

ICS 91.140.70

English Version

Sanitary appliances - Methacrylic dispersions of high filler content

Appareils sanitaires - Dispersions méthacryliques à taux de charge élevée

Sanitärausstattungsgegenstände - Hochgefüllte Methacryl Dispersionen

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 15334:2007) has been prepared by Technical Committee CEN/TC 163 "Sanitary appliances", the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2007, and conflicting national standards shall be withdrawn at the latest by August 2007.

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1 Scope

This European Standard specifies test methods for measuring characteristics of pure methacrylic dispersions and sanitary appliances produced from dispersions by polymerisation.

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.

EN 59, *Glass reinforced plastics - Measurement of hardness by means of a Barcol impressor*

EN 20105-A02, *Textiles - Tests for colour fastness - Part A02: Grey scale for assessing change in colour (ISO 105-A02:1993)*

EN ISO 62:1999, *Plastics - Determination of water absorption (ISO 62: 1999)*

EN ISO 178, *Plastics - Determination of flexural properties (ISO 178: 2001)*

EN ISO 179-1, *Plastics - Determination of Charpy impact properties - Part 1: Non-instrumented impact test (ISO 179-1: 2000)*

EN ISO 306:2004, *Plastics - Thermoplastic materials - Determination of Vicat softening temperature (VST) (ISO 306: 2004)*

EN ISO 3451-1, *Plastics - Determination of ash - Part 1: General methods (ISO 3451-1: 1997)*

EN ISO 4892-2, *Plastics - Methods of exposure to laboratory light sources - Part 2: Xenon-arc lamps (ISO 4892-2: 2006)*

ISO 4586-2, *High-pressure decorative laminates - Sheets made from thermosetting resins - Part 2: Determination of properties*

ISO 9352, *Plastics - Determination of resistance to wear by abrasive wheels*

ISO 11359-2, *Plastics - Thermomechanical analysis (TMA) - Part 2 - Determination of coefficient of linear thermal expansion and glass transition temperature*

3 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

3.1

high filler content

filler percentage of inorganic materials greater than 50 % w/w

3.2

liquid methacrylic resin

solution of PMMA polymers and copolymers dissolved in MMA.

NOTE Other chemicals may be added to this resin (e.g. thixotropes, colorants, mould release agents), to further develop resin properties

3.3**methacrylic dispersion**

dispersion produced by mixing inorganic filler(s) with liquid pure methacrylic resin

3.4**methacrylic dispersion types**

existing methacrylic dispersions used as materials for sanitary appliances

NOTE Methacrylic dispersion types are given in Table 1.

Table 1 - Methacrylic dispersion types

| Filler type | % of filler | Application (examples) | Comments |
|-----------------------------|-------------|---|--|
| ATH (Aluminium tri-hydrate) | Around 60 | Vanity basins Shower trays Work top surfaces | Soft repairable surface |
| Silica | Around 60 | Kitchen sinks Baths Shower trays | |
| Quartz | Around 70 | Kitchen sinks, wash basins, shower trays, work top surfaces | The functional surface has an higher content of filler than the bulk |

Material is purchased either as a resin mixed with filler or as a resin with the filler added by the sanitary appliance manufacturer

4 Characteristics of dispersion

The methacrylic dispersion manufacturer shall provide the following information:

- resin type (methacrylate esters);
- sedimenting /non sedimenting;
- filler type;
- polymerisation process (UV, redox, thermal, other).

5 Test methods**5.1 Tests on dispersion****5.1.1 Ash content****5.1.1.1 General**

See EN ISO 3451-1.

Materials with filler made from aluminium trihydrate are exempt from this test.

5.1.1.2 Apparatus

Source of heat ensuring the combustion of organic materials.

Means of ensuring homogeneity of the dispersion to be tested (this is most important for dispersions with high levels of sedimentation).

Spatula for the sample.

5.1.1.3 Sample

2 g to 15 g of dispersion according to the dimensions of particles.

5.1.1.4 Procedure

Ensure the sample is representative of the material to be tested. Stir or roll a larger sample of product for a minimum of 1 h to ensure redispersion of the product.

Check that the crucible is clean and dry (heat the crucible to red hot to remove any combustible material). Allow to cool.

Weigh the crucible (A).

Give the product a final stir and then rapidly scoop a spatula full of material into the crucible. Do not wait to drain the material from the spatula as consistency at this time is key to achieving reproducible results.

Place the crucible on a scale and reweigh (B).

If additional material is required, repeat the final step (final stir of the product). If too much material has been added, dispose of the contents and repeat from step (check crucible is clean and dry).

Heat the crucible to red hot and burn off the combustible material. Ensure that the material does not spit out of the crucible during heating. This process will take a minimum of 5 min.

