BRITISH STANDARD

BS EN
821-1:1995

Advanced technical ceramics — Monolithic ceramics — Thermo-physical properties —

Part 1: Determination of thermal expansion

The European Standard EN 821-1:1995 has the status of a British Standard

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee RPI/13, Advanced technical ceramics, upon which the following bodies were represented:

AEA Technology
Aluminium Federation
British Ceramic Research Ltd.
British Industrial Ceramic Manufacturers' Association
Department of Trade and Industry (National Physical Laboratory)
Flat Glass Manufacturers' Association
GAMBICA (BEAMA Ltd.)
Institute of Refractories Engineers
Ministry of Defence
Refractories Association of Great Britain
Society of British Aerospace Companies Limited
University of Manchester

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 September 1995

The following BSI references relate to the work on this standard:
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Contents

National foreword

This British Standard has been prepared by Technical Committee RPI/13, and is the English language version of EN 821-1:1995 *Advanced technical ceramics — Monolithic ceramics — Thermo-physical properties — Part 1: Determination of thermal expansion*, published by the European Committee for Standardization (CEN).

EN 821-1 was produced as a result of international discussions in which the UK took an active part.

EN 821 consists of three Parts:

— *Part 1: Determination of thermal expansion;*

— *Part 2: Determination of thermal diffusivity;*

— *Part 3: Determination of specific heat capacity (ENV).*

Cross-references

Publication referred to	Corresponding British Standard
EN 45001	BS 7501:1989 *General criteria for the operation of testing laboratories*
ENV 1006	DD ENV:1994 *Advanced technical ceramics — Methods of testing monolithic ceramics — Guidance on the sampling and selection of test pieces* BS 4937 *International thermocouple reference tables*
HD 446.1 S1	Part 1:1973 *Platinum-10 % rhodium/platinum thermocouples. Type S* Part 2:1973 *Platinum-13 % rhodium/platinum thermocouples. Type R* Part 3:1973 *Iron/copper-nickel thermocouples. Type J* Part 4:1973 *Nickel-chromium/nickel-aluminium thermocouples. Type K* Part 5:1974 *Copper/copper-nickel thermocouples. Type T* Part 6:1974 *Nickel-chromium/copper-nickel thermocouples. Type E* Part 7:1974 *Platinum-30 % rhodium/platinum-6 % rhodium thermocouples. Type B* Part 8:1986 *Nickel-chromium-silicon/nickel-silicon (nicrosil/nisil) thermocouples including composition. Type N*

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 14, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 821-1

January 1995

ICS 81.060.10; 81.060.20

Descriptors: Ceramics, thermodynamic properties, tests, determination, thermal expansion

English version

Advanced technical ceramics — Monolithic ceramics — Thermo-physical properties — Part 1: Determination of thermal expansion

Céramiques techniques avancées — Céramiques monolithiques — Propriétés thermo-physiques — Partie 1: Détermination de la dilatation thermique

Hochleistungskeramik — Monolithische Keramik — Thermophysikalische Eigenschaften — Teil 1: Bestimmung der thermischen Längenänderung

CEN

European Committee for Standardization
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Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. 821-1:1995 E

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 184, Advanced technical ceramics, the secretariat of which is held by BSI.

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 July 1995, and conflicting national standards shall be withdrawn at the latest by July 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

EN 821 consists of three Parts:

— *Part 1: Determination of thermal expansion;*

— *Part 2: Determination of thermal diffusivity;*

— *Part 3: Determination of specific heat capacity (ENV).*

Contents

1 Scope

This Part of EN 821 describes the method for the determination of the linear thermal expansion characteristics of advanced monolithic technical ceramics up to a maximum temperature of 1 500 °C (see **5.2**) and to a specified level of accuracy A or B as defined in Table 1.

The method describes general principles of construction, calibration and operation of suitable apparatus. Specific details, including test piece dimensions, depend on the design of the apparatus. Methods of calibration are given in Annex A and Annex B. Thermal expansion reference data are given in Annex C.

2 Normative references

This European Standard incorporates by dated or undated references, 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.

