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Mazdoor Kisan Shakti Sangathan
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“पुराने की छोट नये के तरफ”
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“Step Out From the Old to the New”

Indian Standard

GRADING GLASS FOR ALKALINITY

PART 2 HYDROLYTIC RESISTANCE OF GLASS CONTAINERS

(First Revision)

UDC 11:620.168.3:543.527.1
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Glassware Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1963, to classify different grades of glass by estimating the alkalinity of glass. To keep it at par with the improved technology and present international standard, the standard is being revised into two parts based on the destructive as well as non-destructive methods. Part 1 deals with the hydrolytic resistance of glass grains and Part 2 deals with hydrolytic resistance of the interior surfaces of glass containers. Part 1 has two sections, namely, Section 1 Hydrolytic resistance of glass at 98°C, Section 2 Hydrolytic resistance at 121°C.

This standard, Part 2 is based on ISO 4802-1 : 1988 'Hydrolytic resistance of glass containers—Part 1 : Determination by titration method and classification'. The resistance is measured by titration of a known aliquot portion of the extraction solutions produced with hydrochloric acid solution, in which case the resistance is inversely proportional to the volume of acid required.

The composition of the committee responsible for the formulation of this standard is given in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1070 : 1992</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>1117 : 1975</td>
<td>Specification for one-maric pipettes (first revision)</td>
</tr>
<tr>
<td>1382 : 1981</td>
<td>Glossary of terms relating to glass and glassware (first revision)</td>
</tr>
<tr>
<td>1997 : 1982</td>
<td>Specification for burettes (second revision)</td>
</tr>
<tr>
<td>6654 : 1992</td>
<td>Glass containers — Glossary of terms (second revision)</td>
</tr>
</tbody>
</table>

( CHD 10 )

Reprography Unit, BIS, New Delhi, India
1 SCOPE

This Indian Standard specifies methods for determining the hydrolytic resistance of the interior surfaces of glass containers when subjected to attack by water at 121°C ± 1°C for 60 min ± 1 min.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<table>
<thead>
<tr>
<th>IS No.</th>
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</tr>
</thead>
<tbody>
<tr>
<td>1070</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>1117</td>
<td>One-mark pipettes (first revision) (Reaffirmed July 1991)</td>
</tr>
<tr>
<td>1997</td>
<td>Burettes (second revision) (Reaffirmed Nov 1993)</td>
</tr>
<tr>
<td>1382</td>
<td>Glossary of terms relating to glass and glassware (first revision) (Reaffirmed June 1992)</td>
</tr>
<tr>
<td>6654</td>
<td>Glossary of terms relating to glass containers (second revision)</td>
</tr>
</tbody>
</table>

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1382 : 1981 and IS 6654:1992 shall apply.

4 FIELD OF APPLICATION

This part of IS 2303 applies to containers, such as bottles, vials, ampoules, flasks, beakers etc made from soda-lime-silica glass, whether surface-treated or not, or from borosilicate glass or neutral glass. This part of IS 2303 does not apply to double-ended ampoules or to the classification of closed ampoules.

5 PRINCIPLE

This method of test is a surface test normally applied to glass containers as delivered. It involves filling of the containers to be tested with specified water to a specified capacity and heating of the containers loosely capped under specified conditions followed by measurement of the degree of the hydrolytic attack by titration of the extraction solutions.

6 REAGENTS

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

6.1 Test Water

Consisting of Grade 2 Water, which complies with the requirements specified in IS 1070 : 1992 and which has been freed from dissolved gases, such as carbon dioxide, by boiling for at least 15 min in a boiling flask of fused silica or borosilicate glass. The boiling flask shall be pretreated once as specified in 9.2 before it is used for the first time.

When tested immediately before use, this test water shall be neutral to methyl red, that is, 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 are added to 50 ml of the test water.

NOTE — The water, to coloured, may also be used at the reference solution (see 9.3).

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

6.2 Hydrochloric Acid

Standard volumetric solution HCl = 0.01 M, 2 M.

6.3 Hydrofluoric Acid

Concentration (HF) = 22 mol/1 (that is = 400 g HF/1 solution).

6.4 Methyl Red Indicator Solution

Dissolve 25 ml of the sodium salt of methyl red (C\textsubscript{14}H\textsubscript{14}N\textsubscript{3}NaO\textsubscript{2}) in 100 ml of the test water.
7 APPARATUS

7.1 Autoclave or Steam Sterilizer
Capable of withstanding a pressure of at least $2.5 \times 10^5 \text{ N/m}^2$ and of carrying out the heating cycle specified in 9.2. It should preferably be equipped with a constant-pressure regulator or other means of maintaining the temperature at $121^\circ\text{C} \pm 1^\circ\text{C}$. 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 ventcock, and a rack for supporting the samples.

