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Guidelines on types of glass of normal bulk-production composition and their test methods

*Lignes directrices sur les types de verre de composition normale de
production en vrac et leurs méthodes d'essai*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 12775 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, Subcommittee SC 5, *Quality of glassware*.

Annex A of this International Standard is for information only.

Introduction

Various committees have been working for many years at national and international levels to produce agreed test methods for measuring the chemical and physical properties of glass as a material and glassware as finished articles. Because the international standardization part of the work has mostly been carried out under the aegis of the International Organization for Standardization, through its Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, the aim of the work has always been slanted towards this type of ware. Even so, the test methods and classifications proposed to date are equally applicable to other kinds of glass and to glassware which is not necessarily used only for laboratory purposes.

It has been suggested that a collection of the information produced by the various committees would provide useful guidance to users and to manufacturers of glassware. This International Standard is, therefore, intended to give such guidance but it is strongly emphasized that a classification according to one test procedure is not necessarily related to classification by another test procedure.

Although not concerning normal bulk-production glass, Technical Committee ISO/TC 172, *Optics and optical instruments*, has established some International Standards for test procedures for optical glass, for example for acid resistance (ISO 8424) and for testing the resistance to attack by aqueous alkaline phosphate-containing detergent solutions (ISO 9689).

To make these guidelines complete, some test methods are also cited which do not have a classification (which is needed for most physical test methods) but that are sometimes of great interest for the glass user or glass manufacturer.

Guidelines on types of glass of normal bulk-production composition and their test methods

1 Scope

This International Standard establishes a survey of glass types and of methods for testing their chemical and physical properties to give, for example, consumers and producers of glass of normal bulk-production composition the possibility to compare the different types of glass and test methods and to decide which are of interest for a special demand or use. For this purpose, these comprehensive guidelines give a classification of the different glass types of normal bulk-production composition according to the chemical composition and indicate the different test methods and, where they exist, the classifications according to chemical resistance.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standard are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 695:1991, *Glass — Resistance to attack by a boiling aqueous solution of mixed alkali — Method of test and classification.*

ISO 718:1990, *Laboratory glassware — Thermal shock and thermal shock endurance — Test methods.*

ISO 719:1985, *Glass — Hydrolytic resistance of glass grains at 98 °C — Method of test and classification.*

ISO 720:1985, *Glass — Hydrolytic resistance of glass grains at 121 °C — Method of test and classification.*

ISO 1776:1985, *Glass — Resistance to attack by hydrochloric acid at 100 °C — Flame emission or flame atomic absorption spectrometric method.*

ISO 3585:1991, *Borosilicate glass 3.3 — Properties.*

ISO 4802-1:1988, *Glassware — Hydrolytic resistance of the interior surfaces of glass containers — Part 1: Determination by titration method and classification.*

ISO 4802-2:1988, *Glassware — Hydrolytic resistance of the interior surfaces of glass containers — Part 2: Determination by flame spectrometry and classification.*

ISO 4803:1978, *Laboratory glassware — Borosilicate glass tubing.*

ISO 7459:1984, *Glass containers — Thermal shock resistance and thermal shock endurance — Test methods.*

ISO 7991:1987, *Glass — Determination of coefficient of mean linear thermal expansion.*

3 Main types of glass of normal bulk-production composition

The main types of glass of normal bulk-production composition are classified in table 1.

NOTE — In many cases it is customary to divide glass into different "types" according to the use or shape (laboratory glass, bottle glass, optical glass, flat glass, hollow glass) and in a general way this division is a kind of classification. Another possibility is a classification according to the chemical composition. This leads to the types listed in table 1, which of course cannot be absolutely pure concerning the composition ranges.

