INTERNATIONAL STANDARD

ISO 28706-2

First edition
2008-12-01

Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 2:
Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours

Émaux vitrifiés — Détermination de la résistance à la corrosion chimique —

Partie 2: Détermination de la résistance à la corrosion chimique par des acides bouillants ou des liquides neutres bouillants, et/ou leurs vapeurs

Reference number
ISO 28706-2:2008(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 28706-2 was prepared by the European Committee for Standardization (CEN) (as EN 14483-2) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in parallel with its approval by the ISO member bodies.

It cancels and replaces ISO 2733:1983, ISO 2742:1998, ISO 2743:1986 and ISO 2744:1998, which have been technically revised.

ISO 28706 consists of the following parts, under the general title *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion*:

— *Part 1: Determination of resistance to chemical corrosion by acids at room temperature*

— *Part 2: Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours*

— *Part 3: Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel*

— *Part 4: Determination of resistance to chemical corrosion by alkaline liquids using a cylindrical vessel*

— *Part 5: Determination of resistance to chemical corrosion in closed systems*

Introduction

Corrosion of vitreous and porcelain enamels by aqueous solutions is a dissolution process. The main component of the enamel, SiO_2, forms a three-dimensional silica network. After hydrolysis, it decomposes and forms silicic acid or silicates. These are released into the attacking medium. Other components, mainly metal oxides, are hydrolysed as well and form the corresponding hydrated metal ions or hydroxides. All corrosion products are more or less soluble in the attacking medium. The whole process results in a loss in mass per unit area.

For some aqueous solutions, the attack on the enamel proceeds linearly during the corrosion time; for other aqueous solutions, the attack on the enamel proceeds in a logarithmic manner during the corrosion time. Only for the first series of solutions can a scientifically exact rate of loss in mass per unit area ($g/m^2 \cdot h$) be calculated as well as a corrosion rate (mm/year).

The most important parameters influencing aqueous corrosion of the enamel are the enamel quality, the temperature and the pH-value. Inhibition effects resulting from the limited solubility of silica can also contribute. The following list describes different types of enamel attack for different corrosion conditions:

a) In aqueous alkali solutions like 0,1 mol/l NaOH (see Clause 9 of ISO 28706-4:2008), the silica network of the enamel is considerably attacked at 80 °C. Silicates and most of the other hydrolysed components are soluble in the alkali. Attack proceeds linearly during regular test times. Therefore, test results are expressed in terms of a rate of loss in mass per unit area (mass loss per unit area and time) and a corrosion rate (millimetres per year).

b) At room temperature, in weak aqueous acids like citric acid (see Clause 9 of ISO 28706-1:2008) or also in stronger acids like sulfuric acid (see Clause 10 of ISO 28706-1:2008), there is only minor attack on the silica network of the enamel. Other constituents are leached to some extent from the surface. Highly resistant enamels will show no visual change after exposure. On less resistant enamels, some staining or surface roughening will occur.

c) In boiling aqueous acids (see ISO 28706-2:2008), the silica network of the enamel is being attacked, and silica as well as the other enamel components are released into solution. However, the solubility of silica in acids is low. Soon, the attacking solutions will become saturated with dissolved silica and will then only leach the surface. The acid attack is inhibited and the rate of corrosion drops markedly.

   NOTE The glass test equipment also releases silica by acid attack and contributes to the inhibition of the corrosion.

   Inhibition is effectively prevented in vapour phase tests. The condensate formed on the test specimen is free of any dissolved enamel constituents.

   Examples of enamel corrosion proceeding in a logarithmic manner [see 1)] and linearly [see 2)] are:

   1) Boiling citric acid (see Clause 10 of ISO 28706-2:2008) and boiling 30 % sulfuric acid (see Clause 11 of ISO 28706-2:2008)

   Since only minute amounts of these acids are found in their vapours, the test is restricted to the liquid phase. The attack is influenced by inhibition effects, and corrosion depends on the time of exposure. Therefore, test results are expressed in terms of loss in mass per unit area; no rate of loss in mass per unit area is calculated.