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

7.2 Burettes
Having a suitable capacity of 50 ml, 25 ml, 10 ml or 2 ml, complying with the requirements specified for Class A burettes in IS 1997:1982 and made of glass of hydrolytic resistance grain class HGA 1 as specified in IS 2303 (Part 1/Sec 2). The capacity of the burettes shall be chosen according to the expected consumption of hydrochloric acid.

7.3 Conical Flasks
Having a capacity of 100 ml and 250 ml. Before use, each new flask shall be pretreated by subjecting it to the autoclaving conditions described in 9.2.

7.4 Pipettes
Having a suitable capacity and complying with the requirements specified for Class A pipettes in IS 1117 : 1975.

7.5 Water Bath
Capable of being heated to approximately 80°C.

7.6 Beakers
Having a suitable capacity. Before use, each new beaker shall be pretreated by subjecting it to the autoclaving conditions described in 9.2.

7.7 Strike-Plates
For measuring the brimful capacity of small bottles and bottles made of rigid, inert, transparent material of any convenient shape but with a central hole approximately 5 mm in diameter. The strike-plate shall be large enough to fit snugly on and completely cover the sealing surface of the container the brimful capacity of which is to be measured.

8 SAMPLE PREPARATION

8.1 Sample Size
The number of containers to be tested depends on the capacity of the container, the volume of
8.2 Determination of the Filling Volume

8.2.1 Flat-Bottomed Containers Up to 30 ml Capacity (Except Ampoules)

Select six containers at random from the sample lot and remove any dirt or packaging debris by shaking the containers. Place each dry container on a flat, horizontal surface and allow to reach a temperature of 27°C ± 2°C. Cover each container with a strike-plate with the hole positioned approximately central to the mouth of the container. Fill each container with distilled water at 27°C ± 2°C from a burette, through the hole in the strike-plate, until the meniscus is just level with the bottom of the hole. Ensure that no air bubbles are trapped at the water/strike-plate interface. Then read the volume of water filled in from the burette to two decimal places. This volume is the brimful capacity of the container.

Calculate the mean value of the results from the six containers. Then calculate 90 percent of this mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.2 Flat-Bottomed Containers of 30 ml Capacity and Greater

Select six containers (having a capacity less than or equal to 100 ml) or three containers (having a capacity greater than 100 ml) at random from the sample lot and remove any dirt or packaging debris by shaking the containers. Allow the dry containers to reach a temperature of 27°C ± 2°C. Fix each container vertically in an appropriate device and determine the brimful capacity according to 8.2.1 or 8.2.2 respectively. Then calculate 90 percent of the mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.3 Round-Bottomed Containers (Except Ampoules)

Select six containers (having a capacity less than or equal to 100 ml) or three containers (having a capacity greater than 100 ml) at random from the sample lot and remove any dirt or packaging debris by shaking the containers. Allow the dry containers to reach a temperature of 27°C ± 2°C. Fix each container vertically in an appropriate device and determine the brimful capacity according to 8.2.1 or 8.2.2 respectively.

Then calculate 90 percent of the mean brimful capacity to one decimal place. This volume is the filling volume for the particular sample lot.

8.2.4 Lipped Containers

Wrap adhesive plastics tape around the rim of the containers such that the tape around the lip is level with the rim. Weigh the container with its strike-plate in place, then fill and reweigh as described in 8.2.2, without taking the strike-plate off.

8.2.5 Ampoules

Place at least six dry ampoules at 27°C ± 2°C on a flat, horizontal surface and fill them with the distilled water (6.6), at the same temperature, from a burette (7.2), until the water reaches point A, where the body of the ampoule declines to the shoulder (see Fig. 2). Read the capacities to two decimal places and calculate the mean value.

### Table 1 Number of Containers for the Determination of the Hydrolytic Resistance by Titration

*(Clause 8.1)*

<table>
<thead>
<tr>
<th>Capacity [Volume Corresponding to Filling Volume (See 8.2)] ml</th>
<th>Minimum Number of Containers for One Titration (2)</th>
<th>Volume of Extraction Sol. for One Titration in ml (3)</th>
<th>Number of Titrations (4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to and including 3</td>
<td>10</td>
<td>25.0</td>
<td>1</td>
</tr>
<tr>
<td>From 3 up to and including 30</td>
<td>5</td>
<td>50.0</td>
<td>2</td>
</tr>
<tr>
<td>From 30 up to and including 100</td>
<td>3</td>
<td>100.0</td>
<td>2</td>
</tr>
<tr>
<td>From 100 upwards including 100</td>
<td>1</td>
<td>100.0</td>
<td>3</td>
</tr>
</tbody>
</table>
This volume, expressed to one decimal place, is the filling volume and shall be filled in ampoules of the same lot.

