

Products and systems for the protection and repair of concrete structures — Test methods — Determination of the coefficient of thermal expansion

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ICS 91.080.40

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National foreword

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Comité Européen de Normalisation
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Foreword

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NOTE This European Standard should be read together with EN 1504-1.

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1 Scope

This European Standard specifies methods for the determination of the coefficient of thermal expansion of hardened structural bonding agents. The first method provides a continuous measurement of linear thermal expansion using thermomechanical analysis techniques. This method may also be used for surface protection systems. The alternative method uses prisms of 40 mm × 40 mm × 160 mm. This method may also be used for repair mortar. Both methods are suitable for bonding agents formulated with fillers of up to 4 mm.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

prEN 12190, *Products and systems for the protection and repair of concrete structures — Test methods — Determination of compressive strength.*

3 Thermomechanical analysis

3.1 Principle

The coefficient of thermal expansion of resinous materials is to be measured at an age of 7 days (storage at (21 ± 2) °C and (60 ± 10) % relative humidity).

Once hardened the bonding agent is subjected to a constant heating rate. This method uses a thermomechanical analyser to determine the changes in length, which is electronically recorded as a function of temperature. The coefficient of linear thermal expansion can be calculated from the recorded data.

3.2 Apparatus

3.2.1 Thermomechanical analyser, or similar device, consisting of:

- a) specimen holder and probe, (constructed from low-expansion materials such as fused quartz) that transmits changes in the length of the specimen to the transducer. The shape and size of the probe shall be such that the load applied shall not cause indentation of the specimen, during testing;
- b) transducer, for sensing movement of the probe resulting from changes in length of the specimen and for translating these movements into an electrical signal suitable for input to a recording system;
- c) temperature sensing element, for measuring the temperature of the test specimen;

d) a recording system to record the changes in specimen length as a function of specimen temperature. The combination of transducer and recorder shall have sufficient sensitivity to produce a minimum of 1 mm of chart deflection per 100 nm of probe movement with provision for less sensitive ranges, where needed.

3.2.2 Furnace, for uniformly heating the specimen (preferably at 2 °C/min), at a predetermined rate over the testing temperature range with provision for cooling the specimen when sub-ambient temperature measurements are to be made.

3.2.3 Means of purging the specimen environment with a dry inert gas such as nitrogen or helium (for temperatures > 100 °C).

3.2.4 Callipers, capable of measuring linear dimensions to an accuracy of not less than 25 µm.

3.3 Test procedure

3.3.1 Sampling material

The bonding agent to be tested shall be taken from one production batch. The specimen may be cut from a prism 40 mm × 40 mm × 160 mm, as used for other tests.

3.3.2 Test specimens

Cylindrical or square shaped specimens shall be between 25 mm and 50 mm in length and have ends flat and parallel to within ± 25 µm. Lateral dimensions shall not exceed 10 mm. Other lengths may be used provided that they are noted in the report.

The specimens shall be measured in the received condition. If some heat or mechanical treatment is applied to the specimen prior to test, this treatment should be noted in the report.

A minimum of three specimens shall be tested.

3.3.3 Storage of samples

Before starting the test procedure the materials shall be stored for 7 days at (21 ± 2) °C and (60 ± 10) % relative humidity.

3.3.4 Procedure

a) Calibration

Temperature calibration can be achieved by observing the penetration by a $(5 \pm 0,5)$ g loaded probe when a crystalline material is heated through its melting point at the same rate as the expansion specimen. The following high purity (> 99 %) materials may be used.

Table 1

Reference material	Melting point °C
Indium	156,6
Tin	232,0
Lead	327,5
Zinc	419,6
Aluminium	660,4

The length change measuring and recording system can be calibrated by measuring the linear expansion of a material, having known expansion, when heated at the same rate as the test specimens. The observed expansion shall be corrected for the difference in expansion between, the specimen holder and probe obtained from a preliminary trial in which either no sample or a specimen of the material of construction of the probe is used. As a working standard high purity alumina (Al_2O_3), platinum, quartz or vacrominium may be used.

b) Procedure

Measure the initial specimen length in the direction of the expansion test to an accuracy of $\pm 25 \mu\text{m}$ at room temperature.

Position the specimen in the holder under the probe, with the temperature sensor in contact with the specimen.

