



ADHESIVE TEST METHODS

Available Test Methods

All the information provided is based on the Spanish versions of the standards listed below, from the period 1999-2003. For the 2007 updates, according to the list in the box, a series of general comments are included, based on working documents of the WG3 Group, from February 2006.

- EN 1308 (July 1997) and EN 1308/A1 (May 1999)
Adhesives for ceramic tiles. Determination of slip
- EN 1322 (July 1997) and EN 1322/A1 (May 1999)
Adhesives for ceramic tiles. Definitions and terminology
- EN 1323 (July 1997) and EN 1323/A1 (May 1999)
Adhesives for ceramic tiles. Concrete slabs for tests
- EN 1324 (July 1997) and EN 1324/A1 (May 1999)
Adhesives for ceramic tiles. Determination of shear adhesion strength of dispersion resin adhesives
- EN 1346 (July 1997) and EN 1346/A1 (May 1999)
Adhesives for ceramic tiles. Determination of open time
- EN 1347 (July 1997) and EN 1347/A1 (May 1999)
Adhesives for ceramic tiles. Determination of wetting capability
- EN 1348 (July 1997) and EN 1348/A1 (May 1999)
Adhesives for ceramic tiles. Determination of tensile adhesion strength for cementitious adhesives
- EN 12002 (August 1997)
Adhesives for ceramic tiles. Determination of transverse deformation for cementitious adhesives and grouts
- EN 12003 (January 2003)
Adhesives for ceramic tiles. Determination of shear adhesion strength of reaction resin adhesives
- EN 12808-1 (September 1999)
Adhesives and grouts for ceramic tiles. Part 1: Determination of chemical resistance of reaction resin mortars

NEW 2007 VERSIONS OF STANDARDS FOR ADHESIVE TEST METHODS	
EN 1323	Adhesive for tiles. Concrete slabs for tests
EN 1308	Adhesive for tiles. Determination of slip
EN 1324	Adhesive for tiles. Determination of shear adhesion strength of dispersion adhesives
EN 1346	Adhesive for tiles. Determination of open time
EN 1348	Adhesive for tiles. Determination of tensile adhesion strength for cementitious adhesives
EN 12003	Adhesive for tiles. Determination of shear adhesion strength of reaction resin adhesives
prEN 12002	Adhesive for tiles. Determination of transverse deformation [Pending approval July 2008]

EN 1308 DETERMINATION OF SLIP

Subject matter and scope

Test method for determining the resistance of adhesives to the vertical slip of ceramic tiles. Applies to all adhesives used for the installation of indoor and outdoor coverings.

Test conditions

Temperature: 23 ± 2 °C
Relative humidity: 50 ± 5 %
Air velocity: max. 0.2 m/s

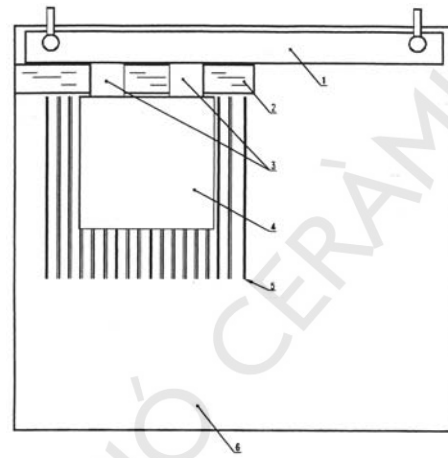
Material

- *Ceramic tiles*: Type V2 [BI_a with $E \leq 0.2\%$, UGL, flat bonding surface, format 100x100 mm and weight (200 ± 10) g]
- *Adhesive*: Mix with water or liquid with the proportions indicated by the manufacturer. Use of a mixer such as described in EN 196-1, section 4.4, with a rotation speed of 140 ± 5 rpm and a planetary movement speed of 62 ± 5 rpm. 2 kg of adhesive are used
- *Preparing the adhesive*: Under the standard conditions proposed in the standard:
 - Pour the liquid into the mixer container
 - The powder slowly disperses in the liquid
 - Mix for 30 s and then clean off the blade and container (1 min)
 - Final mix and mixing for 1 min
 - When necessary, leave the adhesive to rest (resting or maturing time)

Following the manufacturer's instructions, leave the adhesive to rest and then mix for 15 s.