   2) Boiling 20 % hydrochloric acid (see Clause 12 of ISO 28706-2:2008)

   Since this is an azeotropic boiling acid, its concentration in the liquid and the vapour phase are identical, and liquid phase testing need not be performed. Vigorous boiling supplies an uninhibited condensate, and the attack proceeds linearly with time of exposure. Therefore, test results are only expressed in terms of rate of loss in mass per unit area (mass loss per unit area and time) and the corrosion rate (millimetres per year).

d) At high temperatures, with tests in the liquid phase under autoclave conditions (see ISO 28706-5), aqueous acid attack is severe. To avoid inhibition, the test time is restricted to 24 h and the ratio of attacking acid to attacked enamel surface is chosen so that it is comparatively high (similar to that in a chemical reaction vessel). In addition, only low-silica water is used for the preparation of test solutions. Under these conditions, attack will proceed linearly with time of exposure. Therefore, test results with 20 % hydrochloric acid (see Clause 8 of ISO 28706-5:2008), artificial test solutions (see Clause 10 of ISO 28706-5:2008) or process fluids (see Clause 11 of ISO 28706-5:2008) are also expressed in terms of a rate of loss in mass per unit area (loss in mass per unit area and time).

e) In boiling water (see Clause 13 of ISO 28706-2:2008), the silica network is fairly stable. The enamel surface is leached and silica is dissolved only to a small extent. This type of attack is clearly represented by the vapour phase attack. In the liquid phase, some inhibition can be observed with highly resistant enamels. However, if the enamel being tested is weak, leached alkali from the enamel can raise pH-values to alkaline levels, thus increasing the attack by the liquid phase. Both liquid and vapour phase testing can give valuable information.

f) Since the attack may or may not be linear, the results are expressed only in terms of loss in mass per unit area, and the test time should be indicated.

g) For standard detergent solution (see Clause 9 of ISO 28706-3:2008), it will not be certain whether the linear part of the corrosion curve will be reached during testing for 24 h or 168 h. Calculation of the corrosion rate is therefore not included in the test report.

h) For other acids (see Clause 14 of ISO 28706-2:2008) and other alkaline solutions (see Clause 10 of ISO 28706-3:2008 and Clause 10 of ISO 28706-4:2008), it will also not be known if a linear corrosion rate will be reached during the test period. Calculation of the corrosion rate is therefore not included in the test reports of those parts of this International Standard.

For vitreous enamels fired at temperatures below 700 °C, the test parameters (media, temperatures and times) of this International Standard are not appropriate. For such enamels, for example aluminium enamels, other media, temperatures and/or times should be used. This can be done following the procedures described in the clauses for “Other test solutions” of Parts 1, 2, 3 and 4 of this International Standard.

Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 2: Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours

1 Scope

This part of ISO 28706 describes a test method for the determination of the resistance of flat surfaces of vitreous and porcelain enamels to boiling acids, boiling neutral liquids and/or their vapours.

This method allows the determination of the resistance of vitreous and porcelain enamels to the liquid and vapour phases of the corrosive medium simultaneously.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 649-1, *Laboratory glassware — Density hydrometers for general purposes — Part 1: Specification*

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

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 4799, *Laboratory glassware — Condensers*

ISO 28764, *Vitreous and porcelain enamels — Production of specimens for testing enamels on sheet steel, sheet aluminium and cast iron*

3 Principle

A set of similarly enamelled test specimens is placed in the liquid zone and/or in the vapour zone of the test apparatus, as required, and exposed to attack by a boiling acid or boiling neutral liquid, or its vapour, under specified conditions.

The same design of test apparatus and the same test principle is employed for the different liquids.

The loss in mass is determined and used to calculate the rate of loss in mass per unit area and, if necessary, the corrosion rate.

4 Reagents

During the determination, use only reagents of recognized analytical grade, unless otherwise specified.

4.1 Water, conforming to the requirements of grade 3 of ISO 3696, i.e. distilled water or water of equivalent purity.

4.2 Acetic acid solution, volume concentration 50 ml/l, for cleaning the test apparatus and test specimens.

4.3 Degreasing solvent, such as ethanol, or water containing a few drops of liquid detergent, suitable for cleaning the test apparatus and test specimens.

4.4 Citric acid monohydrate, ($C_6H_8O_7 \cdot H_2O$), crystalline.

4.5 Sulfuric acid, analytical grade, 30 % (by mass) solution, density range 1,217 g/ml to 1,220 g/ml (measured with a hydrometer — see 5.8).

4.6 Hydrochloric acid, analytical grade, 20 % (by mass) solution, density range 1,097 g/ml to 1,099 g/ml (measured with a hydrometer — see 5.8).

5 Apparatus and materials

5.1 Test apparatus

5.1.1 General description

The test apparatus (see Figures 1 and 2) consists of a cylinder (5.1.2) (see Figure 3), with an adjacent support, having a standard socket for holding a reflux condenser (5.1.3) with a graduated collector (5.1.4) on one side.