Place the specimen holder in the furnace. If measurements at sub-ambient temperature are to be made, cool the specimen to at least 20°C below the lowest temperature of interest. The refrigerant used for cooling shall not come into direct contact with the specimen.

Select an appropriate sensitivity setting on the recorder.

Heat the specimen at a constant heating rate of $(2 \pm 1)^\circ\text{C}/\text{min}$ over the desired temperature range. Other heating rates may be used provided that they are noted in the report.

3.4 Calculation

3.4.1 Calculate the mean coefficient of linear thermal expansion over the temperature range as specified in the instructions supplied with the equipment, as follows, for example:

$$\alpha_m = \frac{\Delta L_{\text{sp}} \times k}{L \times \Delta T}$$

$$k = \frac{\alpha_{\text{ref}} \times L_{\text{ref}} \times \Delta T_{\text{ref}}}{\Delta L_{\text{ref}}}$$

where:

- α_m is the mean coefficient of linear thermal expansion, in $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$;
- α_{ref} is the mean coefficient of linear thermal expansion, for reference material, in $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$;
- k is the calibration coefficient;
- L is the specimen length at room temperature, in metres (m);
- ΔL_{ref} is the change of reference material length due to heating, in micrometres (μm);
- L_{ref} is the reference material length at room temperature, in metres (m);
- ΔL_{sp} is the change of specimen length, in micrometres (μm);

ΔT_{ref} is the temperature difference over which the change in reference material length is measured, in $^\circ\text{C}$;

ΔT is the temperature difference over which the change in specimen length is measured, in $^\circ\text{C}$.

3.4.2 Select ΔT from a smooth portion of the thermal curves in the desired temperature range; then obtain ΔL as depicted in Figure 1. The α_m shall not be calculated from a temperature range in which a transition point is noted.

3.4.3 Calculate the average value of α_m from the tests on three specimens.

3.5 Test report

The following information shall be included in the test report:

- a) identification of the material, including the name of the manufacturer and information on batch number and chemical composition when known;
- b) method of test specimen preparation;
- c) dimensions of test specimen;
- d) description of the thermomechanical analysis apparatus;
- e) purge gas and cooling medium, if used;
- f) temperature range in which the coefficient of linear thermal expansion has been determined;
- g) average value of the coefficient of linear thermal expansion in $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$ as determined from the three specimens;
- h) expansion curves obtained;
- i) reference to this European Standard.

4 Alternative method

4.1 Principle

The coefficient of thermal expansion of resinous materials shall be measured after being stored for 7 days (storage at $(21 \pm 2)^\circ\text{C}$ and $(60 \pm 10)\%$ relative humidity).

Once hardened the bonding agent is subjected to a number of temperature cycles while movements of the ends and changes in temperature of the mortar are monitored. The measurement is done with a dial gauge via two pegs, fixed to the end of the longer axis of the specimen. In the test equipment the sample shall be free to move in this axis.

The method may also be used to determine the coefficient of thermal expansion of repair mortars.

4.2 Apparatus

a) Mould

Steel mould containing three impressions of $(40 \pm 0,1) \text{ mm} \times (40 \pm 0,1) \text{ mm} \times (160 \pm 10,4) \text{ mm}$. (See Figure 2).

b) Tamper

The tamper is shown in Figure 3. It shall have a mass of $(500 \pm 20) \text{ g}$. It shall be made of wood with sheet protection, of light metal or of light plastic.

c) Measuring pegs

Measuring pegs shall be designed so that they can be glued to the end faces of the specimens. For this purpose, they shall have a flat base plate with a thickness of about 1,5 mm and a diameter of not less than 15 mm. The measuring pegs shall be $6,5^{+0,5}_0 \text{ mm}$ in length and of form A or B (see Figure 4). The measuring pegs shall be made of stainless steel, the heads shall be polished and shall exhibit no undulations.

d) Measuring equipment

The measuring equipment (see Figure 5) shall consist of a base, on which a column is arranged in a fixed vertical position. A vertically adjustable holder secured against rotation for mounting the dial gauge with 0,001 mm scale intervals shall then be attached above a fixed measurement axis.

NOTE Alternative measuring equipment as shown in Figures 6 and 7 may also be used.

The measurement cups shall be made of stainless steel. The shape of the seatings for the measuring pegs shall be conical, and they shall be hardened and polished.