9 PROCEDURE

9.1 Cleaning of Samples

This cleaning process shall be completed from the first rinsing in not less than 20 min and not more than 25 min.

Remove from all open samples any packaging debris or dirt which has collected during storage and transport. Rinse each sample thoroughly at least twice with the distilled water (6.6) at ambient temperature, then allow to stand, filled with the distilled water. Immediately before testing, empty the samples, rinse once with the distilled water and then once with the test water (6.1). Allow to drain completely.

Closed ampoules shall be warmed in a water bath or air-oven at about 50°C for approximately 2 min before opening. They shall not be rinsed before testing.

9.2 Filling and Heating

Fill each container, selected for the sample in accordance with 8.1 and cleaned in accordance with 9.1, to the filling volume with the test water by means of suitable volumetric measuring devices.

Each container including ampoules shall be loosely capped with an inert material, for example with inverted beakers (7.6) of such a size that the bottoms of the beakers fit snugly down on the rims of the sample, ampoules, for example with clean aluminium foil.

NOTE — Ensure that the foil does not release ions to be measured into the test water.

Place the samples in groups in petri dishes, on the rack in the autoclave, containing distilled water at ambient temperature, and ensure that they are held above the level of the water in the vessel. Close the autoclave lid or door 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 a rate of 1°C/min to 121°C. Maintain the temperature at 121°C ± 1°C for 60 min ± 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.

NOTE — Experience has shown that the rate of heating to 121°C, the holding temperature of 121°C ± 1°C and the rate of cooling to 100°C are critical. Variations from the specified conditions can produce variable results even to the extent of invalidating them.

Remove the hot samples from the autoclave, place in the water bath, heated to about 80°C, and run cold water into and out of the bath at a rate which will cool the samples to ambient temperature as quickly as possible; account shall be taken of the size and wall thickness of the samples and the type of glass from which the samples are made in order to avoid losses by thermal shock. The cooling time shall not exceed 30 min. Start with the determinations after cooling.

Take care that the cooling tap water does not contact the loose foil caps. This is very dangerous, especially in vials.

9.3 Analysis of Extract Solutions

Combine the extraction solutions of the containers (see col 2 of Table 1). When emptying small stemmed ampoules there is a danger of neutralization of the solution by absorption of carbon dioxide (CO₂), from the atmosphere. To obviate this, invert the ampoules and heat the bases gently. In the case of the combined extraction solutions from containers having a capacity less than or equal to 3 ml, pipette a volume of 25.0 ml (see col 3 of Table 1) into a conical flask having a capacity of 100 ml In the case of the combined extraction solutions from containers having a capacity from 3 to 30 ml or from 30 to 100 ml (see col 1 of Table 1), pipette volumes of 50.0 and 100.0 ml, respectively (see col 3 of Table 1), into separate conical flasks having a capacity of 250 ml.
In the case of samples with a capacity above 100 ml (see col 1 of Table 1), pipette a volume of 100.0 ml from each container into separate conical flasks having a capacity of 250 ml.

Prepare reference solutions by pipetting volumes, equivalent to those taken from the extraction solutions, of the test water into conical flasks having a capacity commensurate with the size of the containers being tested. Add two drops of methyl red indicator solution to each 25 ml of test water.

Add two drops of methyl red indicator solution to each flask for each 25 ml of extraction solution and titrate with hydrochloric acid until the colour matches exactly that of coloured reference solutions.

Titration values of less than 1.0 ml shall be expressed to two decimal places, titration values greater than or equal to 1.0 ml to one decimal place.

9.4 Testing to Determine Whether the Containers Have Been Surface-Treated

9.4.1 Vials and Bottles

The hydrolytic resistance of the interior surface of vials and bottles made from soda-lime-silica glass can be considerably increased by treating those surfaces during the course of production.

If it is necessary to determine whether or not a container has been surface-treated, the samples previously tested shall be used.

Fill the samples with the mixture of 1 volume of hydrofluoric acid and 9 volumes of hydrochloric acid to the brimful point. Allow the filled samples to stand at ambient temperature for 10 min, then empty the solution very carefully. Rinse the samples three times with the distilled water then at least twice with the test water. Then test the samples as specified in 9.2 and 9.3.

If the results are considerably higher than those obtained from the original surfaces (about five to ten times), the samples shall be considered to have been surface-treated.

9.4.2 Ampoules

Ampoules made from glass tubing are not normally subjected to an internal surface treatment, because their high chemical resistance is dependent upon the chemical composition of the glass as a material (see 4).

If it is necessary to determine whether or not the ampoules have been surface-treated, use the samples previously tested.

Apply the surface etching treatment as specified in 9.4.1, and test the samples as specified in 9.2 and 9.3.1.

