International Standard 4534

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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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ISO 4534-1980 (E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4534 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in December 1978.

It has been approved by the member bodies of the following countries :

Australia	Israel	Romania
Czechoslovakia	Italy	South Africa, Rep. of
Germany, F.R.	Japan	Switzerland
Hungary	Netherlands	Turkey
India	Poland	USA

The member bodies of the following countries expressed disapproval of the document on technical grounds :

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

0 Introduction

The fusion flow test described 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 may vary considerably from one type to another.

1 Scope and field of application

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.

It is applicable to molten enamels, but not to sintered ground coat enamels.

2 Principle

Dry or wet grinding of test samples in accordance with the processing conditions. Pressing of cylindrical specimens of specified weight from the enamel powder, or the dried enamel slip, and from the agreed reference enamel.

Placing of the specimens in a laboratory oven at an agreed temperature on an unglazed ceramic tile in the horizontal position and melting to hemispherical shape. Tilting of the tile to permit the enamel to flow at an angle of 45° for an agreed period.

Calculation of the length flow number, F_l, and the breadth flow number, F_b, on the basis of the flow lengths and flow breadths of the specimens.

3 Material and apparatus

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

3.2 Ball mill.

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

3.4 Mortar.

3.5 Pestle.

3.6 Balance, accurate to 0,01 g.

3.7 Press, giving a pressure of at least 5 N/mm^2 (5 MPa), and a **mould** having an internal diameter of 8 mm for preparation of the test specimens.

3.8 Flow plate, consisting of a square smooth unglazed ceramic tile, of side 75 mm, 5 to 6 mm thick and prefired at a temperature of at least 1 100 °C. It shall have a water absorption at atmospheric pressure of not more than 25 % and a homogeneous fine ceramic body. Flow plates may also be cut from a larger plate (see clause A.1).

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.

3.9 Tilting frame, (see figures 1 and 2), which permits the placing of a flow plate inside a laboratory oven in a horizontal position and tilting of the plate by 45°.

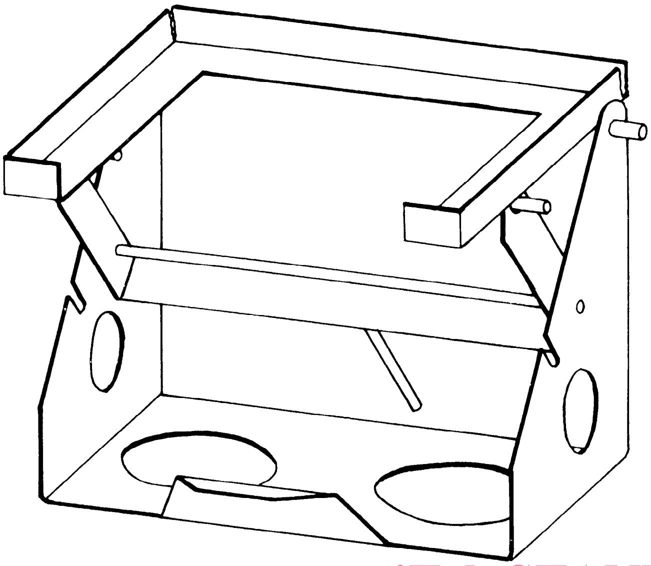

Figure 1 — Example of tilting frame, set for receiving the flow plate, in a horizontal position

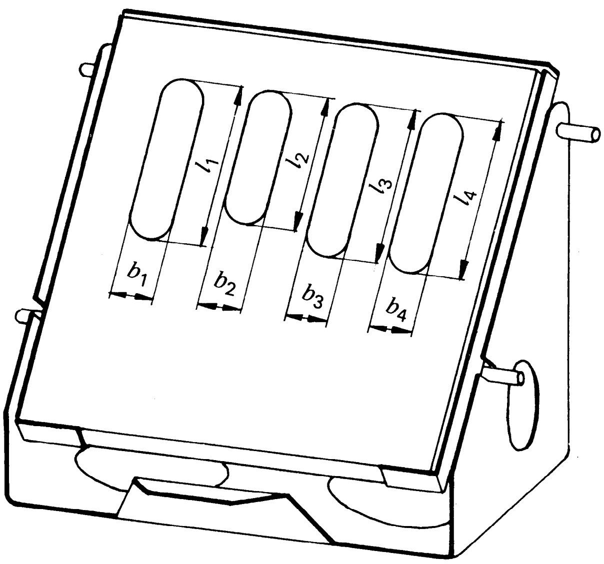

Figure 2 — The tilting frame with flow plate and four specimens tilted by 45°

NOTE — l_1, l_2, l_3 and l_4 are the flow lengths of the four specimens; b_1, b_2, b_3 and b_4 are their maximum flow breadths (see clause 6).