Procedure

- 1- Mount the device in the figure on the concrete slab manufactured according to EN 1323.
- 2- The adhesive is combed with a 6 x 6 mm notched trowel with a distance of 12 mm between adjacent notches (cementitious adhesives) or a 4 x 4 mm notched trowel with a distance of 8 mm (dispersion and reaction resin adhesives)
- 3- Remove the adhesive tape and place the spacing wedges
- 4- After two minutes, install the tiles and place a weight of (50 ± 0.1) N on them for (30 ± 5) s.
- 5- Remove the spacing wedges and measure the distance from the edge of the tiles using the ruler, with an accuracy of ± 0.1 mm
- 6- Place the slab in the vertical position and measure the distance again after (20 ± 2) min
- 7- The difference between the two distances is the slip, with an accuracy of ± 0.1 mm



- 1 Steel ruler
- 2 25-mm-wide adhesive tape
- 3 25x25x10 mm spacing wedges
- 4 Type V2 100x100 mm tile
- 5 Adhesive
- 6 Concrete slab

For each type of adhesive, the test is performed on three tiles and the slip measurement is the arithmetic mean of the three obtained values.

The new 2007 version uses the same type of trowel for the three adhesives, i.e. 6 x 6 mm.

EN 1324

DETERMINATION OF SHEAR ADHESION STRENGTH OF DISPERSION ADHESIVES

Subject matter and scope

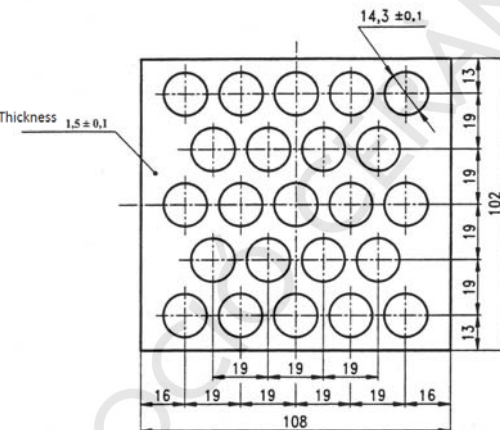
Test method for determining the shear adhesion strength of dispersion resin adhesives used for the installation of ceramic tiles and other compatible modular materials in indoor coverings.

Test conditions

As described in EN 1308.

Material

- *Ceramic tiles:* Type **P2** [BIII, with WAC $E=(15\pm 3)\%$, with flat bonding surface, format 108x108 mm and thickness of 6 mm]



Measurements in millimetres

Material: PTFE or the like, with non-bonding properties

Hole diameter: (14.3 ± 0.1) mm

Contact surface of the adhesive when compressed $(50\pm 5)\%$

Thickness: (1.5 ± 0.1) mm

The ready-to-use adhesive must be within its expiry date

Procedure

The test specimens are prepared with the help of a template and spacers, according to the figure, for the application of adhesive by points on the frame. This frame is placed on the rear of the tile, the adhesive is applied and the spacers are placed after removing the frame. Then it is placed facing a second tile by its rear, offset 6 mm from the first tile.

After exerting a force of (70 ± 0.15) N for 3 min., the spacers are removed, making sure to maintain the relative position of the tiles. After **14 days** under laboratory conditions, they can be subjected to tests. Ten test specimens are required for each test.

- ▶ *Initial bonding force:* After 14 days under normal laboratory conditions, the test specimens are mounted in the device for testing shear adhesion strength (traction machine or vertical compression machine), applying a movement force until they break. The measured breakage force, divided by the surface area of the tile ($5,480 \text{ mm}^2$), gives the initial shear adhesion strength (N/mm^2).
- ▶ *Bonding strength after immersion in water:* The same process is followed using test specimens that have been **seven days** under laboratory conditions and another **seven days** submerged in water at room temperature. Feature to be measured on dispersion adhesives intended to be used for indoor coverings exposed to damp.



- ▶ *Bonding strength after ageing by heat:* The test is performed on 10 test specimens that have been **14 days** under normal temperature conditions and another **14 days** in a kiln at (70 ± 2) °C with a permanent air flow. The shear adhesion test is carried out 24 hours after removing the test specimens from the kiln.
- ▶ *Bonding strength at high temperature:* The test is performed on test specimens that have undergone the same cycle as for ageing by heat, but **1 minute after** removing the test specimens from the kiln.