The vertically positioned column shall be in steel with a low coefficient of thermal expansion. The measuring equipment shown in Figure 6 is suitable for testing specimens with a compressive strength of about $0,5 \text{ N/mm}^2$ or larger.

e) Thermocouples

Thermocouples shall be connected into a signal conditioning unit with automatic cold junction compensation, which gives a linear output proportional to temperature.

f) Environmental chamber

An environmental chamber large enough to contain the prisms. The chamber shall be capable of maintaining a temperature to within $\pm 1 \text{ }^\circ\text{C}$ over the range $-25 \text{ }^\circ\text{C}$ to $+60 \text{ }^\circ\text{C}$ at a relative humidity of not less than 50%. Alternatively, the specimens may be wrapped in a water vapour proof material.

g) Reference specimen

The reference specimen shall be a rod made of steel with a low coefficient of thermal expansion which can expand freely along its longitudinal axis. It shall be 176 mm in length and about 10 mm in diameter and encased in wood or other thermal insulating material; the ends of the rod shall be designed to match the measuring pegs with shape A or shape B depending on the measuring equipment.

4.3 Test procedure

4.3.1 Sampling material

The bonding agent to be tested shall be taken from one production batch.

NOTE Specimens made for compressive strength testing can be used for determining the coefficient of thermal expansion.

a) Samples made of fresh mortars

The preparation of the samples shall be carried out as described in prEN 12190. If highly reactive materials are used (exothermic reaction), the moulds shall be cooled.

b) Samples of hardened mortar

If it is not possible to prepare samples as described in 4.3.1a) then test pieces can be cut from preformed samples. Samples should be of size $40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}$.

4.3.2 Number of test specimens

A minimum of three specimens shall be tested.

4.3.3 Storage of samples

Before starting the test procedure, materials shall be stored for 7 days at $(21 \pm 2) \text{ }^\circ\text{C}$ and $(60 \pm 10) \%$ relative humidity.

4.3.4 Procedure

a) Conditioning of the test specimen.

Place the specimen into the chamber and locate the chamber thermocouple immediately adjacent to the specimen. Set the chamber to the initial temperature T_1 which shall be $(23 \pm 2) \text{ }^\circ\text{C}$.

Condition the specimen at T_1 , until the specimen and chamber temperatures correspond within $0,5 \text{ }^\circ\text{C}$, and are stable for at least 1 h.

Record the mean of the two thermocouple readings at $(T_1 \pm 1) \text{ }^\circ\text{C}$.

b) Measurement of specimen length

The temperature of the air in the test room shall be equal to the conditioning temperature.

Each specimen shall only be removed from the environmental chamber immediately prior to measurement. The time between removal from the environmental chamber and fitting into the measuring equipment shall not exceed 1 min. The actual duration of measurement shall be as short as possible. Immediately after the measurement, the specimens shall be replaced in the storage container.

c) Evaluation

The position of the specimen in the measuring apparatus shall be the same for each measurement. To ensure this, the specimen shall be marked.

When the reference specimens and specimens are rotated about their axis in the measuring apparatus, no changes in reading of the dial gauge greater than $\pm 1 \text{ } \mu\text{m}$ shall result.

Check measurements on the reference specimen shall be made before and after the measurements. If a change with respect to the initial measurement on the reference specimen is found during the first check measurement, the measuring apparatus shall either be corrected or the difference taken into account in the evaluation.

If a difference is found between the check measurements before and after the measurement on the specimen, the measuring arrangements shall be checked (for seating and cleanliness of measurement cups, for example) and the measurement on the specimen repeated.

For the purpose of measurement, the specimen or reference specimen shall be carefully placed vertically in the measurement cup on the base of the measuring apparatus after the measurement cup on the dial gauge has been fully pulled back. Then the upper measurement cup shall be carefully placed on the upper measuring peg, contact being effected solely by the spring force of the dial gauge.

d) Reading

The measured values shall be read off and stated to the nearest 0,001 mm. The gauge length shall be the specimen length. As a rule, the change in length shall be determined with respect to the initial value (initial measurement). Its value referred to the gauge length shall be converted to mm/m and stated to the nearest 0,01 mm/m.

The measurements are made successively at specimen temperatures of 23 °C, 0 °C, -20 °C, 40 °C and 60 °C (± 2 °C in each case). The permissible deviation of the core temperature from the storage temperature is $\pm 0,5$ °C.

