INTERNATIONAL STANDARD

ISO 4534

Second edition
2010-12-15

Vitreous and porcelain enamels — Determination of fluidity behaviour — Fusion flow test

Émaux vitrifiés — Détermination du comportement de fluidité — Essai d'écoulement

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Reference number
ISO 4534:2010(E)

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Published in Switzerland

Foreword

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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 4534 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 4534:1980), which has been technically revised. The revised edition allows for the use of flow plates, other than unglazed ceramic tiles, such as enamelled steel or cast iron which is common practice in some countries.

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Introduction

The fusion flow test specified in this International Standard is a comparative method which can be carried out with simplified equipment to provide data on the fluidity behaviour of molten enamel. The results from this test allow conclusions on the flow properties of the enamel to be inferred in a much simpler manner than is possible from the results of the much more expensive measurements made using the usual viscosity-measuring instruments.

The results of extensive tests[1)] have shown that there is a well-defined relationship between the results of the flow test and the viscosity-temperature curve, so that the flow test could also be used as an absolute method. However, more effort would be required to enable the various laboratories to obtain comparable results of similar quality than when using the method for comparative purposes.

When using this method, the reference (comparison) enamel must be similar to the enamel to be tested, as the fluidity behaviour of the various types of enamel might vary considerably from one type to another.

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1) Dekker, P. Calculation of viscosity-temperature curves for porcelain enamels from the flow-button test. *Journal of the American Ceramic Society*, **48** (1965), 6, pp. 319 to 327.

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Vitreous and porcelain enamels — Determination of fluidity behaviour — Fusion flow test

WARNING — This International Standard calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This International Standard does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health, safety and environmentally acceptable practices and take suitable actions for any national and international regulations. Compliance with this International Standard does not in itself confer immunity from legal obligations.

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1 Scope

This International Standard specifies a comparative method of determining the fluidity behaviour of vitreous and porcelain enamels in the viscous condition during firing. It is not intended for use as an absolute method.

2 Principle

The test samples are dried or wet-ground in accordance with the processing conditions. Cylindrical specimens of specified mass are pressed from the enamel powder, or the dried enamel slip, and from the reference enamel agreed upon between the parties concerned.

The test specimens are placed in a laboratory furnace at an agreed temperature on an unglazed ceramic tile in the horizontal position and melted to hemispherical shape. The tile is tilted to permit the enamel to flow at a specified angle and for an agreed time period.

The length flow number, F_l, and the breadth flow number, F_b, are calculated on the basis of the flow lengths and flow breadths of the test specimens.

3 Material and apparatus

3.1 Reference enamel, to be agreed upon by the parties concerned, having similar fluidity behaviour to the enamel to be tested.

3.2 Ball mill.

3.3 Evaporation device, for example, a hot-air oven, a hot plate, a sand bath.

3.4 Mortar.

3.5 Pestle.

3.6 Balance, with an accuracy of 0,01 g.

3.7 Press, able to generate a pressure of at least 5 N/mm^2, with a cylindrical mould having an internal diameter of 8 mm to 10 mm for preparation of the test specimens.

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3.8 Flow plate, consisting preferably of a smooth unglazed ceramic tile, with side lengths of approximately 75 mm and a thickness of 5 mm to 6 mm, prefired at a temperature of at least 1 100 °C. It shall have a water absorption at atmospheric pressure $\leqslant$ 25% and a homogeneous fine ceramic body. Flow plates may also be cut from a larger plate (see A.1).

3.9 Tilting frame, (see Figures 1 and 2), for placing a flow plate inside a laboratory furnace in a horizontal position and tilting the plate to an angle between 30° and 90°.

NOTE For very viscous samples, it can be favourable to increase the slope; for samples of low viscosity, it can be favourable to decrease the slope.