The new 2007 version also provides clearer indications on the type of tile to be tested: water absorption capacity of (15 ± 1) %, thickness of 6 to 8 mm, maximum relief of 0.25 mm and final surface area for testing bonding of 5508 mm².

EN 1346 DETERMINATION OF OPEN TIME

Subject matter and scope

Defines the test method for determining the open time of all types of adhesives used for installing ceramic tiles or compatible modular materials in coverings and floorings, both indoors and outdoors.

The **open time**, expressed in minutes, is the maximum time interval during which the adhesive has at least the tensile adhesion strength specified in standard EN 12004; in other words, 0.5 N/mm² as the minimum value for every type of adhesive.

Test conditions

As described in **EN 1308**.

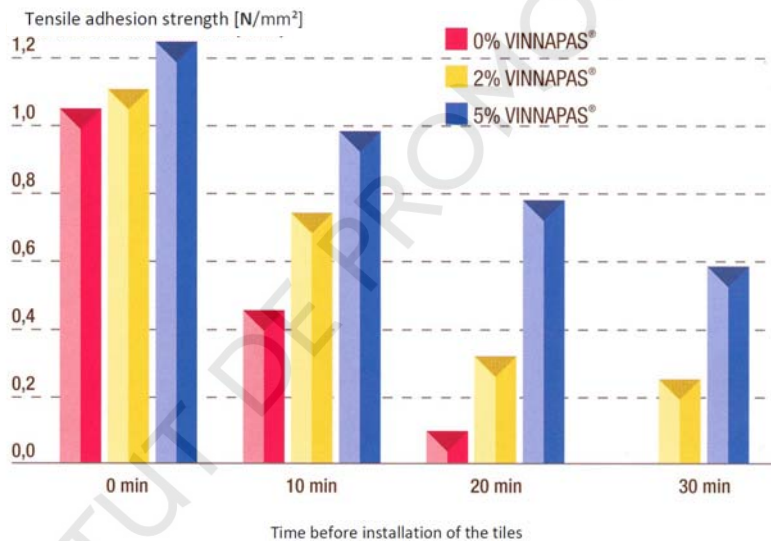
Material and equipment

- Type **P1** ceramic tiles [BIII, with WAC $E=(15\pm 3)$ %, with flat bonding surface, cut to 50x50 mm]
- Concrete slab according to EN 1323
- Pile caps of 50x50 mm with a thickness of 10 mm, and tensile testing machine which provides a tensile force, increasing by (250 ± 50) N per second
- The cementitious adhesives must be prepared in accordance with the procedure described in **EN 1308**

Procedure

- 1- A first contact layer of the adhesive to be tested is applied to the concrete slab. Immediately afterwards, a second layer is applied, combing it with a 6x6 mm (cementitious adhesives) or 4x4 mm (dispersion or reaction resin adhesives) notched trowel. The notched trowel must form an angle of 60° with the concrete slab.
- 2- After waiting for 5, 10, 20 and 30 min, the type **P1** ceramic tiles are installed and a force of (20±0.05) N is exerted on them for 30 s.
- 3- After 27 days of storage under laboratory conditions, the pile caps are adhered using epoxy resin and, after 24 hours, the tensile adhesion strength is tested.

The test specimens that exceed the tensile adhesion strength of 0.5 N/mm² will define the open time, **as a maximum time interval** between applying the adhesive using a notched trowel and the placement of the corresponding tile.



Open time according to tensile adhesion strength (EN 1346) of an adhesive for ceramic tiles (35% Portland cement, 0.3% cellulose ether) tested on stoneware tiles (storage: 28 days at 23 °C / 50% RH).

Only cementitious adhesive with a polymer resin content of 5% exceeds the tensile adhesion strength of 0.5.

The new 2007 version also provides greater details regarding the type of tile, like EN 1324. The type of trowel is standardised for every type of adhesive (6x6 mm).

EN 1347

DETERMINATION OF WETTING CAPABILITY

Subject matter and scope

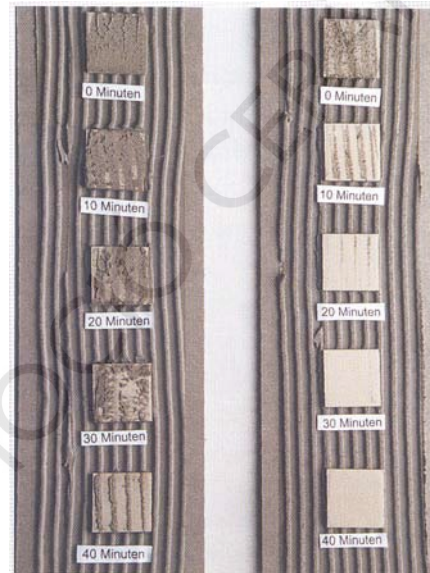
Test method designed for measuring the wetting capability of any type of adhesive, this capability being understood to be the surface of the rear of the tile that is covered with the adhesive spread over the installation surface and combed with a notched trowel once the tile has been pressed against the surface and then removed for checking.