4.4 Calculation of the coefficient of thermal expansion

The coefficient of thermal expansion $\alpha_{-20/40}$ should be calculated from the measured length changes, as follows:

$$\alpha_m = \frac{\Delta L_{sp}}{L \times \Delta T}$$

with the notation given in 3.4.1.

A further correction may be made to take into account the coefficient of thermal expansion of the measuring pegs.

4.5 Test report

The following information shall be included in the test report:

- a) identification of all the constituents in the mortar mix including manufacturer's name, batch numbers if applicable, type, description and date of production;
- b) temperature and duration of pre-conditioning of the constituents;
- c) date and location of mortar specimen preparation and test;
- d) the cure and storage history of the specimen prior to test;
- e) description of test equipment used;
- f) the effective length of the specimen, to the nearest millimetre;
- g) tabulation of the results obtained, giving for each temperature the change in length of the prism (with an accuracy of 0,01 mm);
- h) the coefficient of thermal expansion α_m in the range from -20 °C to 40 °C, rounded to $0,5 \times 10^{-6}/\text{°C}$;
- i) the amount of any post-cure shrinkage observed;
- j) mean value of the permanent expansion to 0,01 mm/m;
- k) reference to this European Standard.

5 Reproducibility

The test method based upon thermomechanical analysis will provide a reproducibility of ± 1 °C. The alternative test method will provide a reproducibility of ± 5 °C for determinations above 23 °C and more than ± 5 °C for determinations below 23 °C. The inferior reproducibility of the alternative test method arises due to temperature changes which occur whilst the specimen is removed from environmental chamber for length measurements to be taken.