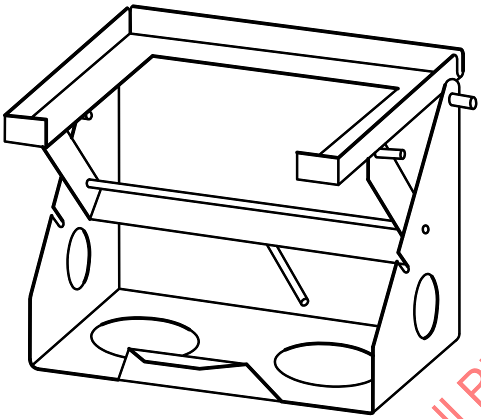
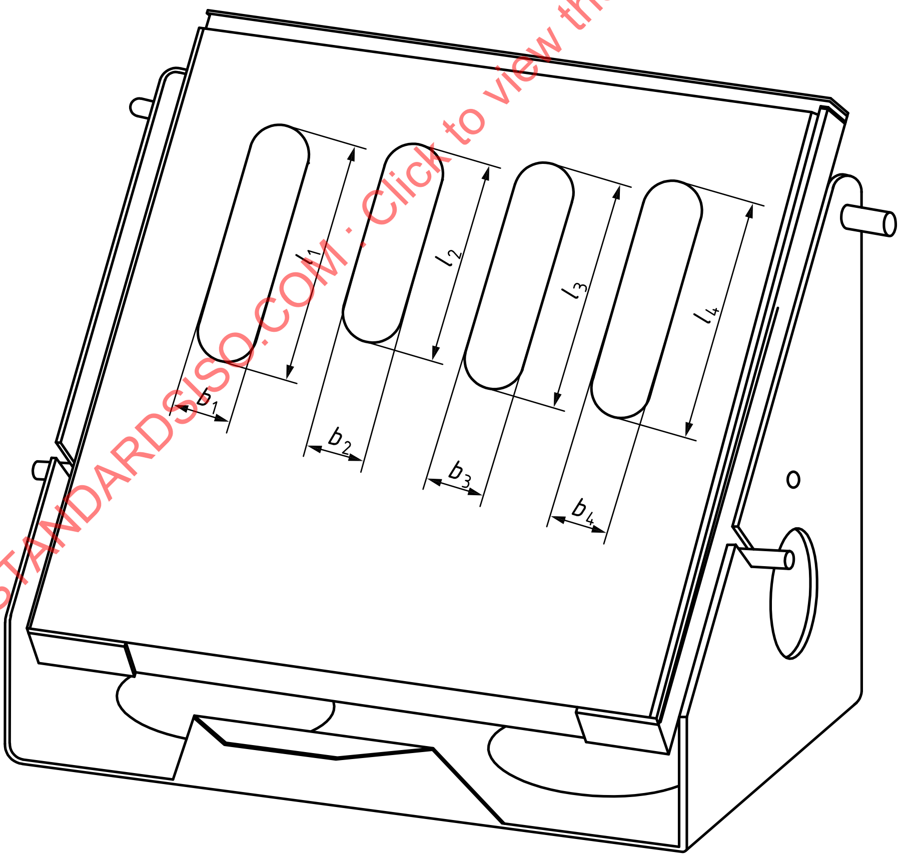


Figure 1 — Example of tilting frame set in a horizontal position for receiving the flow plate



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Key

l_1, l_2, l_3 and l_4 flow lengths of the four specimens

b_1, b_2, b_3 and b_4 maximum flow breadths of the four specimens (see Clause 6)

Figure 2 — Tilting frame tilted by 45° with flow plate and four specimens

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3.10 Electrically heated laboratory furnace, capable of being controlled at (900 ± 10) °C.

3.11 Stop watch.

4 Test specimens

4.1 Preparation of enamel

The sample may be taken from the enamel powder readily ground or it may be ground separately in the ball mill (3.2). Mill additives and the fineness of grinding depend on the manufacturing conditions. In special cases, the complete grain-size distribution shall be taken into account.

Wet-ground enamels shall be evaporated to dryness in an evaporation dish. After cooling, the dried enamel shall be loosened and again pulverized using the pestle (3.5) and mortar (3.4).

If agreed, mill additives, which are completely or partially soluble in water and are only used as a setting-up agent, may be omitted.

Where the fluidity of frits only is to be determined, they should be milled dry.