Test conditions

As described in **EN 1308**.

Test material

- Concrete slab according to **EN 1323**
- Dry, clean sheets of glass, 100x100 mm and 6 mm thick, with polished edges
- The cementitious adhesives are prepared according to the method described in **EN 1308**



Different wetting capability or capacity between a cementitious adhesive that contains polymers (left) and one that does not contain any (right), for different open times.

SOURCE: Wacker Polymer Systems

Procedure

A first thin (contact) layer of the adhesive to be tested is applied to the concrete slab, using the sharp edge of the notched trowel.

A second thicker layer of adhesive is applied and combed using the notched trowel with square 6x6 mm notches with a separation of 12 mm (notch centre-to-centre distance) for the cementitious adhesives, and the notched trowel with square 4x4-mm notches with a separation of 8 mm for dispersion resin or reaction resin adhesives.

The sheets of glass are placed on the combed adhesive immediately and after 10, 20 and 30 min., and subjected to a load (force) of (50 ± 0.5) N for 30 s.

Three sheets of glass are required for each test. The contact surface between the combed layer of adhesive and the sheet of glass is determined, expressing the **wetting capability** as the percentage (%) of “wet” surface (covered with the adhesive) compared with the total surface area of the sheet ($10,000 \text{ mm}^2$).

For greater accuracy, the test method recommends calculating the wet surface by planimetry.

Observation

Especially in outdoor installations, but recommended for all ceramic tile installation work, it is advisable to unstick tiles periodically to check their wetting capability, since working conditions will always be worse than laboratory conditions.

With high temperatures and wind, **wetting capability** and **open time** are reduced drastically!

EN 1348

DETERMINATION OF TENSILE ADHESION STRENGTH FOR CEMENTITIOUS ADHESIVES

Subject matter and scope

Measures the tensile adhesion strength, in N/mm^2 , of the cementitious adhesives used for the installation of ceramic tiles and compatible modular rigid materials, in floorings and coverings, indoors and outdoors.

Test conditions

As described in **EN 1308**.

Material

- Concrete slab according to **EN 1323**
- Ceramic tiles: Type **VI** [BI_a UGL tiles, with water absorption capacity $E \leq 0.2\%$, with flat bonding surface and dimensions of 50×50 mm]
- The cementitious adhesives are prepared according to the method described in **EN 1308**
- Pile caps and tensile testing machine capable of applying a force of (250 ± 50) N per second, without causing any deformation

Procedure

A first thin layer of adhesive is applied to the surface of the concrete slab using the smooth edge of the notched trowel. Then, a second layer is applied, which is combed using the 6×6 mm notched trowel, holding the trowel at an angle of 60° . The beads of adhesive will be parallel to the edges of the concrete slab.



Equipment for testing tensile adhesion strength

After **5 min.** 10 type **VI** tiles are placed on the adhesive and subjected to a load of (20 ± 0.05) N for 30 s.

The following tests are performed on each slab with 10 tiles:

■ *Initial bonding*

- 1- Keep the slab for **27 days** under laboratory conditions [(23 ± 2) °C, (50 ± 5) % relative humidity and air velocity of less than 0.2 m/s]
- 2- Adhere the pile caps using a reaction resin adhesive
- 3- Keep the slab for another **24 hours** under laboratory conditions
- 4- Measure the tensile adhesion strength according to the force exerted in Newtons (N) at the time of breakage

In fast-setting cementitious adhesives (**F**) the tensile adhesion strength is also measured **24 hours** after placing the tiles.