Table 1 — Classification of main types of glass of normal bulk-production composition according to their chemical composition

Descriptors	Alkali/alkaline earth/silicate glass ¹⁾	Borosilicate glass		Alkaline earth/alumino-silicate glass	Alkaline lead silicate glass
		Alkaline-earth free (Borosilicate glass 3.3) ²⁾	Containing alkaline earths (Known as "neutral glass" ³⁾)		
Key oxides % (m/m)	Na ₂ O, CaO > 10	B ₂ O ₃ > 8	B ₂ O ₃ > 8	Al ₂ O ₃ > 10	PbO > 10
Typical field of composition					
SiO ₂ % (m/m)	70 to 75	≈ 81	≈ 75	52 to 60	54 to 58
Alkali oxides % (m/m) (Na ₂ O + K ₂ O)	12 to 16	= 4	4 to 8	—	up to 15
Alkaline-earth oxides % (m/m) (MgO + CaO + BaO + SrO)	10 to 15	—	up to 5	up to 15	up to 4
Al ₂ O ₃ % (m/m)	0,5 to 2,5	2 to 3	2 to 7	17 to 25	up to 4
B ₂ O ₃ % (m/m)	—	12 to 13	8 to 12	—	—
PbO % (m/m)	—	—	—	—	up to 35
Coefficient of mean linear thermal expansion, 10 ⁻⁶ K ⁻¹ α (20 °C; 300 °C)	8 to 10	3.3	4 to 5	≈ 4	7 to 9
Hydrolytic resistance	Medium, low	Very high	Very high	High, very high	Medium
Acid resistance	Very high	Very high	Very high	Low	Low, medium
Alkali resistance	Medium	Medium	Medium	Low	Medium
Main fields of application	Container bottle (glass); float glass; drawn sheet glass	Laboratories for chemical, pharmaceutical and food industries ⁴⁾ Technical purposes with demands for high chemical and thermoshock resistance	Pharmaceutical containers Technical purposes with demands for high chemical resistance	Purposes with demands for high thermal resistance: high-temperature thermometers, resistors capable of high thermal and electrical loading, combustion tubes	Noble table glasses; lamp stems; cathode ray tubes; radiation-shielding glass

NOTE — The given compositions are mean levels of typical glass types. They are only for information and shall not be understood as "limit values". It is known that actual glasses differ to a certain degree, which does not affect the chemophysical properties.

1) This is the oldest glass type, which makes the largest percentage of the worldwide glass production; also belonging to this type are glasses with higher BaO and SrO contents such as alkaline-earth oxides, with reduced alkali content (e.g. for X-ray protection, as used in cathode ray tube components), and also certain crystal glasses (drinking glasses).

2) In accordance with ISO 3585.

3) See ISO 4802-1 and ISO 4802-2.

4) See ISO 4803.

4 Chemical properties of glass and glass articles

4.1 General

The chemical durability, or chemical resistance, of glass as a material or of glass articles is the ability of the exposed surfaces to withstand attack by acid, neutral or alkaline solutions. In general for silicate glasses this ability decreases with increasing alkalinity of the attacking medium and test methods are available for measuring these resistances.

4.2 Glass as a material and glass as-delivered

When properties of glass articles are considered, it must be emphasized that the surface of a finished glass article can have different properties than the glass itself, called "glass as a material". This glass as a material (i.e. the bulk glass) has, of course, the same properties as the glass of glass articles when their surface layers are removed, for instance by grinding, or polishing, or acid etching with hydrofluoric acid. But the properties of glass surfaces can be modified: for instance, fire-polishing by pressing or blowing creates a slightly different surface layer because of the evaporation of glass constituents at the glass-forming temperature. Also the glass surface can be treated when the articles are finished: ion-exchange (salt melts, for instance) can result in a strengthening effect; or acid vapour (such as SO₂) can reduce the alkali concentration in the surface of soda-lime glasses, and thus their surfaces show a better hydrolytic resistance; there are many other examples.

So when glass articles are tested, care shall be taken to define whether the surface is tested "as-delivered" (i.e. with the original surface layer) or "as a material", which is to be achieved by acid etching, grinding/polishing or breaking. The qualitative descriptions of the types of glass in tables 2 and 3 concern glass as a material only; tables 4 to 6 concern non-treated glasses/articles except for class HC 2 in ISO 4802 (in tables 4 and 5).

4.3 Test methods

4.3.1 Hydrolytic resistance (see ISO 719, ISO 720, ISO 4802-1 and ISO 4802-2)

Grain tests, such as described in ISO 719 and ISO 720, measure the resistance of glass as a material to attack by neutral solutions (i.e. water) and this is called the "hydrolytic grain resistance". These tests apply to glass as a material because the sample is broken and new bulk surfaces are produced.

The hydrolytic resistance of glass containers is measured by a "whole article" test in which the container is filled with water and the resistance of the "as-produced" surface is measured.

