

INTERNATIONAL
STANDARD

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Ceramic tiles —

Part 15:

Determination of lead and cadmium given off by
glazed tiles

Carreaux et dalles céramiques —

*Partie 15: Détermination de la teneur en plomb et en cadmium dégagés par
les carreaux émaillés*



Reference number
ISO 10545-15:1995(E)

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 10545-15 was prepared by Technical Committee ISO/TC 189, *Ceramic tile*.

ISO 10545 consists of the following parts, under the general title *Ceramic tiles*:

- *Part 1: Sampling and basis for acceptance*
- *Part 2: Determination of dimensions and surface quality*
- *Part 3: Determination of water absorption, apparent porosity, apparent relative density and bulk density*
- *Part 4: Determination of modulus of rupture and breaking strength*
- *Part 5: Determination of impact resistance by measurement of coefficient of restitution*
- *Part 6: Determination of resistance to deep abrasion for unglazed tiles*

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- Part 7: Determination of resistance to surface abrasion for glazed tiles
- Part 8: Determination of linear thermal expansion
- Part 9: Determination of resistance to thermal shock
- Part 10: Determination of moisture expansion
- Part 11: Determination of crazing resistance for glazed tiles
- Part 12: Determination of frost resistance
- Part 13: Determination of chemical resistance
- Part 14: Determination of resistance to stains
- Part 15: Determination of lead and cadmium given off by glazed tiles
- Part 16: Determination of small colour differences
- Part 17: Determination of coefficient of friction

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Ceramic tiles —

Part 15:

Determination of lead and cadmium given off by glazed tiles

1 Scope

This part of ISO 10545 specifies a method for the determination of lead and cadmium given off by the glaze of ceramic tiles.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10545. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10545 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 3696:1987 *Water for analytical laboratory use — Specification and test methods.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

3 Principle

Exposition of the glazed surface of a ceramic tile to an acetic acid solution. Determination of the amount of lead and cadmium released into the solution by an appropriate method.

4 Reagents

During the analysis, unless otherwise indicated, use only the reagents specified in ISO 6353-2 if indicated, if not, use reagents of recognized analytical grade, and grade 2 complying with ISO 3696:1987.

4.1 Test solution: acetic acid solution, 4 % (V/V). Add 40 ml of glacial acetic acid (R 1 of ISO 6353-2:1983) to 960 ml of distilled water of grade 2.

5 Apparatus and materials

5.1 Atomic absorption spectrometer, or other suitable apparatus for the analysis of lead and cadmium from solution.

5.2 Silicone sealant in a tube or dispenser, enabling a ribbon of clear silicone (neutral cure) sealant approximately 6 mm in diameter to be formed.

5.3 Impervious cover, made of glass or plastics.

5.4 Detergent.

5.5 White cloth, made of cotton or flax.

5.6 Measuring cylinder.

6 Test specimens

6.1 Number of test specimens

A minimum of three whole tiles shall be tested.

6.2 Preparation of test specimens

The surface of a tile to be tested shall be clean and free from grease or other material that may interfere with the performance of the test. To ensure cleanliness, the tile shall be thoroughly washed in tap water containing a small amount of detergent (5.4),

and rinsed with grade 2 water, then dried either by draining or with a soft clean cloth (5.5). After washing, care shall be taken to avoid handling the glazed surface.

Apply a ribbon of silicone sealant (5.2) 6 mm wide, to the glazed surface around the whole perimeter. Ensure visually that the ribbon is complete and has contact with the glazed surface around the whole perimeter. Also ensure that it is high enough to allow a sufficient volume of the test solution (4.1) to be added. The minimum height of the silicone sealant over the glazed surface shall be 4 mm. Allow the sealant to dry overnight.

Measure and calculate the area A , in square decimetres, of the surface to be tested.

7 Procedure

7.1 Extraction with acetic acid

Place each tile on a flat horizontal surface in a room at a temperature of $(20 \pm 2)^\circ\text{C}$. Fill the volume formed by the ribbon of silicone sealant with a volume, V , of the test solution (4.1), measured at the same temperature using the measuring cylinder (5.6).

Place the cover (5.3) on the tile to minimize contamination and evaporation. A convenient way of doing this is illustrated in figure 1. Ensure that the temperature of the room remains at $(20 \pm 2)^\circ\text{C}$ during the test and protect the test assembly from sunlight or other thermal sources.

After 24 h, remove the cover, stir the acid thoroughly to ensure its homogeneity and remove an aliquot portion of the solution for analysis.

7.2 Determination of lead and cadmium

Determine the amount of lead and cadmium extracted by an appropriate method: atomic absorption spectrometry is a possible method. Take into account any small amounts of lead and cadmium present in the reagents and water used, for example by a blank determination.

8 Expression of results

The mass of lead (Pb) and cadmium (Cd) extracted per unit of surface $\rho_A(M)$, expressed in milligrammes per square decimetre, is calculated using the equation

$$\rho_A(M) = \rho(M) \times \frac{V}{1000} \times \frac{1}{A}$$

where

M is the metal extracted (Pb or Cd);

$\rho(M)$ is the concentration, in milligrammes per litre, of metal, M , in the extract, determined according to 7.2;

V is the volume, in millilitres, of acetic acid added to the tile;

A is the area, in square decimetres, of surface tested.

9 Test report

The test report shall include the following information:

- reference to this part of ISO 10545;
- a description of the tiles, including sample preparation;
- mass concentration of lead in the extract, $\rho(\text{Pb})$, in milligrammes per litre, and mass of lead extracted per unit of surface, $\rho_A(\text{Pb})$, in milligrammes per square decimetre;
- mass concentration of cadmium in the extract, $\rho(\text{Cd})$, in milligrammes per litre, and mass of cadmium extracted per unit of surface, $\rho_A(\text{Cd})$, in milligrammes per square decimetre.