with a few hammer blows. Transfer at least 30 g of pieces between 10 and 30 mm diameter to the hardened steel mortar, insert the pestle and strike it sharply, once only, with the hammer.** Transfer the glass from the mortar to the upper sieve 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 minutes. 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 a magnet through them to remove any iron particles. Transfer the sample to the storage vessel and insert the stopper.

5. PROCEDURE

Transfer 2.00 g of the freshly prepared sample into each of three 50 ml volumetric flasks. Remove any adherent fine particles by swirling the grains six times in separate 30 ml portions of water, decanting as much water as possible after each washing. Fill the flasks with water to the graduation line and fill a fourth flask with distilled water to serve as a blank test. 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 maintained at $98 \pm 0.5^\circ\text{C}$, 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 so that the specified temperature is recovered within 3 minutes; after 5 minutes, when the flasks have been warmed, insert the stoppers. Continue the heating for 60 minutes from the time of immersion, maintaining the heating bath at $98 \pm 0.5^\circ\text{C}$.

Remove the flasks from the bath, take out the stoppers, place the flasks in the cooling bath, cool in running water and adjust the contents of each flask to the graduation line 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, transfer 25 ml of the clear solution from each flask into separate 100 ml conical flasks, add to each of these flasks two drops of methyl red indicator and titrate with 0.01 N hydrochloric acid, matching the end-point to 25 ml of buffer solution plus two drops of indicator contained in a similar conical flask. Titrate all three sample solutions and the blank test solution in the same way.

* The results obtained from the test may differ according to whether the glass is tested in the strained or unstrained condition.

** 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.