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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1776

DETERMINATION OF THE RESISTANCE OF GLASS TO ATTACK BY 6N HYDROCHLORIC ACID AT 100 °C

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BRIEF HISTORY

The ISO Recommendation R 1776, *Determination of the resistance of glass to attack by 6N hydrochloric acid at 100 °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 led to the adoption of Draft ISO Recommendation No. 1776, which was circulated to all the ISO Member Bodies for enquiry in December 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Iran	Thailand
Belgium	Israel	Turkey
Canada	Italy	U.A.R.
Colombia	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.S.R.
France	Peru	U.S.A.
Germany	Poland	Yugoslavia
Greece	South Africa, Rep. of	
India	Spain	

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No Member Body opposed the approval of the Draft.

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

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ISO Recommendation	R 1776	October 1970

DETERMINATION OF THE RESISTANCE OF GLASS TO ATTACK BY 6N HYDROCHLORIC ACID AT 100 °C

1. SCOPE

1.1 This ISO Recommendation describes a procedure for determining the resistance of laboratory glassware to attack by an aqueous solution of 6N hydrochloric acid at 100 °C. The resistance is measured inversely by the amount of alkali oxide extracted per unit area of the glass.

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1.2 The method may be applied to pieces, preferably flat but if necessary curved, cut from articles of laboratory glassware. It enables the acid resistance to be measured either of *the glass as delivered* (by omitting the preliminary acid treatment described in clause 4.3) or of *the glass as a material.* This distinction is especially significant in the case of surface-treated soda-lime glasses, but does not apply to heat-resisting borosilicate glasses, for which the acid-etching procedure is therefore unnecessary.

2. APPARATUS

2.1 *Drying ovens*

(*a*) suitable for adjustment to 100 ± 1 °C, and

(*b*) suitable for operation at a temperature of 100 to 110 °C.

2.2 *Flame photometer* or *atomic absorption spectrophotometer.*

2.3 *Measuring instruments,* suitable for measuring lengths and diameters to an accuracy of 0.2 mm.

2.4 *Platinum dishes*

A : about 90 ml capacity with a top diameter of about 70 mm, and

B : about 150 ml capacity with a top diameter of about 100 mm. A beaker cover of polypropylene may be used in place of the platinum dish B as shown in Figure 1.

2.5 *Test vessel,* of about 2 litres capacity, for example a Witt's filtration apparatus, with ground rims and convex lid and with a stand on which to rest the platinum dishes, as shown in Figure 1, or a suitable alternative.

2.6 *Tongs,* tipped with rubber or a suitable chemically resistant plastics material. The tips should be pre-treated by boiling in hydrochloric acid and should be washed in distilled water immediately before use.

3. REAGENTS

Highest quality reagents, with alkali content of less than 5 μg per 25 ml, should be used throughout.

3.1 *Deionized water* or *distilled water*, to be used wherever *water* is mentioned below. Distilled water should be freshly prepared for each test.

3.2 *Ethanol* or *acetone.*

3.3 *Hydrochloric acid,* 2N solution.

3.4 *Hydrochloric acid,* 6 ± 0.2N solution freshly prepared for each test as follows :

Use the assembly shown in Figure 3, fitting an anti-splash device to the boiling flask and ensuring that the plastics tubing does not enter the water in the collecting flask. Add hydrochloric acid (d = 1.18) to the boiling flask, add water to the collecting flask, connect up the assembly and heat to boiling. Allow only the hydrochloric acid vapour to pass over into the water. As soon as liquid begins to distil replace the acid with a fresh supply and continue the distillation until the acid concentration in the collecting flask exceeds 6N. Adjust to 6 ± 0.2N by dilution.

3.5 *Hydrofluoric acid,* 400 g/l solution.

4. PREPARATION OF SAMPLE

4.1 Test sample

The sample piece or pieces should have a total surface area of 30 to 40 cm^2, should be well annealed and should preferably be not more than 2 mm thick. If thicker pieces are tested, the actual thickness should be stated in the test report.

Measure all dimensions to the nearest 0.2 mm and calculate the actual surface area.

4.2 Washing procedure (to be included when the resistance of glass *as delivered* is required.)

Wash the sample piece or pieces thoroughly with water (3.1), then rinse with ethanol or acetone (3.2). Dry the sample in the oven (2.1 (b)) for 30 minutes at 100 to 110 °C.

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4.3 Preliminary acid treatment (to be included when the resistance of glass *as a material* is required.)

Place the sample piece or pieces in a platinum wire rack fitted inside a 250 ml plastics beaker, as shown in Figure 2. Introduce a plastics-coated magnetic rotor and then add carefully, down the wall of the beaker, a mixture containing 1 part of hydrofluoric acid (3.5) and 9 parts of 2N hydrochloric acid (3.3) until the sample is completely immersed. The temperature of the solution should be about 20 °C. Stir the solution magnetically for 1 minute. Holding the sample in position with a plastics rod, pour out the acid mixture. Fill the beaker with water and again decant the liquid. Remove the sample, using the tipped tongs, and wash each piece thoroughly in water. Dry the sample for 30 minutes in the oven (2.1 (b)) at 100 to 110 °C.

5. PROCEDURE

5.1 Preparation of the test solution

Prepare the apparatus as shown in Figure 1 and add 25 ml of 6N hydrochloric acid (3.4) to the platinum dish A.

Heat the assembly in the oven (2.1 (a)) to 100 °C. Using the tongs transfer the hot sample from the drying oven (2.1 (b)) at 100 to 110 °C to the rack in the platinum dish A, making sure that it is completely immersed in the acid. Cover the dish with platinum dish B (or a beaker cover of polypropylene), replace the lid of the test vessel and maintain the temperature of oven (2.1 (a)) at 100 ± 1 °C for 3 hours.