Gently shake the crucible to make sure the remaining inorganic particles are free to move. If there are any agglomerates, repeat the procedure (heat the crucible to red hot).

Allow to cool.

Weigh the crucible (C).

Calculate the ash content (A_C), as percentage, as follows:

$$A_C = \frac{C - A}{B - A} \times 100 \quad (1)$$

5.1.2 Agglomeration

5.1.2.1 General

This test is for solid coloured material only and applies to materials with a particle size lower than 50 μm .

5.1.2.2 Apparatus

Steel filter, mesh size $(180 \pm 20) \mu\text{m}$.

5.1.2.3 Sample

100 g of dispersion.

5.1.2.4 Procedure

Dilute 100 g of the material under test with an equal volume of clean methyl methacrylate monomer. Pour this through the filter and rinse with more clean monomer.

Check as to if agglomerates are left on the filter.

5.1.3 Contamination

5.1.3.1 Apparatus

Light box with an opal cover.

Clear polyester film (30 cm wide roll).

Laboratory balance.

Two glass plates (300 mm x 300 mm x 4 mm).

5.1.3.2 Sample

100 g of dispersion.

5.1.3.3 Procedure

Place one glass plate on the balance. Cut a piece of polyester film approximately 0,5 m long and place it on the glass. Tare the balance and weigh 50 g of the sample onto the polyester film and cover with another similar sized piece of polyester film. Place the glass plate over the opal panel on the light box. Place the second glass on top of the "cell" allowing the sample to spread to form a circle approximately 0,25 m in diameter.

Count the number of contaminants within the viewing area.

Record the number of particles in the following size ranges:

- $0,01 \text{ mm}^2$ to $0,02 \text{ mm}^2$

- $0,02 \text{ mm}^2$ to $0,05 \text{ mm}^2$

- $0,05 \text{ mm}^2$ to $0,10 \text{ mm}^2$

- greater than $0,10 \text{ mm}^2$

5.1.4 Density

5.1.4.1 Apparatus

Conical flask (50 ml nominal capacity) complete with ground glass disc.

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Thermometer (Range -10°C to + 50°C).

Balance capable of determining weight up to 200 g to two decimal figures.

Acetone.

Lidded labelled container.

5.1.4.2 Sample

Dispersion, enough to fill the flask. Avoid any sedimentation before pouring into the flask.

5.1.4.3 Procedure

Ensure that the conical flask and glass disc are clean and dry. Record the combined weight of the flask and disc (Weight = A grams).

Fill the flask with water at (23 ± 2) °C and place the glass disc on top of the flask. The correct volume of water has been dispensed into the flask when no air bubbles can be seen entrained below the glass disc. If the flask has been overfilled, excess water on the underside of the disc and the outside of the flask must be removed before reweighing (Weight = B grams). Discard the water and thoroughly dry the flask and disc before using with the dispersions under test.

Assuming a density of $1,0 \text{ g/cm}^{-3}$ for water at 23 °C, the volume of the flask at 23 °C (V_F) is calculated as:

$$V_F = \frac{B - A}{1} \text{ cm}^3 \quad (2)$$

Ensure that the dispersion to be tested is fully re-dispersed at (23 ± 1) °C. Pour the dispersion into the dry flask until full and place the glass disc on the top. As when determining the flask volume, ensure there are no air bubbles beneath the glass disc. Remove any excess dispersion on the underside of the disc or on the outside of the flask before weighing (Weight = C grams). Rinse the flask and disc repeatedly with acetone until clean. Dry the flask thoroughly before filling it with the next test sample.

Density of the dispersion at 23 °C (ρ_d) is determined as follows:

$$\rho_d = \frac{C - A}{V_F} \text{ g/m}^3 \quad (3)$$

The density should be quoted to two decimal figures.

5.1.5 Viscosity

5.1.5.1 General

This test is not applicable for sedimenting products.

5.1.5.2 Apparatus

Brookfield viscometer.

Viscometer spindles as defined on the product test specification sheet.

Thermometer.

Stopwatch.

5.1.5.3 Sample

Dispersion under consideration.

5.1.5.4 Procedure

Ensure that the sample has been rolling for at least 4 h at 30 to 40 rev/min and is at (23 ± 2) °C to ensure product dispersion and correct temperature. Make sure the viscometer is level and select the appropriate speed and spindle for the material to be tested.

Place the sample under the spindle and lower the viscometer until the spindle immersion mark is level with the surface of the sample and in the centre of the tin.

Wait the specified standing time, then press down on the clutch lever and start the machine. After approximately 10 s, release the clutch lever and start the stopwatch.