■ *Bonding after immersion in water*

- 1- Keep the slab for **7 days** under laboratory conditions
- 2- Submerge the slab in water at (23 ± 2) °C and keep it there for **20 days**
- 3- Remove the slab and attach the pile caps. After 7 hours, submerge the slab again for another 24 hours
- 4- Measure the tensile adhesion strength immediately after removing the slab from the water

■ *Bonding after ageing with heat*

- 1- Keep the slab under laboratory conditions for **14 days**
- 2- Keep the slab in a kiln with air flow at (70 ± 2) °C for another **14 days**
- 3- Remove the slab from the kiln and attach the pile caps. Keep the slab under laboratory conditions for another 24 hours
- 4- Measurement of tensile adhesion strength

■ *Bonding after frost-thaw cycles*

In this case, double adhesive is applied when installing the tiles, spreading a thin layer of adhesive on the rear of the tile before adhering it to the combed adhesive on the surface of the concrete slab. The purpose is to achieve 100% wetting and, thus, no cavities that would allow water to be stored.

- 1- Keep the slab for **7 days** under laboratory conditions
- 2- Submerge the slab in water at room temperature and keep it there for **21 days**
- 3- Subject the slab to **25 frost-thaw cycles**, under the following conditions:
 - 3.1- After removing the slab from the water, cool it down to (-15 ± 3) °C, in no more than 2.5 hours.
 - 3.2- Keep the slab at (-15 ± 3) °C for $2 \text{ h} \pm 20 \text{ min.}$

3.3- Submerge the slab in water at (20 ± 3) °C again and keep it there for 2 h ± 20 min. at (15 ± 3) °C.

4- Immediately after completing the 25 cycles, the tensile adhesion strength is measured.

Expression of results

In every case, the tensile adhesion strength is obtained by dividing the tensile force of the device by the surface area of the tile ($2,500 \text{ mm}^2$), which gives the result in Newtons per square millimetre (N/mm^2), with an accuracy of one tenth of a unit (0.1 N/mm^2).

Of the ten tensile forces measured, any whose values diverge by $\pm 20\%$ from the arithmetic mean are discarded. If five or more values remain, the mean is calculated and used as the final result of the test; if fewer than five values remain, the test must be repeated.

The new 2007 version includes the following novelties:

- ▶ The kiln must be capable of controlling the temperature with a margin of 3 °C
- ▶ The measurement of the adhesion of fast-setting cementitious adhesives must be carried out **after 6 hours** (instead of 24 hours)
- ▶ The removal of parts after the frost-thaw cycles must be carried out like in ISO 13007-2
- ▶ The breakage type specification must be the most frequent type

EN 12002

DETERMINATION OF TRANSVERSE DEFORMATION FOR CEMENTITIOUS ADHESIVES AND GROUTS

January 2003 version. Replaces EN 12002:1997

Subject matter and scope

Measurement of the transverse deformation of cement-based adhesives and grouting materials used in the installation of ceramic tiles or other modular rigid materials that are compatible with these materials.

It does not include materials with elastic properties, such as dispersion resin adhesives and certain reaction resins.

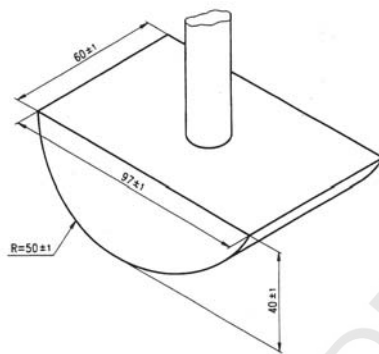
Test conditions and devices

As described in **EN 1308**.