4.2 Preparation of test specimens

The enamel shall have a mass of 1 g to 2 g, with a relative deviation of maximum 3 %. As specified in 4.1, the enamel powder shall be mixed with a defined amount of water in an empty mould (see 3.7 and A.2). Immediately press the specimen at a pressure of at least 5 N/mm^2. It is important to prepare all test specimens in the same manner with the same number of water drops.

4.3 Number of tests

Each flow test shall be performed with an agreed number of specimens made of the test enamel and at least one specimen made of the reference enamel (see Clause 5).

5 Procedure

Place the agreed number of test specimens and at least one specimen of the reference enamel on the flow plate (3.8) within the supporting area (see Figure 3). If various flow tests are carried out, interchange the positions of the test specimens and the specimen made of the reference enamel (see A.3).

It is recommended to have one reference in the side position and to have another reference in the middle of the plate.

Predetermine, by one or more pre-tests, the holding time, i.e. the time required from placing the flow plate in the laboratory furnace (3.10), maintained at the agreed temperature for the test enamel, until the reference specimen softens sufficiently to form an approximate hemisphere.

The test specimens shall be completely dry before the flow test is carried out. Carefully place the flow plate in the laboratory furnace, horizontally on the tilting frame (3.9), and at the end of the holding time, then tilt it (see A.3).

At the end of the flow period, i.e. the agreed period over which the enamel is allowed to flow, remove the flow plate from the furnace.

Measure the flow lengths and maximum flow breadths of the test specimens, in millimetres (see Figure 2).

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Dimensions in millimetres

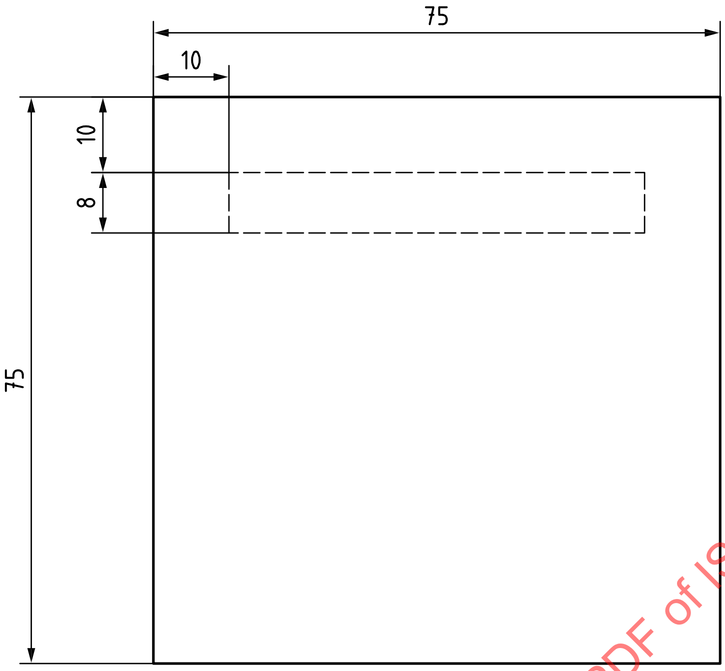

Figure 3 — Example of supporting area of specimens on flow plate

6 Calculation and expression of results

Calculate the length and breadth flow numbers, F_l and F_b, from Equations (1) and (2):

Length flow number:

$$F_l = \frac{l_t}{l_r} \tag{1}$$

where

l_t is the flow length of the test enamel;

l_r is the flow length of the reference enamel.

Breadth flow number:

$$F_b = \frac{b_t}{b_r} \tag{2}$$

where

b_t is the maximum flow breadth of the test enamel;

b_r is the maximum flow breadth of the reference enamel.

If several test specimens or reference specimens are used, use the mean flow length and the mean maximum flow breadth for the calculation.

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7 Test report

The test report shall include the following information:

a) a reference to this International Standard, ISO 4534:2010;

b) name of testing establishment;

c) designations of the test enamel and the reference enamel;

d) temperature in the laboratory furnace;

e) flow period;

f) number of specimens used in the test;

g) number of tests;

h) length flow number, F_l, and breadth flow number, F_b, as individual values and arithmetic means;

i) any deviations from the procedure specified;

j) any unusual features observed during the test;

k) date of test.

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