Wait the specified test time and press and hold down the clutch lever again to lock the reading. When the pointer is visible in the window stop the machine and take the reading from the dial before releasing the clutch lever.

Calculate the viscosity by multiplying the scale reading by the spindle factor (see Table 2). Note that this gives the viscosity in centipoise (1 centipoise = 0,01 Ps).

Repeat the test at additional speeds as defined on the product test specification sheet.

If required, calculate the thixotropy index (TI) as follows:

$$TI = \frac{v_{30}}{v_{12}} \quad (4)$$

where

v_{30} is the viscosity at 30 rpm

v_{12} is the viscosity at 12 rpm

(4)

Table 2- Spindle factors for Brookfield model LV viscosimeter

| Speed (min ⁻¹) | Spindle 1 | Spindle 2 | Spindle 3 | Spindle 4 |
|----------------------------|-----------|-----------|-----------|-----------|
| 6 | 10 | 50 | 200 | 1 000 |
| 12 | 5 | 25 | 100 | 500 |
| 30 | 2 | 10 | 40 | 200 |
| 60 | 1 | 5 | 20 | 100 |

5.1.6 Shrinkage during curing

5.1.6.1 General

The casting resins should be examined for shrinkage from liquid to solid using a mould of defined dimensions.

5.1.6.2 Apparatus

Equipment for preparing the resin mixture (see product specific statements in the mixing instructions).

Casting mould made from steel.

Interior dimensions of 250 mm x 100 mm x 25 mm.

Preferably coated with PTFE.

Open to the top.

Thermometer (accurate to +/- 1 °C).

Watch with a second hand.

Paintbrush.

5.1.6.3 Test conditions

Ambient temperature (23 ± 2) °C.

Specimen and mould temperature (23 ± 2) °C.

Conditioning time (see product specific statements in the mixing instructions).

5.1.6.4 Procedure

Preparing the mixture:

The mixture may only be prepared after the minimum maturing period has elapsed (time after the end of production) and after the components have been conditioned to 23 °C.

The mixture should be stirred, e.g. by a wooden spatula, for 2 min to 3 min.

A ventilation phase should be allowed for 20 min to 30 min, during which time the air adhering to the fillers and pigments is vented.

After ventilation, stir hardener, e.g. peroxide, into the slurry to start the polymerization reaction.

Fill the cast able mixture, slowly to avoid air bubbles, to the upper edge of the above mentioned casting mould. Allow the system to shrink freely.

5.1.6.5 Evaluation

After cooling at least 4 h, the dimensional change is measured as the shrinkage (the polymerized specimen is smaller than the casting mould).

Shrinkage is the distance (gap) between the dimensions of the casting mould and the specimen.

$$S = \frac{d}{l_m} 100 \quad (5)$$

where

S is shrinkage, in percentage

d is the distance, in mm

l_m is the length of the casting mould, in mm

The result is documented in suitable form.

5.2 Tests on finished part¹

5.2.1 General

Test specimens shall be cut from the sanitary appliance under consideration and comply with the requirements of ISO standards describing each test method.

For any sedimented material, all samples shall be taken so that the show face is used.

For very hard materials, it could be difficult to get a very smooth surface.

5.2.2 Water absorption

Water absorption test is carried out according to method 1 of EN ISO 62:1999.

5.2.3 Impact strength

Impact strength test is carried out by impacting the show face and according to EN ISO 179-1 fn (unnotched).

5.2.4 Flexural modulus and flexural strength

Flexural modulus and flexural strength are determined according to the method described in EN ISO 178.

5.2.5 Abrasion resistance

Abrasion resistance is determined according to the method described in ISO 9352.

5.2.6 Scratch resistance

Scratch resistance is determined according to the method described in ISO 4586-2.

5.2.7 Barcol hardness

Barcol hardness is determined according to the method described in EN 59.

¹ Where product standards exist on appliances made of cast filled acrylics, the tests described in those standards apply for testing finished products.

5.2.8 Vicat softening point

Vicat softening point is determined according to the method B 50 of EN ISO 306:2004, with the temperature raised at a rate of (50 ± 5) °C/h.

5.2.9 Resistance to chemical agents**5.2.9.1 Reagents**

A list of reagents is given in Table 3. Each aqueous solution shall be prepared immediately before application. The reagents shall be made up and applied at (23 ± 2) °C.

Table 3- Reagents

| Family | Product | Concentration |
|----------------|----------------|------------------------|
| Acids | Acetic acid | 10 % V/V |
| Alkalis | NaOH | 10 % m/m |
| Alcohol | Ethanol | 70 % V/V |
| Bleaches | NaOCl | 5 % available chlorine |
| Staining agent | Methylene Blue | 1 % m/m |

5.2.9.2 Apparatus**5.2.9.2.1 Borosilicate watch glasses**

40 mm nominal diameter.