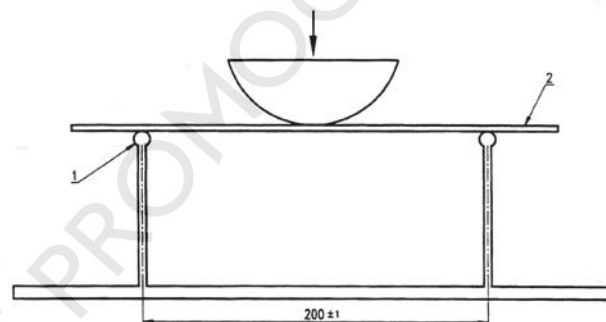
Material

- Polyethylene film substrate with thickness of 0.2 mm

- Plastic container, with hermetic seal, with internal capacity of (26 ± 5) litres; for example, a container with dimensions $600 \times 400 \times 110$ mm
- Mounting for extending the polyethylene film
- Test head, with the geometry and dimensions of the figure
- Test device made up of two 10 mm metal cylinders, with a separation of 200 mm and a minimum length of 60 mm
- **Template A:** A rigid rectangular frame, with smooth, non-absorbent surface, with inner dimensions of (280 ± 1) mm x (45 ± 1) mm and thickness of (5 ± 0.1) mm; for example made of metal or polytetrafluoroethylene (PTFE)
- **Template B:** A rigid mould, with a smooth, non-absorbent surface, capable of producing a test sample with dimensions (300 ± 1) mm x (45 ± 1) mm x (3 ± 0.05) mm
- Press for coupling the head, with a travel speed of 2 mm/min.
- Vibrating table with dimensions (280 ± 1) mm x (45 ± 1) mm and (5 ± 0.1) mm for compacting samples, in accordance with standard EN 459-2



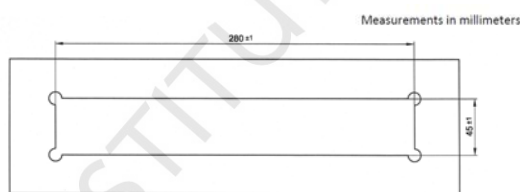
Test head



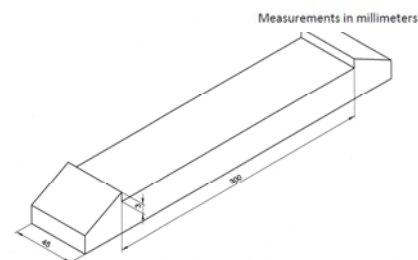
1 Cylindrical substrate with a diameter of (10 ± 0.1) mm and minimum length of 60 mm.

2 Thickness of the adhesive or grouting material (3 ± 0.3) mm.

Test device



Template A



Template B

Mixing the adhesive or grouting material

Under the mixing conditions specified by the manufacturer, 2 kg of mixture are prepared in a mixer such as that described in section 4.4 of European standard EN 196-1, at a rotation speed of $(140 \pm 5) \text{ min}^{-1}$ and a planetary movement speed of $(62 \pm 5) \text{ min}^{-1}$. The mixing operation must follow the procedure below:

- the liquid is poured into the mixer
- the powder slowly disperses in the liquid
- it is mixed for 30 s
- it is allowed to rest for 1 min after cleaning off the blade and container
- it is mixed again for 1 min

According to the manufacturer's recommendations, it is allowed to rest for another period of time and then mixed again for 15 s.

Procedure

Once the polyethylene film has been attached to the rigid mounting, guaranteeing a smooth surface with no creases or bubbles, a sufficient amount of material is applied to template A and it is subjected to compacting by 70 knocks on the vibrating table.

After removing template A, the sample is placed in template B and subjected to a force of (100 ± 0.1) N on a surface of (290×45) mm, in order to ensure that the thickness of 3 mm is reached after 1 hour.



Equipment for testing transverse deformation
SOURCE: BASF

After 48 h, template B and the polyethylene film are removed. The samples are stored in the hermetic plastic container for **12 days** at (23 ± 2) °C and outside the container for another 14 days under laboratory conditions [$T = (23 \pm 2)$ °C, $RH = (50 \pm 5)\%$ and maximum air velocity of 0.2 m/s].

After conditioning, the mean thickness of the samples is measured using a gauge with an accuracy of 0.01 mm, at the centre and 50 mm from each edge.

If the samples are deformed before the test, the deformation must be measured relative to the horizontal plane, since the goal is to measure the *net transverse deformation* caused by the force exerted by the head, with a movement of 2 mm/min, until breakage occurs.

If no cracks appear, the maximum deformation and the corresponding force are recorded.

The test is performed on at least three samples.

Expression of results

The transverse deformation is determined with an accuracy of 0.1 mm, calculating the mean of the deformation values obtained in the tests.

According to the transverse deformation value obtained, the **cementitious adhesives** are classified as two types:

- S1: Deformable adhesives with a transverse deformation of **no less than 2.5 mm and less than 5 mm.**
- S2: Deformable adhesives with a transverse deformation of **no less than 5 mm.**

Observations

For now, this is the only test method available in the European standards for measuring the deformability of cementitious adhesives.

Although the title extends the measurement of deformability to cover grouting materials, it appears that in a version of EN 12002 published after the January 2003 version, it will only be applicable to bonding materials, since no consensus was reached among the manufacturers.

However, in cementitious adhesives, there are still certain omissions regarding how to assess deformability, especially considering that deformability should result in *real shear adhesion strength* of the bonding material.