Two test specimens shall form the top and bottom of the cylinder. One of them may be replaced by a glass plate (5.1.14) if required. The cylinder with the specimens shall be supported between two plates (see Figure 2) locked at the corners by threaded bolts (5.1.8), wing nuts (5.1.7) and hexagonal nuts (5.1.6). A synthetic-fibre washer (5.1.9) is fixed between the plates (5.1.5) and each specimen. The specimens are sealed against the ends of the cylinder with packing rings (5.1.10), the material of which is dependent on the type of test solution. Any uncoated area of the test specimen shall be protected from exposure to the attacking medium.

When testing specimens cut from an enamelled article, the packing rings (5.1.10) are replaced by protective envelopes (see Figure 5) in which the specimens are placed.

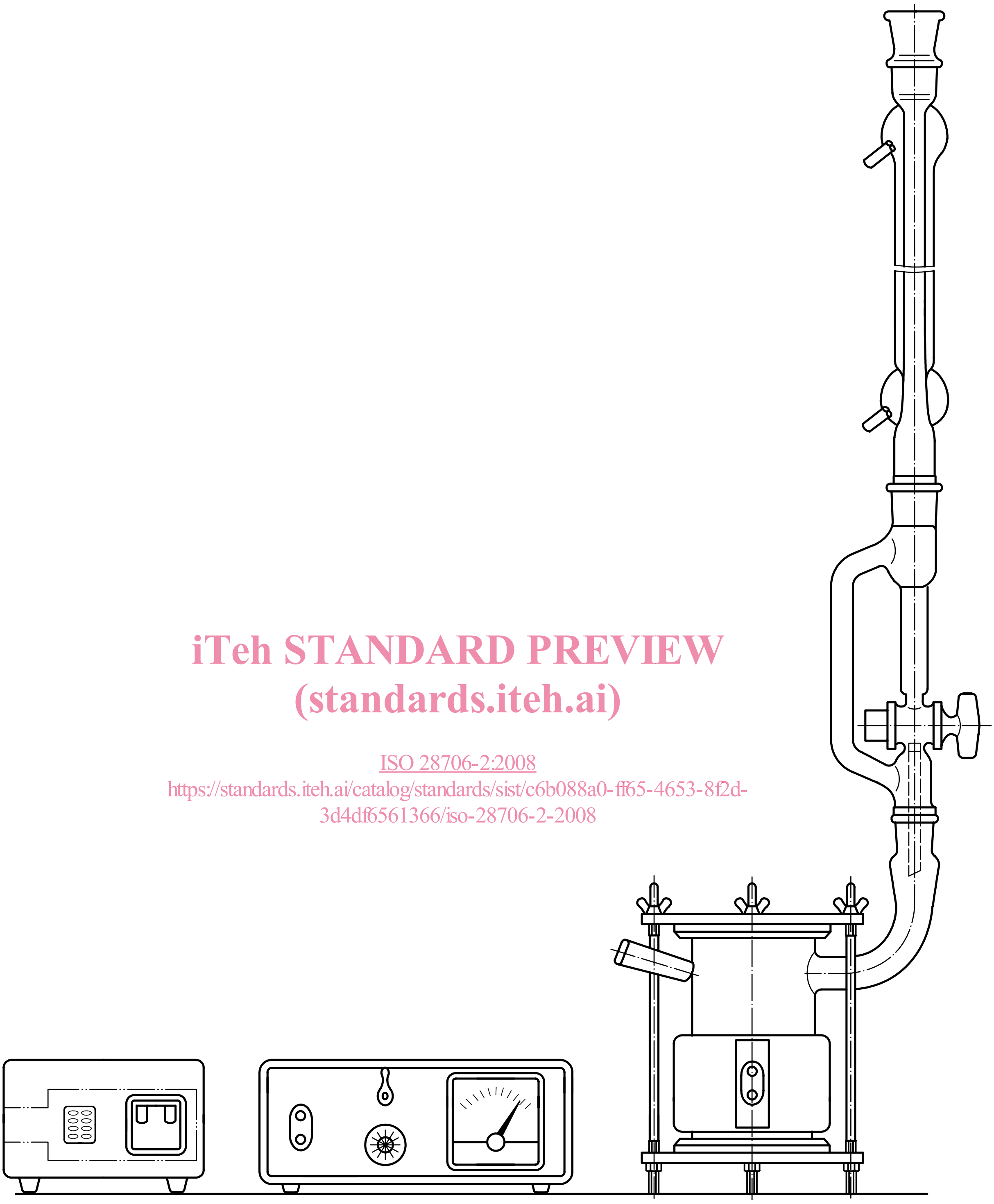
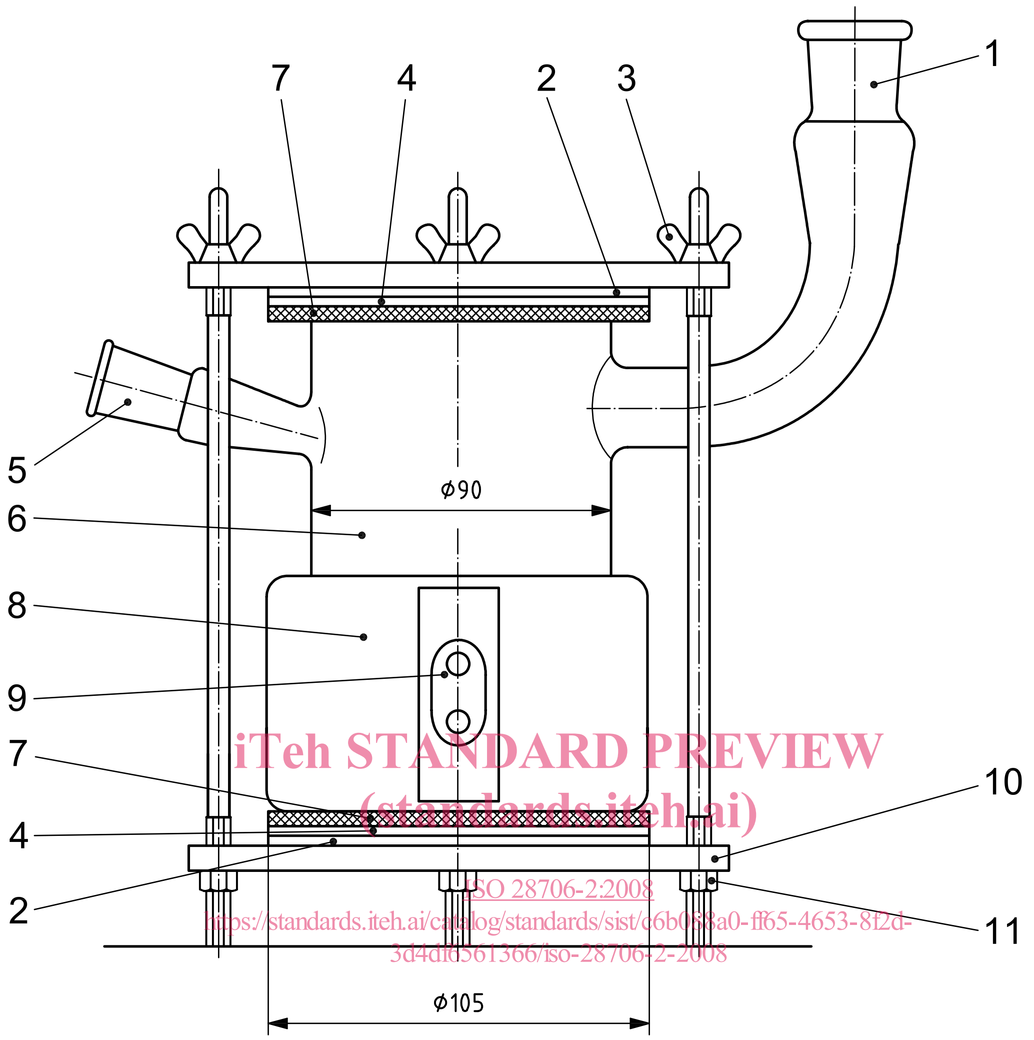