5.2.9.2.2 Pipettes**5.2.9.2.3 Cleaning device**

The cleaning device is illustrated in Figure 1. It is comprised of a synthetic flexible open cell foam disc 75 mm in diameter and 15 mm thick. Use any rotating device applying a mass of $(1\ 000 \pm 50)$ g that fits loosely with the device. The lateral cleaning force shall only be that exerted by the mass of the cleaning device; this can be effected by a floating action between the drive shaft and the disc.

5.2.9.2.4 Test specimens

Specimens shall measure (100 ± 5) mm x (100 ± 5) mm.

5.2.9.3 Procedure

Use a separate test specimen for each reagent test. Clean the test area thoroughly with hot soapy water, rinse and then dry with a clean soft cloth.

Place a drop of the test solution on each test specimen. Cover the drop with a watch glass concave downward. The drop size shall be determined once completely covered by the watch glass. Allow to act for $(16,0 \pm 0,25)$ h, at (23 ± 5) °C, with the test areas protected from the affects of sunlight.

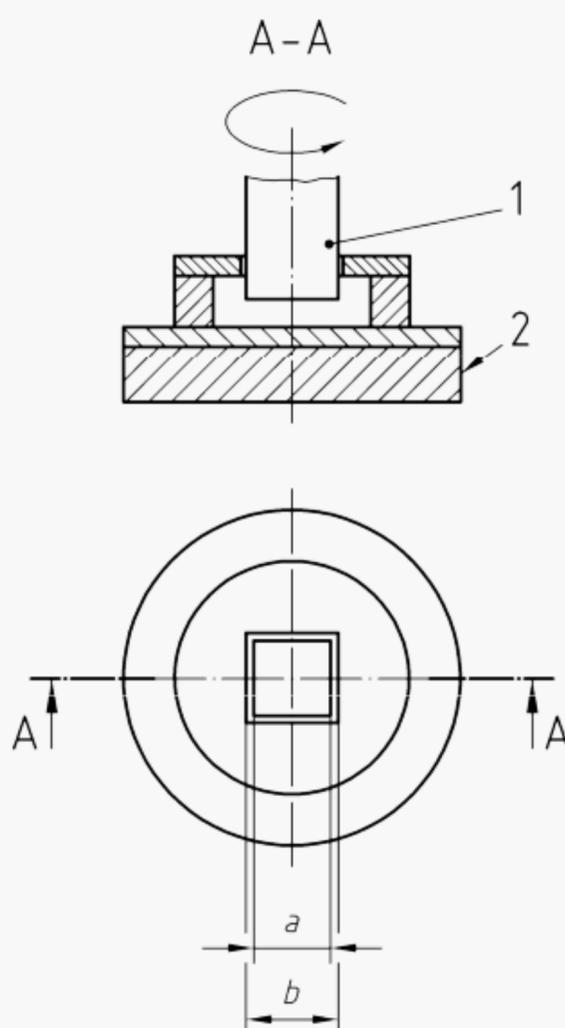
Thoroughly rinse the test specimen with demineralised water and by visual examination, check for adverse changes in appearance. If deterioration has occurred, dip the foam disc of the cleaning device into demineralised water and place it on the surface to be cleaned. Rotate the device at 60 rev/min.

Clean for 30 rev.

Rinse with demineralised water and visually examine the test area. If deterioration persists repeat the cleaning process using demineralised water with an abrasive agent added. This abrasive agent is defined as follows: alumina used for surface polishing with particle size comprised between 0,1 μm and 2 μm and centered on 0,5 μm .

5.2.9.4 Results

Note whether or not the reagent causes a stain or deterioration and whether or not such staining or deterioration can be removed with either water or water containing an abrasive agent. If the stain cannot be removed by water with abrasive agent, record the reagent as permanently stained.



Key

- 1 square axle $a = b - 1 \text{ mm}$
- 2 foam

Figure 1 - Detail of cleaning apparatus

5.2.10 UV resistance

When tested on apparatus complying with requirements of xenon arc lamp for 250 h by the method specified in EN ISO 4892-2, the colour change noted on the show face of the specimen used shall be recorded in terms of grey scale to assess the colour change as specified in EN 20105-A02.

5.2.11 Thermal expansion

Thermal expansion is determined according to the method described in ISO 11359-2.

Bibliography

- [1] EN 13310, *Kitchen sinks - Functional requirements and test methods*
- [2] EN 14516, *Baths for domestic purposes*
- [3] EN 14527, *Shower trays for domestic purposes*