NOTE — The European Pharmacopœia uses the latter type of test to measure the performance of containers and the former to distinguish between the resistances imparted to glass by compositional and surface treatment processes.

The values given in table 4 show the limiting titration values required to neutralize 100 ml of extraction solution from various capacities of containers, as given in ISO 4802-1.

NOTE — The values for container classes HC 1, 2 and 3 are identical to those published by the European Pharmacopœia, where they are called "glass container types". Since there are more container types, ISO 4802 includes also the container class HC B (a glass used especially for "buables", i.e. drinking ampoules and vials) and HC D (for "dry substances").

Furthermore, a classification according to the direct determination of the released alkali, different from the determination of the sum parameter of hydroxide ions by titration (as done by flame spectrometric methods on the extract solution) is given in ISO 4802-2 (see table 5). A direct conversion from one system to the other (titration to flame-spectrometric values) is not clear nor easily achievable, but a conversion factor between 2,5 and 3,0 was proved by interlaboratory tests to be true, so long as the values are near the limits between the container classes.

4.3.2 Acid resistance (see ISO 1776)

The resistance of glass articles to attack by acid solutions (usually 6 mol/l hydrochloric acid) can be measured by the procedure described in ISO 1776. In this procedure the glass sample is prepared in the form of a piece, of 30 cm² to 40 cm² surface area. When the area of the cut edges does not exceed 10 % of the total surface area, the pieces can be regarded as tested as-delivered. When the area of the edges is greater than 10 %, the rest of the original surfaces shall be removed for the acid resistance test in accordance with ISO 1776 by acid etching and

then the test results refer to glass as a material. The etching procedure shall be applied when thicker samples of articles, such as from glass plant, pipeline or fittings, are to be tested in accordance with ISO 1776.

4.3.3 Alkali resistance (see ISO 695)

The resistance of glass articles to alkaline attack is measured in accordance with ISO 695. Pieces with surfaces of 10 cm² to 15 cm² each (20 cm² to 30 cm² in total) are submitted to attack by a boiling alkaline mixture, and the loss in mass is determined. Because of the strength of the attack, distinction concerning the new cut edges is not necessary.

4.3.4 Other chemical test methods

For optical glass, the test methods are specified in ISO 8424 (acid resistance, SR), in ISO 9689 (phosphate resistance, PR) and ISO 10629 (alkali resistance, AR). Since this type of glass is not of general interest, International Standards for the test methods are given for information only.

4.4 Classifications of glass and glass articles according to their chemical properties

4.4.1 Principle of classifications

In the classification tables which follow, the class number has an additional abbreviation to designate the type of ISO test procedure. For instance, the grain tests can be distinguished by the class designations: ISO 719 by HGB (hydrolytic resistance of glass grains according to the boiling water test method, at 98 °C) and ISO 720 by HGA (hydrolytic resistance of glass grains according to the autoclave test method, at 121 °C). For the container test, the designation of ISO 4802 is HC (hydrolytic resistance container class). The alkali resistance class is ISO 695, class A.

It should be emphasized that because the test conditions are different, class HGB 2 of ISO 719 is **not** the same as class HGA 2 of ISO 720. Furthermore, neither of them is related to class HC 2 of ISO 4802, or class A2 of ISO 695, which is class 2 in the alkali resistance table.

4.4.2 Classification table and/or limit values

4.4.2.1 For the hydrolytic resistance of glass grains at 98 °C according to ISO 719, and at 121 °C according to ISO 720, see table 2 and table 3, respectively.

Table 2 — Hydrolytic resistance of glass grains at 98 °C in accordance with ISO 719

Class	Consumption of hydrochloric acid solution [$c(\text{HCl}) = 0,01 \text{ mol/l}$] per gram of glass grains	Equivalent of alkali expressed as mass of sodium oxide (Na_2O) per gram of glass grains	Relative resistance of glass ¹⁾
	ml/g	$\mu\text{g/g}$	
HGB 1	Up to and including 0,10	up to and including 31	very high
HGB 2	From 0,10 up to and including 0,20	from 31 up to and including 62	high
HGB 3	from 0,20 up to and including 0,85	from 62 up to and including 264	medium
HGB 4	from 0,85 up to and including 2,0	from 264 up to and including 620	low
HGB 5	from 2,0 up to and including 3,5	from 620 up to and including 1085	very low

1) This is not included in ISO 719.