EN 45001, *General criteria for the operation of testing laboratories.*

ENV 1006, *Advanced technical ceramics — Methods of testing monolithic ceramics — Guidance on the sampling and selection of test pieces.*

HD 446.1 S1, *Thermocouples — Part 1: Reference tables.*

ISO 3611, *Micrometer callipers for external measurement.*

ISO 6906, *Vernier callipers reading to 0,02 mm.*

3 Definitions

For the purposes of this Part of EN 821, the following definitions apply.

3.1
linear thermal expansion

the proportional extension which occurs when a material is heated

3.2
linear thermal expansion coefficient

the proportional extension which occurs when a material is heated over a temperature interval of 1 K at temperature T

3.3
mean linear thermal expansion coefficient

the average value of the thermal expansion coefficient over a temperature range T_1 to T_2

4 Principle

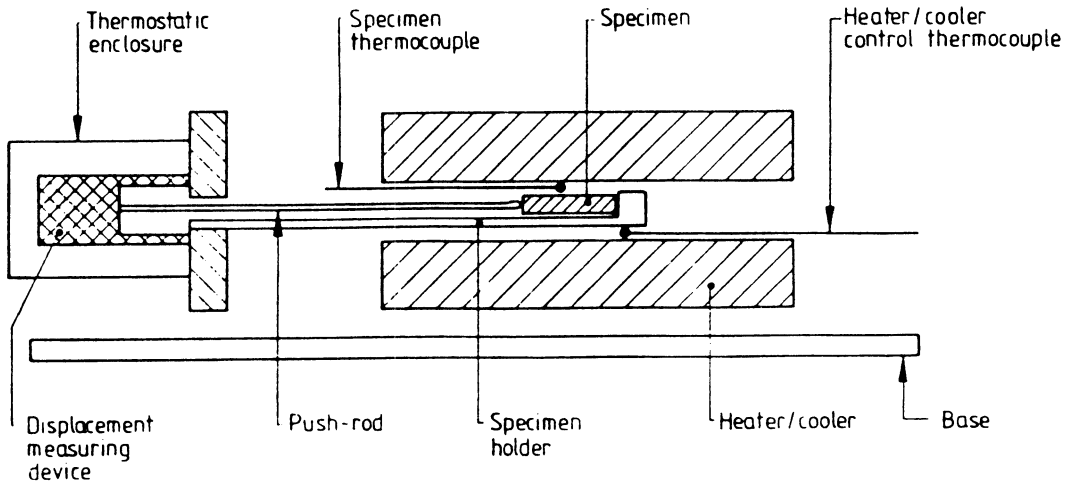
A test piece is heated and subsequently cooled, either at a specified uniform rate or using defined temperature increments. Its change of length and its temperature is measured continuously during the heating and cooling. The percentage expansion or contraction over the required temperature range is calculated and the results presented both as a mean linear thermal expansion coefficient over chosen temperature ranges, and as a graph of thermal expansion against temperature.

5 Apparatus

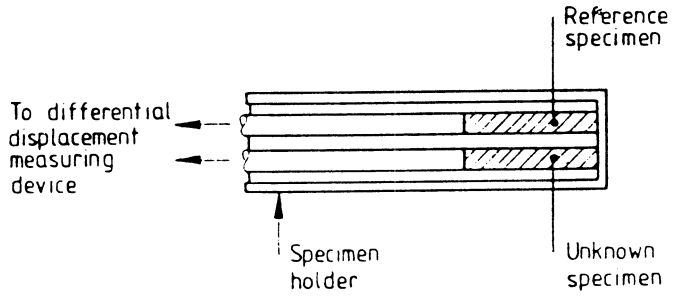
5.1 *General.* The apparatus shall conform to the specification given below and be capable of calibration according to the procedures given either in Annex A for direct measurement or Annex B for differential measurement. Any suitable proprietary apparatus may be used and suitable designs are shown in Figure 1.

Table 1 — Requirements for accuracy levels A and B

Test method requirement	Measurement accuracy required	
	A	B
Required accuracy of result over 100 K temperature interval	$\pm 0{,}1 \times 10^{-6}\ K^{-1}$	$\pm 0{,}5 \times 10^{-6}\ K^{-1}$
Temperature variation along test piece during test	< 2 K	< 5 K
Deviation from smooth temperature ramp or hold	< ± 1 K	< ± 2 K
Expansion measurement device sensitivity and repeatability (% of specimen length)	0,001 %	0,005 %
Sensitivity of recording of thermocouple temperatures	± 0,1 K	± 0,5 K



a) Horizontal dilatometer for a single test piece



b) Differential measurements using a reference material

NOTE Arrangement a) may also be inclined at an angle to the horizontal or may be vertical (see Figure 2).