The new values are slightly lower than the previous ones, if the ampoules are not treated.

10 EXPRESSION OF RESULTS

10.1 Calculation

Calculate the mean value of the titration results, in millilitres of hydrochloric acid solution per 100 ml of the extraction solution. The results may also be calculated and expressed as micrograms of sodium oxide (Na$_2$O) per 100 ml of the extraction solution:

$$1 \text{ ml of hydrochloric acid solution}$$

$$[c (\text{HCl}) = 0.01 \text{ mol/l}] = 310 \text{ g of sodium oxide}.$$  

10.2 Grading

The containers shall be graded as shown in Table 2, according to the consumption of hydrochloric acid solution when tested as specified in 9.3 and calculated according to 10.1.

10.3 Distinction Between Containers of Hydrolytic Resistance Container Grade HC 1 and Hydrolytic Resistance Container Grade HC 2

After etching and re-testing in accordance with 9.4, containers of hydrolytic resistance container Grade HC 1 will satisfy the requirements for hydrolytic resistance container Grades HC 1 and HC 2 in Table 2.

Table 2 Maximum Values In the Hydrolytic Resistance Container Surface Test (Titration Method)

<table>
<thead>
<tr>
<th>(Clauses 10.2 and 10.3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capacity of Container [Volume Corresponding to the Filling Volume (see 8.2)]</td>
</tr>
<tr>
<td>(Grades and HC2)</td>
</tr>
<tr>
<td>ml</td>
</tr>
<tr>
<td>Up to and including 1</td>
</tr>
<tr>
<td>From 1 up to and including 2</td>
</tr>
<tr>
<td>From 2 up to and including 5</td>
</tr>
<tr>
<td>From 5 up to and including 10</td>
</tr>
<tr>
<td>From 10 up to and including 20</td>
</tr>
<tr>
<td>From 20 up to and including 50</td>
</tr>
<tr>
<td>From 50 up to and including 100</td>
</tr>
<tr>
<td>From 100 up to and including 200</td>
</tr>
<tr>
<td>From 200 up to and including 500</td>
</tr>
<tr>
<td>From 500 upwards</td>
</tr>
</tbody>
</table>
After etching and re-testing in accordance with 9.4, containers of hydrolytic resistance container Grade HC 2 will produce values which are significantly greater than those given in col 2 of Table 2 and which are much closer to those values for hydrolytic resistance container Grade HC 3 in Table 2.

10.4 Designation

The hydrolytic resistance of the interior surface of glass containers measured in accordance with this part of IS 2303 shall be designated as follows:

*Example:*

The designation for a 9 ml capacity container with a consumption of 1.6 ml of hydrochloric acid solution \([c(\text{HCl}) - 0.01 \text{ mol/1}]\) per 100 ml extraction solution shall be:

Glass, hydrolytic resistance container glass IS 2303-HC B

11 TEST REPORT

The test report shall include the following information:

a) A reference to this part of IS 2303;

b) An identification of the samples;

c) Mean brimful capacity of samples (except ampoules);

d) The filling volume of the samples;

e) The number of samples used for one titration;

f) The mean value of the titrations;

g) Hydrolytic resistance container Grade HC (designation of the container tested);

h) For hydrolytic resistance container Grade HC 2, a statement whether the test has been repeated after etching of the surface (see 9.4) and the results obtained;

j) A statement whether closed ampoules were tested; and

k) Any unusual features noted during the determination.
ANNEX A

COMMITTEE COMPOSITION

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DR. R. N. DWIVEDI

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Member-Secretary
SMT MEENAL PASSI
Assistant Director (Chem), BIS

(Continued on page 8)
### Methods of Test for Glass and Glassware Subcommittee, CHD 10:01

**Members**

<table>
<thead>
<tr>
<th>Name</th>
<th>Representing</th>
</tr>
</thead>
<tbody>
<tr>
<td>DR P. K. GANGOPADHYAY</td>
<td>College of Ceramic Technology Calcutta</td>
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<td>Department of Applied Chemistry, Calcutta University, Calcutta</td>
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<tr>
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<td>Haryana Sheet Glass Ltd, Rail</td>
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<tr>
<td>PROF V. C. JOSHI (Alternate)</td>
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</tr>
<tr>
<td></td>
<td>National Physical Laboratory, New Delhi</td>
</tr>
<tr>
<td></td>
<td>Banaras Hindu University, Varanasi</td>
</tr>
</tbody>
</table>

(Continued from page 7)
Bureau of Indian Standards

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This Indian Standard has been developed from Doc: No. CHD 010 (0517).

<table>
<thead>
<tr>
<th>Amend No.</th>
<th>Date of Issue</th>
<th>Text Affected</th>
</tr>
</thead>
</table>

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