3.10 Electrically heated laboratory oven, allowing temperatures of up to 900 °C to be kept constant to within 10 °C.

3.11 Stop watch.

4 Test specimens

4.1 Preparation of enamel

The sample may be taken from the already ground enamel powder or 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 evaporating dish. After cooling, the dried enamel shall be loosened and again pulverised using the pestle (3.5) and mortar (3.4).

NOTES

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

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

4.2 Preparation of test specimens

Place one drop of water in an empty mould (see 3.7), add 1 ± 0,01 g of the enamel prepared as specified in 4.1 and then another drop of water (see clause A.2). Immediately press the specimen at a pressure of at least 5 N/mm² (5 MPa).

4.3 Number of test specimens

In each flow test, an agreed number of specimens made of the test enamel and one specimen made of the reference enamel shall be used (see clause 5).

4.4 Number of tests

In each determination, at least two tests for each set of specimens shall be carried out.

5 Procedure

Place the agreed number of test specimens and one specimen of the reference enamel on the flow plate (3.8) within the supporting area (see figure 3). During the various flow tests, interchange the positions of the test specimens and the specimen made of the reference enamel (see clause A.3).

Dimensions in millimetres

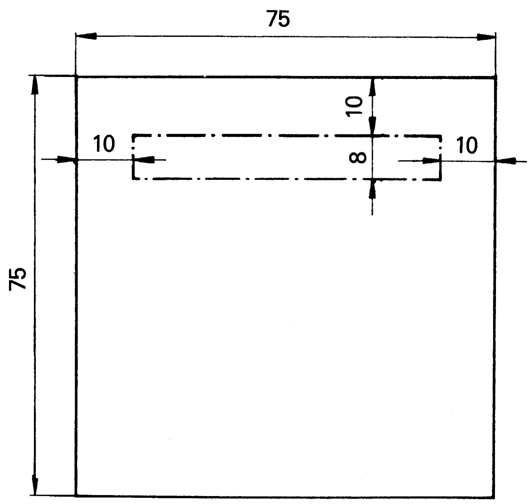

Figure 3 — Supporting area of specimens on flow plate

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

At least 30 min after preparation of the specimens, carefully place the flow plate in the laboratory oven, horizontally on the tilting frame (3.9), and, at the end of the holding period, tilt it through 45° (see clause 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 oven.

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

6 Expression of results

Calculate the length and breadth flow numbers, F_l and F_b, from the following formulae :

a) length flow number :

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

where

l_t is the flow length of the test enamel,

l_r is the flow length of the reference enamel;

b) breadth flow number :

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

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 are used, use the mean flow length and the mean maximum flow breadth for the calculation.

7 Test report

The test report shall include the following particulars :

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

b) the temperature in the laboratory oven;

c) the holding period;

d) the flow period;

e) the number of specimens used in the test;

f) the number of tests;

g) the length flow number, F_l, and the breadth flow number, F_b, giving both their individual values and their arithmetic means;

h) date of test.

Annex

Additional information

A.1 Flow plate (3.8)

Unglazed ceramic tiles have proven their value as flow plates. They can be used equally satisfactorily for powder enamel (dry-process enamelling) and wet-cast enamel, and for sheet steel porcelain enamel.

They are lower in cost than plates made of a casting or of sheet metal, which have to be enamelled first with a ground enamel matching the enamel to be tested. Ground enamel on sheet metal and fusible ground enamel on castings would, of necessity, be brought back into the fused, molten condition during the fusion flow test and might thereby influence the flow of the specimen. Another inhibiting factor during reheating of the previously fired ground enamel would be the possibility of reactions between the ground enamel and the specimen.

Porcelain boats are more expensive than unglazed ceramic tiles and their side walls would also obstruct the enamel in its spread, which provides an indication of the wetting porperties of the enamel in relation of the ceramic bases used for enamelling.

A.2 Mass of test sample for moulding (see 4.2)

A given quantity (1 ± 0,01 g) has been specified for the specimens, as, otherwise, density and fineness of grinding of the enamel would have to be considered.

A.3 Procedure (clause 5)

In the tests for each set of specimens, the positions on the flow plate of the test and reference specimens are interchanged so that the influence of any temperature gradients which might be present in the oven is eliminated.

The start of the flow period has been defined as occuring when the reference specimen has attained roughly a hemispherical shape. If the specimen has proceeded beyond the hemispherical shape prior to tilting, that is, if the specimen has spread too far, it will contract again during the flow process and become narrower from the top to the bottom. If the specimen has not attained the hemispherical shape, it will effectively roll down the flow plate instead of flowing. If the flow plate is tilted while the specimen is still in its cylindrical shape, perfect results cannot be expected, since the sintering and melting processes, occurring inside the specimen, may prevent reproducible flow.

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