Figure 1 — Schematic diagrams of apparatus arrangements

5.2 *Construction materials*. For measurements from below ambient temperature to 1 000 °C, transparent fused silica or fused quartz shall be used for construction of the test piece holder. For measurements from ambient temperature to temperatures above 1 000 °C, an alumina ceramic of at least 99,8 % Al_2O_3 shall be used for construction of the test piece holder.

NOTE The use of fused silica or fused quartz at temperatures above 800 °C can lead to structural changes or crystallization, and thus to changes in the thermal expansion coefficient of the apparatus. Calibration of the apparatus (see Annex A and Annex B) should be carried out frequently, and if there is any sign of discontinuities in the expansion curves resulting from phase transitions of cristobalite (150 °C to 250 °C) or quartz (573 °C), the apparatus should be replaced.

The push rod for transmitting the displacement of the test piece to the measuring device shall be of the same material as the test piece holder.

5.3 *Test piece holder*. Some possible constructions of the test piece holder are shown in Figure 2. For use with round-ended test pieces (see clause **6**) the outer sleeve shall have an end-plate with a surface ground flat to within 10 µm. The normal to the surface of the end-plate shall be visually square to the measurement axis. For use with flat-ended test pieces (see clause **6**) the outer sleeve shall have an end-plate which is rounded to a radius of curvature of between 1 mm and 20 mm. The push-rod end in contact with the test piece shall have similar shape to the test piece holder end-plate.

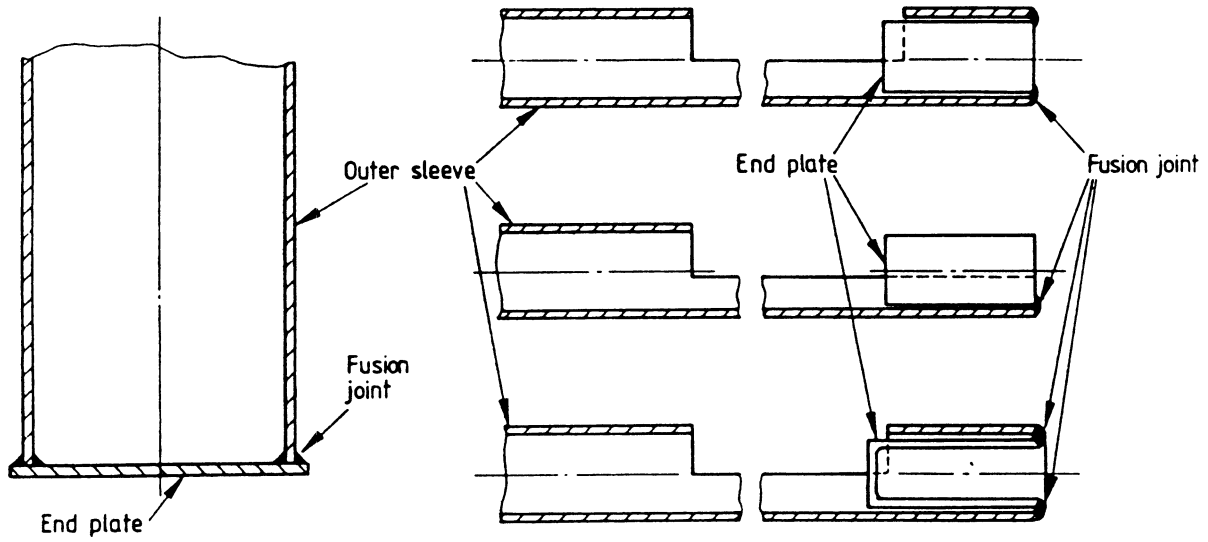
For test piece holders constructed from fused silica tube or rod (see **5.3**) the end-plate shall be rigidly fixed to the outer sleeve by flame fusing at a point remote from the end-plate surface. Alternatively, for outer sleeves constructed from fused silica rod, the end-plate may be prepared by machining from solid material. Examples of constructions are shown in Figure 2 a).