Research and tests are currently under way in accordance with the following methods:

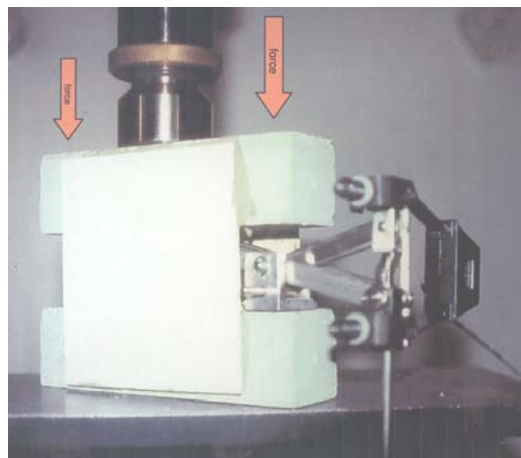
- Measurement of shear adhesion strength and resistance to deformation according to **DIN 18153/3**, stated in the UEAtc Directive
- Measurement of shear adhesion strength and resistance to deformation, according to the method stipulated in **EN 1324**, with certain variations (for example, replacing the buttons of bonding material with a continuous film of cementitious adhesive applied using a notched trowel)
- Correlating the tensile adhesion strength after heat treatment at 70 °C measured according to EN 1348.



$$\operatorname{tg} \alpha = \frac{d}{e} = \frac{2(1 + \sigma)}{E} \cdot \frac{F}{S}$$

d = deformation
 e = thickness of the layer exposed to shear
 σ = Poisson's ratio of the material
 E = Young's modulus of the material
 F = shear force applied
 S = surface involved in the shear

DEFORMATION BY SHEAR



The new version of the standard is pending final approval (July 2008). It is expected to introduce the following modifications:

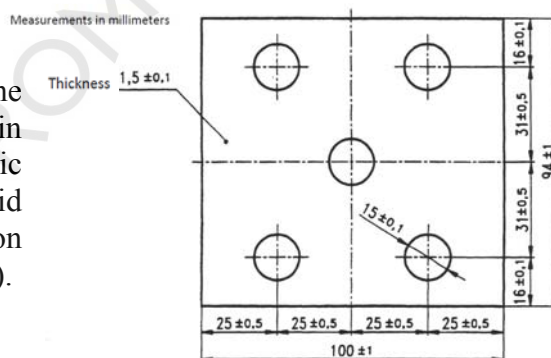
- ▶ Removal of the reference to cementitious grouting materials from the title
- ▶ Section 9.2 will specify that a release agent or polyethylene film can be used
- ▶ Codes **S1** and **S2** are deleted, since they have already been included in EN 12004.

EN 12003

DETERMINATION OF SHEAR ADHESION STRENGTH OF REACTION RESIN ADHESIVES

Subject matter and scope

Test method for determining the shear adhesion strength of reaction resin adhesives used in the installation of ceramic tiles and other compatible modular rigid materials, in every type of application (floorings, coverings, indoors and outdoors).



Test conditions

As described in **EN 1308**.

Material

- Ceramic tiles: Type **V2** [BI_a UGL, with water absorption capacity $E \leq 0.2\%$, with flat bonding surface and dimensions of 100x100 mm]
- Template made of non-absorbent material (polytetrafluoroethylene, PTFE) for the application of adhesive by points, with the geometry of the figure
- Spacing rods with a diameter of (0.8 ± 0.1) mm, for controlling the thickness of the adhesive applied
- 97x97 mm slab capable of exerting a force of (70 ± 0.15) N
- Machine for applying gradual force at constant speed, for coupling with the shearing device, either by horizontal pulling or vertical compression

Procedure

After preparing the adhesive according to the instructions of the manufacturer and applying it to the rear of a V2 tile using the template, the spacers are placed and a second tile is attached to the rear of the first tile. For 3 min. a load is applied to the assembly using the 70 N slab.



Ten test specimens are prepared and kept under laboratory conditions for **7 days**.

After this time, the shear adhesion strength tests are performed:

- ▶ *Initial bonding force*: the force at the time of breakage is measured, after exerting a gradual force which causes the shearing equipment to move by 5 mm/min.
- ▶ *Bonding strength after immersion in water*: The test is carried out after keeping the ten test specimens submerged in water at (23 ± 2) °C for **21 days**
- ▶ *Bonding strength after thermal shocks*: After subjecting the ten test specimens to **four cycles** of immersion in water at (23 ± 2) °C for **30 min** and in water at 100 °C for another **30 min**, they are allowed to cool for one last time to 23 °C during 30 min. before performing the test.