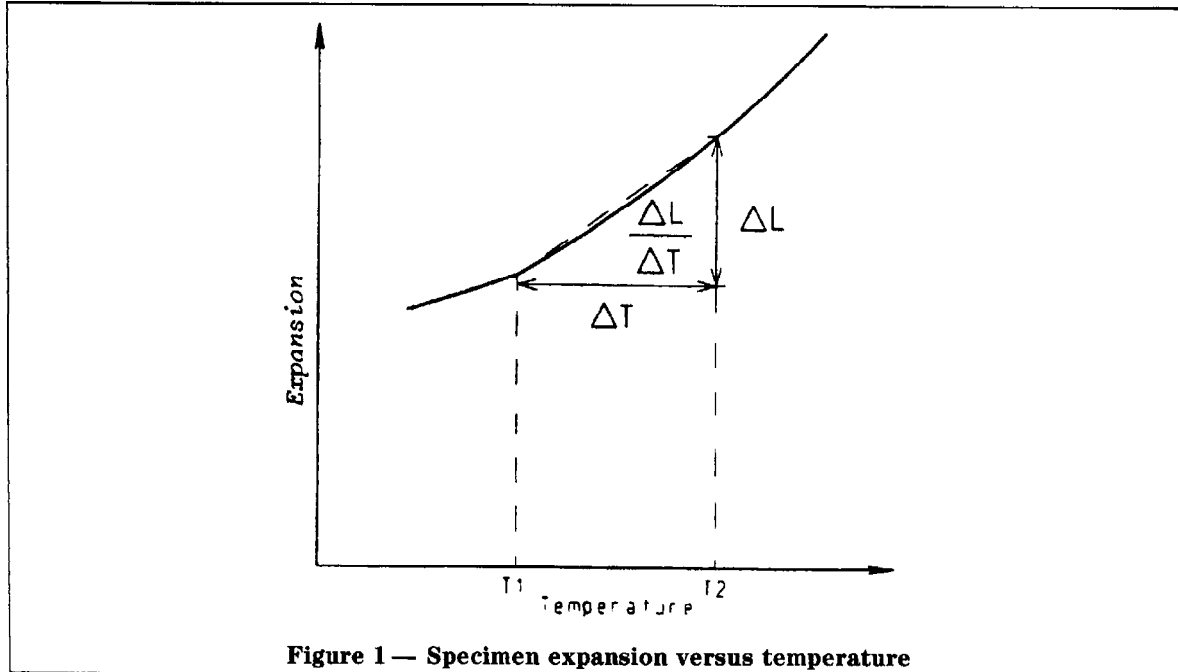


Figure 1 — Specimen expansion versus temperature

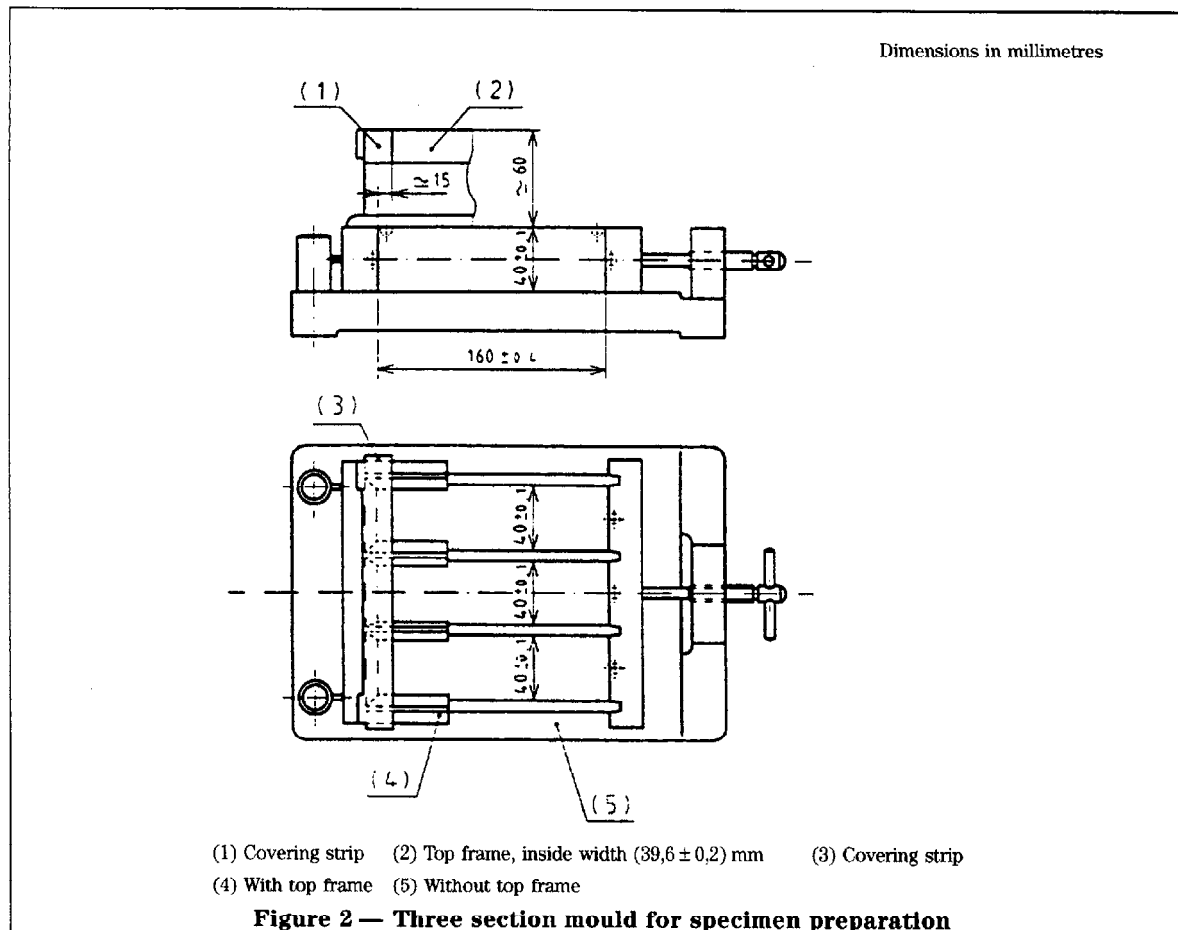
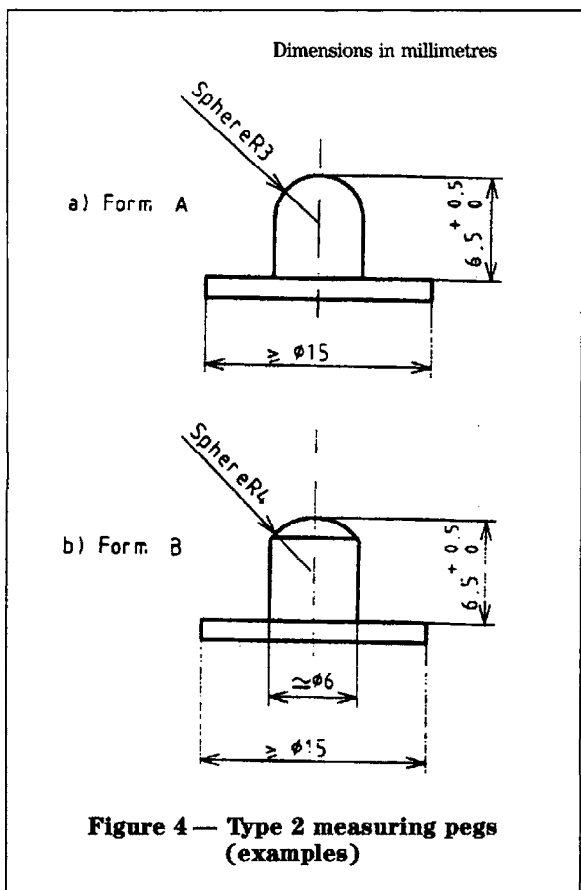
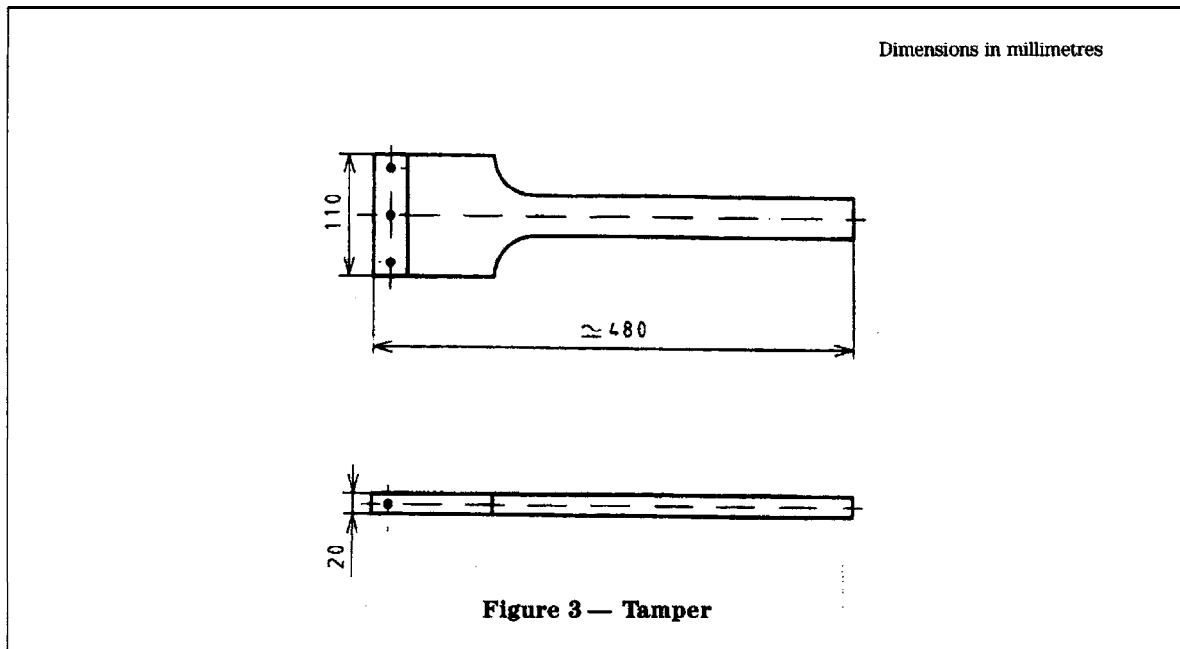