Table 3 — Hydrolytic resistance of glass grains at 121 °C in accordance with ISO 720

Class	Consumption of hydrochloric acid solution [$\alpha(\text{HCl}) = 0,01 \text{ mol/l}$] per gram of glass grains	Equivalent of alkali expressed as mass of sodium oxide (Na_2O) per gram of glass grains	Relative resistance of glass ¹⁾
	ml/g	$\mu\text{g/g}$	
HGA 1	Up to and including 0,10	up to and including 62	very high
HGA 2	From 0,10 up to and including 0,85	from 62 up to and including 527	medium
HGA 3	From 0,85 up to and including 1,50	from 527 up to and including 930	low

1) This is not included in ISO 720.

4.4.2.2 For the hydrolytic resistance of the interior surfaces of glass containers in accordance with ISO 4802-1 (titration method) and ISO 4802-2 (determination by flame spectrometric methods), see table 4 and table 5, respectively.

Table 4 — Hydrolytic resistance of the interior surfaces of glass containers determined by the titration method given in ISO 4802-1

Capacity of container (volume corresponding to the filling volume) ml	Maximum values for the consumption of hydrochloric acid solution [$\alpha(\text{HCl}) = 0,01 \text{ mol/l}$] per 100 ml of the extraction solution ml/100 ml			
	Classes HC 1 and HC 2	Class HC 3	Class HC B	Class HC D
Up to and including 1	2,0	20,0	4,0	32,0
From 1 up to and including 2	1,8	17,6	3,6	28,0
From 2 up to and including 5	1,3	13,2	2,6	21,0
From 5 up to and including 10	1,0	10,2	2,0	17,0
From 10 up to and including 20	0,80	8,1	1,6	13,5
From 20 up to and including 50	0,60	6,1	1,2	9,8
From 50 up to and including 100	0,50	4,8	1,0	7,8
From 100 up to and including 200	0,40	3,8	0,80	6,2
From 200 up to and including 500	0,30	2,9	0,60	4,6
From 500 upwards	0,20	2,2	0,40	3,6
Relative resistance of container ¹⁾	very high	medium	high	low

1) This is not included in ISO 4802-1.

Table 5 — Hydrolytic resistance of the interior surfaces of glass containers determined by flame spectrometric methods given in ISO 4802-2

Capacity of container [volume corresponding to the filling volume] ml	Maximum values for the consumption of oxides expressed as micrograms of sodium oxide (Na ₂ O) per millilitre of extraction solution ml/100 ml			
	Classes HC 1 and HC 2	Class HC 3	Class HC B	Class HC D
Up to and including 1	5,00	60	12	96
From 1 up to and including 2	4,50	53	11	84
From 2 up to and including 5	3,20	40	7,8	63
From 5 up to and including 10	2,50	30	6,0	51
From 10 up to and including 20	2,00	24	4,8	40
From 20 up to and including 50	1,50	18	3,6	30
From 50 up to and including 100	1,20	14	3,0	23
From 100 up to and including 200	1,00	11	2,4	18
From 200 up to and including 500	0,75	8,7	1,8	14
From 500 upwards	0,50	6,6	1,2	10
Relative resistance of container ¹⁾	very high	medium	high	low

1) This is not included in ISO 4802-2.

Table 6 — Resistance to attack by a boiling aqueous solution of mixed alkali determined in accordance with ISO 695

Class	Loss in mass per total surface area after 3 h mg/dm ²	Relative resistance of glass ¹⁾
A1	Up to and including 75	high
A2	Above 75 to 175	medium
A3	Above 175	low

1) This is not included in ISO 695.

4.4.2.3 Concerning the acid resistance of glasses and glass articles made of borosilicate glass 3.3 in accordance with ISO 3585, the release of the sum of alkali oxides ($\text{Na}_2\text{O} + \text{K}_2\text{O}$) determined according to the flame emission or flame atomic absorption spectrometric method described in ISO 1776 shall not exceed 100 μg of alkali oxide per square decimetre (calculated as Na_2O).

Soda-lime silicate glasses release significantly more than 100 $\mu\text{g}/\text{dm}^2$.

4.4.2.4 For the alkali resistance in accordance with ISO 695 see table 6.

4.4.2.5 For the classification of the chemical properties of optical glass, information is given in ISO 8424, ISO 9689 and ISO 10629.