Remove the sample and the rack from the dish, again using the tongs, wash thoroughly with a jet of water and add the washings (about 25 ml) to dish A.

5.2 Determination of alkali oxide

Transfer dish A to a boiling water bath and evaporate the solution to about 5 ml. Add 1 drop of hydrofluoric acid (3.5) and continue the evaporation to dryness. Dissolve the residue in 0.2 ml of 2N hydrochloric acid (3.3), add a few millilitres of water, transfer to a 10 ml volumetric flask, dilute to the mark and mix thoroughly.

Determine the alkali oxide content of the sample solution by flame photometry or atomic absorption spectrophotometry and calculate the mass of alkali extracted per 100 cm^2 surface area of glass.

5.3 Blank determination

Carry out a blank determination by proceeding as in clauses 5.1 and 5.2 above, but omitting the sample.

6. EXPRESSION OF RESULTS

Calculate the mean value from three determinations, subtracting the value obtained in the blank determination in each case. Report the result either as the mass of sodium oxide (Na_2O) or, if required, as the masses of the separate alkali oxides extracted per 100 cm^2 of attacked surface. Report also the thickness of the glass, if greater than 2 mm, and whether the preliminary acid treatment (see clause 4.3) has been applied.

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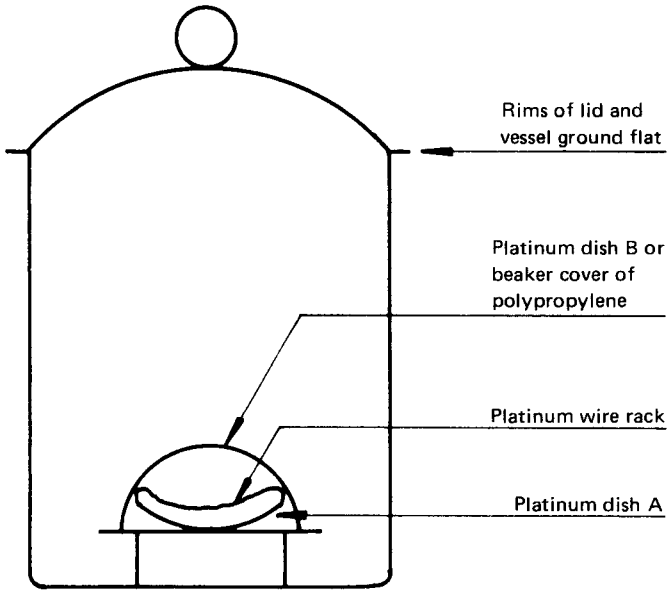

FIG. 1 – **Assembly of apparatus**

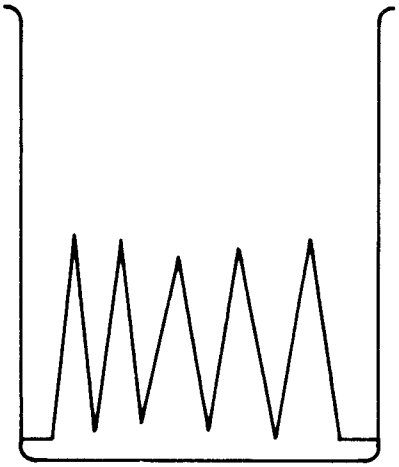

FIG. 2 – **Plastics beaker with platinum wire rack to hold samples during the preliminary acid treatment**

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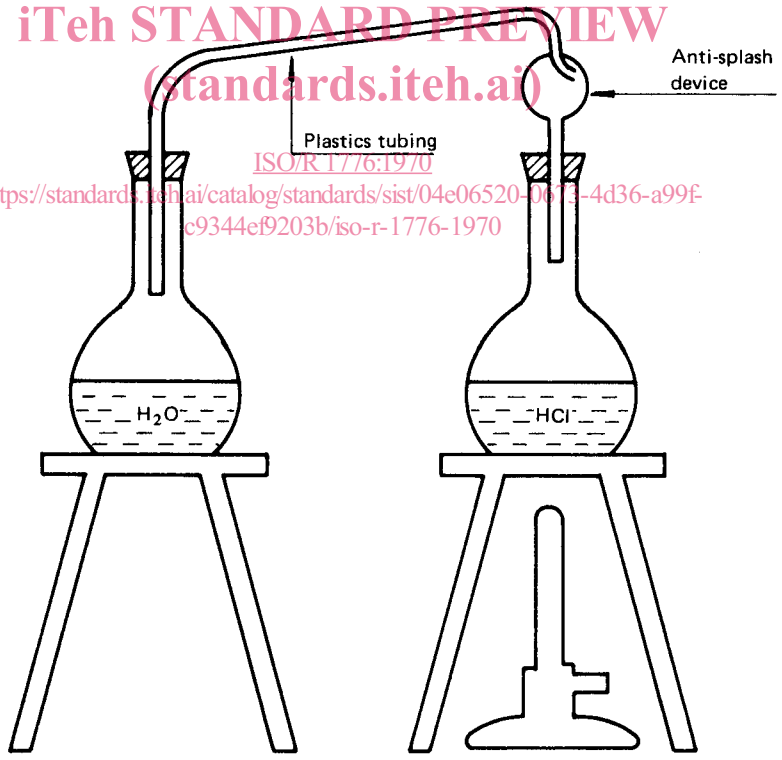

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FIG. 3 – **Preparation of 6N hydrochloric acid with very low Na_2O content**

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