Figure 1 — View of assembled test equipment

Dimensions in millimetres



Key

1 socket for reflux condenser
2 synthetic-fibre washer
3 wing nut
4 specimen
5 socket for thermometer
6 cylinder
7 packing ring
8 heater
9 electric socket
10 triangular plate
11 hexagonal nut

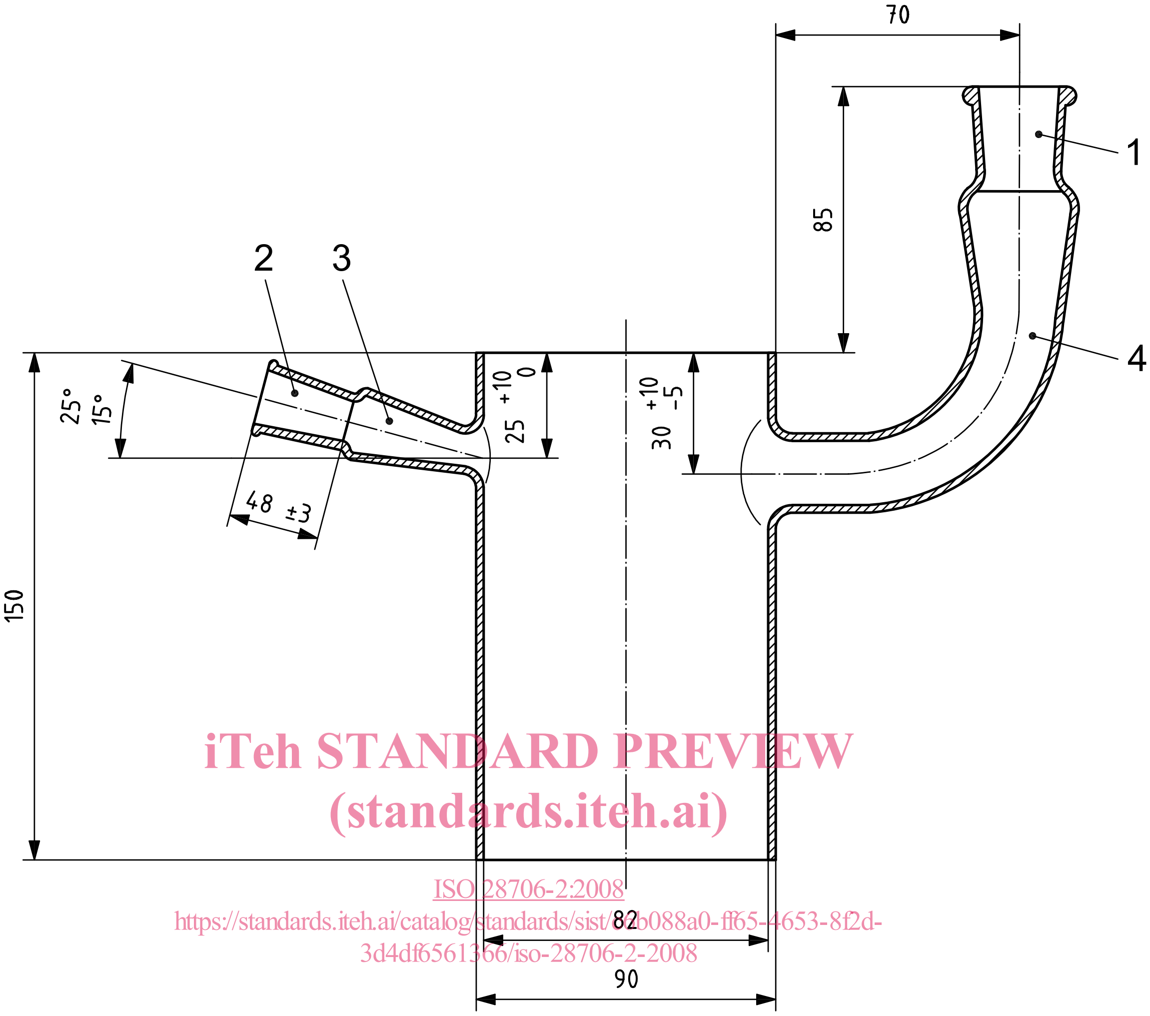
Figure 2 — Test apparatus

The apparatus is heated externally by a heater (5.1.11) placed round the lower half of the cylinder (5.1.2) such that the lower edge is, at the most, 3 mm above the lower packing ring. The test apparatus is composed of the following parts:

5.1.2 Cylinder (see Figure 3), made of borosilicate glass 3,3 conforming to the requirements of ISO 3585, with ground ends. When tested in accordance with ISO 718, the cylinder shall pass the test without breaking at a difference in temperature of at least 120 °C.

NOTE Cylinders having two sockets can also be used if the smaller socket is closed by a stopper which is resistant to the boiling solution.

Dimensions in millimetres



Key

1 ground-glass socket for condenser

2 ground-glass socket for thermometer

3 connecting piece

4 connecting piece

Figure 3 — Cylinder

5.1.3 Liebig-West reflux condenser, or equivalent reflux condenser conforming to ISO 4799, in which there is no volume change during the test, with a nominal jacket length of 400 mm and standard ground joint of borosilicate glass 3,3 conforming to the requirements of ISO 3585.

5.1.4 Graduated collector (see Figure 4), with a standard ground joint of borosilicate glass 3,3 conforming to the requirements of ISO 3585, arranged in the apparatus to collect the condensate produced in the reflux condenser. The graduation interval shall be 0,1 ml.