5 Physical properties

5.1 Thermal properties

One of the important requirements for the types of glass used in the production of laboratory ware is that they should be compatible. Compatibility is defined as the ability to fuse together different glasses whose properties, especially thermal expansion and viscosity, are sufficiently similar to permit a good seal.

Thermal expansion or, more specifically, the coefficient of mean linear thermal expansion, is defined as the ratio of the change in length of a specimen within a temperature interval to that temperature interval, related to the initial specimen length, and this property can be measured by the test method described in ISO 7991.

Thermal expansion is related to thermal shock resistance in that a low expansion normally means a high resistance to thermal shock and vice versa. But it must be emphasized that thermal shock resistance depends also strongly on the geometrical properties of the test samples, such as surface conditions (fire-polished, damaged), wall thickness (and its homogeneity in mass distribution), transition radii (e.g. from wall to bottom) and others. Thermal shock resistance is defined as the ability of glass articles to withstand thermal shock resulting from a sudden change in temperature. The thermal shock resistance of an article (i.e. the temperature difference the article will withstand) can be measured by the procedures described in ISO 718 for laboratory glassware, or in ISO 7459 for soda-lime silicate glass containers. In these procedures, the thermal shock resistance of an article is determined by heating a number of articles of identical size, shape and thickness to a uniform temperature and plunging them suddenly into cold water.

When the upper temperature is raised stepwise and the observations of the proportionate breaking are plotted against the upper temperature, the difference between the upper and lower temperature is frequently termed the "thermal shock resistance" and the value of this measure giving a 50 % probability of fracture is termed "thermal shock endurance".

The thermal shock resistance and the thermal shock endurance of glass as a material, as opposed to a finished article, can also be measured by the method given in ISO 718. In such cases, test pieces of the glass or glasses shall be prepared to be of the same sizes and dimensions and to be annealed to the same degree.

NOTE — Because of the reasons given, it is not realistic to establish classification tables.

5.2 Other physical properties

For other physical properties, test methods are described in the following International Standards:

- Knoop hardness, ISO 9385;
- birefringence, ISO 11455;
- stress optical coefficient, ISO 10345-1 and ISO 10345-2;
- stresses in glass-to-glass sealings, ISO 4790;
- viscosity and viscometric fixed points, ISO 7884-1 to ISO 7884-8.

Annex A (informative)

Bibliography

ISO 4790:1992, *Glass-to-glass sealings — Determination of stresses.*

ISO 7884-1:1987, *Viscosity and viscometric fixed points — Part 1: Principles for determining viscosity and viscometric fixed points.*

ISO 7884-2:1987, *Viscosity and viscometric fixed points — Part 2: Determination of viscosity by rotation viscometers.*

ISO 7784-3:1987, *Viscosity and viscometric fixed points — Part 3: Determination of viscosity by fibre elongation viscometer.*

ISO 7884-4:1987, *Viscosity and viscometric fixed points — Part 4: Determination of viscosity by beam bending.*

ISO 7884-5:1987, *Viscosity and viscometric fixed points — Part 5: Determination of working point by sinking bar viscometer.*

ISO 7884-6:1987, *Viscosity and viscometric fixed points — Part 6: Determination of softening point.*

ISO 7884-7:1987, *Viscosity and viscometric fixed points — Part 7: Determination of annealing point and strain point by beam bending.*

ISO 7884-8:1987, *Viscosity and viscometric fixed points — Part 8: Determination of (dilatometric) transformation temperature.*

ISO 8424:1996, *Raw optical glass — Resistance to attack by aqueous acidic solutions at 25 °C — Test method and classification.*

ISO 9385:1990, *Glass and glass ceramics — Knoop hardness test.*

ISO 9689:1990, *Raw optical glass — Resistance to attack by aqueous alkaline phosphate-containing detergent solutions at 50 °C — Testing and classification.*

ISO 10345-1:1992, *Glass — Determination of stress-optical coefficient — Part 1: Tensile test.*

ISO 10345-2:1992, *Glass — Determination of stress-optical coefficient — Part 2: Bending test.*

ISO 10629:1996, *Raw optical glass — Resistance to attack by aqueous alkaline solutions at 50 °C — Test method and classification.*

ISO 11455:1995, *Glass — Determination of birefringence.*

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