Expression of results

In every case, the shear adhesion strength is obtained by dividing the force measured at the time of breakage by the surface area of the tile in contact with the adhesive (1,660 mm²).

The mean of the ten values obtained is calculated. Any values that deviate by $\pm 20\%$ from the mean value are discarded. If five or more values remain, the mean is calculated again, producing the final result. If fewer than five values remain, the test must be repeated.

EN 12808-1

PART 1: DETERMINATION OF THE CHEMICAL RESISTANCE OF REACTION RESIN MORTARS

Subject matter and scope

Test method designed for determining the chemical resistance of adhesives and grouting materials used in the installation of ceramic tiles on walls and floors, outdoors and indoors.

Chemical etching is measured according to variations in the weight and diameter of the test specimens, as well as by variations in their resistance to compression.

Test conditions

As described in **EN 1308**.

Material

- Cylindrical mould with a diameter of (25±1) mm and a height of (25±1) mm, made of an inert, non-stick material for obtaining the test specimens
- Different types of containers for the test, made of materials that cannot be etched by the chemical products to be tested
- Erlenmeyer flasks with reflux condenser lids for volatile products
- Variable-speed compression machine

Test specimens

The number of test specimens depends on the number of chemical products to be tested, the temperatures at which the test must be performed and the frequency of the test intervals. The standard defines a formula for calculating the number of test specimens to be prepared.

Procedure

After modelling the test specimens, they are kept under laboratory conditions for **7 days**. After conditioning, the diameter of all the test specimens is measured with an accuracy of 0.03 mm and they are weighed on scales with an accuracy of up to 0.001 g. Then the colour and surface appearance of the test specimens and the chemical products to be used are recorded.

The test specimens are placed in containers, resting on their curved surface, without touching one another, and the chemical product is added until they are submerged [(100±5) ml per test specimen]. After remaining submerged for **28 days** they are washed in cold water and left to dry. The diameter and weight are measured again on the dry test specimens. Their resistance to compression is measured:

- ▶ Immediately after the conditioning period
- ▶ After the period of exposure to each temperature and each chemical product
- ▶ After ageing in the air during the total test period, at each temperature

The result of the compression test is the maximum load at the time of breakage of the test specimen.

Expression of results

- ▶ *Weight variation*: For every period of exposure to chemical etching, the weight loss percentage is calculated, with an accuracy of 0.01%, according to the formula:

$$\Delta W = \frac{W - C}{C} \times 100 \quad \text{in which:} \quad \left\{ \begin{array}{l} \Delta W: \text{ is the weight variation as a \%} \\ W: \text{ is the weight after immersion (g)} \\ C: \text{ is the weight after initial conditioning (g)} \end{array} \right.$$

The result is expressed as the mean value of all the tests performed (at least three test specimens). The result is normally given as positive (+) when the weight increased and as negative (-) if the weight dropped.

- ▶ *Diameter variation*: As in the case of the weight, the diameter variation is calculated as a percentage, with an accuracy of 0.01%:

$$\Delta D = \frac{D_2 - D_1}{D_1} \times 100 \quad \text{in which:}$$

D :	is the diameter variation as a %
D₁ :	diameter of the test specimen after initial conditioning (mm)
D₂ :	diameter of the test specimen after chemical etching (mm)

The result is the arithmetic mean of the measured values. The result is positive (+) when the diameter increased and negative (-) if it dropped.

- ▶ *Variation of resistance to compression*: Calculated as a percentage variation of the load per surface unit. The surface is calculated according to the measured diameter (surface area of the circle or base of the cylinder)

$$\Delta P = \frac{P_2 - P_1}{P_1} \times 100 \quad \text{in which:}$$

P :	variation of resistance to compression as a %
P₁ :	resistance to compression following the conditioning period, in megapascals (MPa)
P₂ :	resistance to compression following exposure to chemical etching (in MPa)

A positive result (+) indicates an increase in the resistance to compression, while a negative result (-) indicates a reduction.

- ▶ *Appearance*: Changes to surface appearance after chemical etching must be recorded, including loss of shine, change in colour, appearance of cracks, scaling, pores, softening, etc.