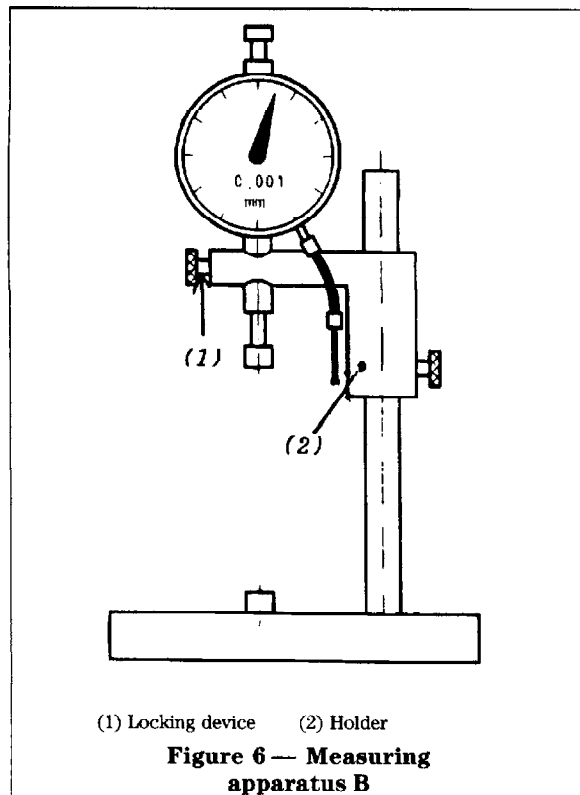
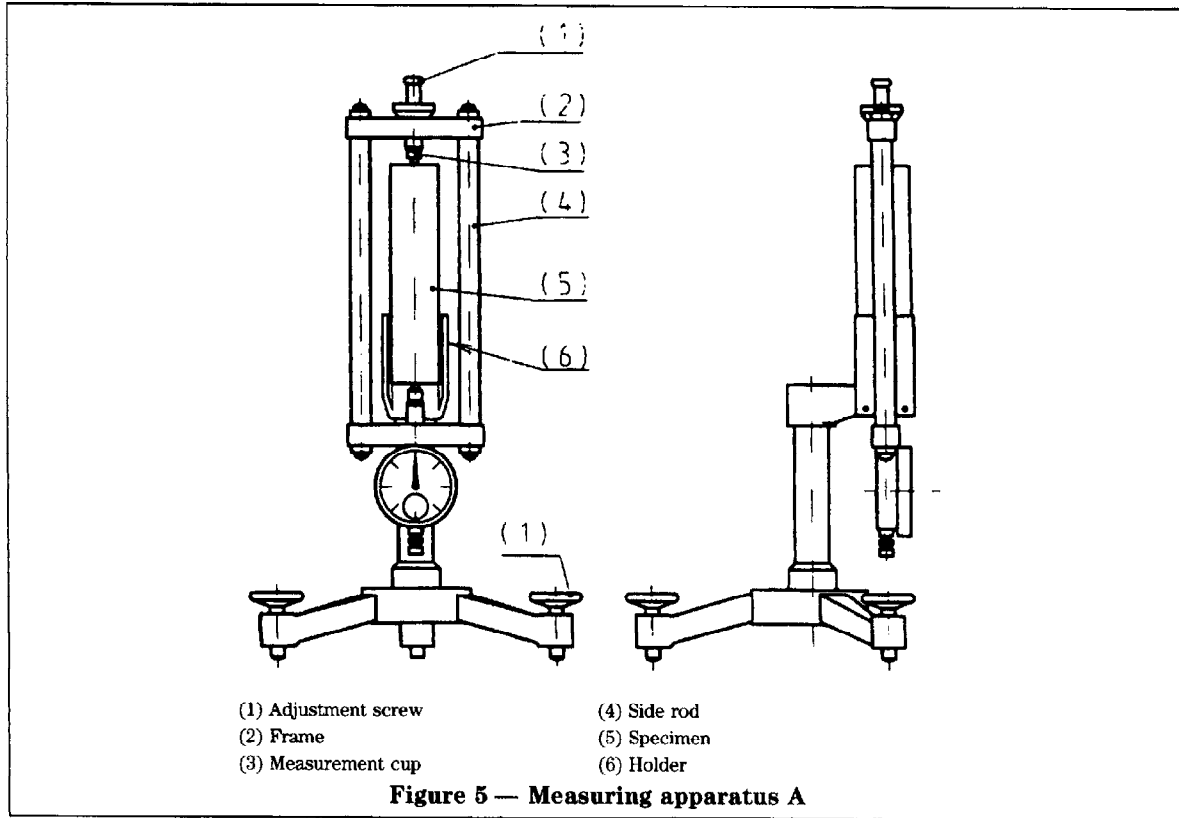
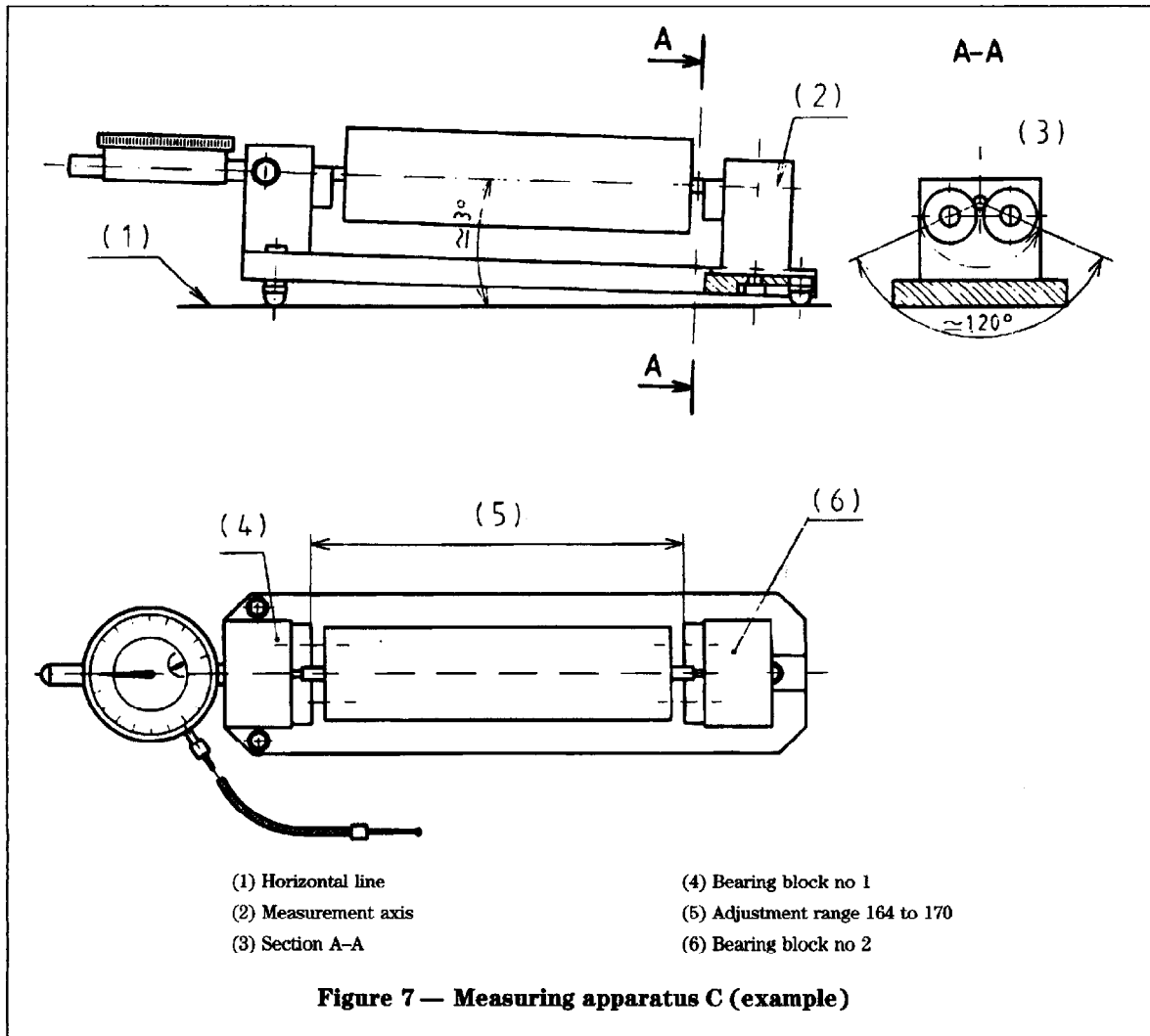


Figure 2 — Three section mould for specimen preparation







Annex A (informative)

Bibliography

- [1] DIN 51290-3, *Prüfung von Reaktionsharzbeton im Maschinenbau — Teil 3: Prüfung gesondert hergestellter Probekörper.*
- [2] ASTM C 531, *Linear shrinkage and coefficient of thermal expansion of mortars.*
- [3] ASTM E 831, *Standard test method for linear thermal expansion of solid materials by thermomechanical analysis.*
- [4] ASTM D 696, *Test method for coefficient of linear thermal expansion of plastics.*

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