For test piece holders constructed from alumina (see **5.2**) rod, the end-plate may be prepared by machining from solid material. If constructed from alumina tube, fusion is not possible.

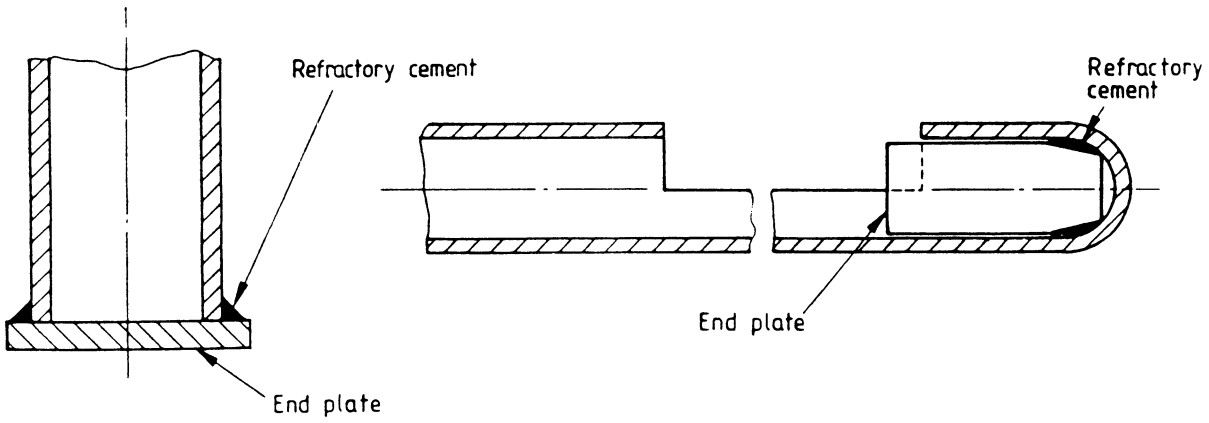
For vertical measuring apparatus, the outer sleeve shall be fixed to a flat plate using refractory cement outside the joint as shown in Figure 2 b). For non-vertical measuring apparatus in which the sleeve end is free-standing, a closed end alumina tube shall be used, and the end-plate machined to fit inside the tube end as shown in Figure 2 c). The assembly shall then be fixed in position with a suitable refractory cement, and then fired to a temperature greater than 1 400 °C under an axial load.

NOTE The firing under load minimizes the risk of movement of the assembly during use. Some movement may still occur during initial use. Thermal cycling should be continued until the net movement over a thermal cycle to the highest temperature at which the apparatus is to be used is less than 1 µm.

Alternatively, an end-plate may be shaped to fit in tangential slots in the tube. If this approach is adopted, the components shall be designed to fit together with no possibility of relative movement in use, e.g. by wedging into position.



a) Fused silica test piece holders for horizontal and vertical use



b) Alumina test piece holder for vertical use c) Alumina test piece holder for horizontal or vertical use

Figure 2 — Suitable constructions of test piece holders in silica and alumina

5.4 *Test piece mounting*. For vertical measuring apparatus, the test pieces shall be free-standing and mechanically stable on the end-plate (see **5.3**). For measuring apparatus which is horizontal or inclined to the horizontal, the sideways movement or twist of the test piece shall be restricted, without any restriction of axial movement, by a suitable arrangement.

NOTE This may be done, for example, by
a) using a vee-block cut to fit into the test piece holder; or
b) by using test pieces of such dimensions that either a neat sliding fit (but see clause **6**) or support on two edges is obtained; or
c) using an arrangement of supporting silica glass or alumina balls, as shown in Figure 3.

Where the apparatus is designed for differential measurements, two test pieces shall be suitably mounted parallel to each other, contacting on a single end-plate.

5.5 *Thermocouples*. The thermocouples shall be type R, S or K, in accordance with HD 446.1. One thermocouple shall be placed with its junction in contact with the surface of the test piece near its mid-point. Two other thermocouples shall be placed at each end of the test piece, and used in differential mode as shown in Figure 4, to periodically determine the temperature distribution along the test piece.

5.6 *Heating or cooling device*, comprising a suitable tube furnace or cooling device designed to give a uniform temperature zone of length greater than that of the test piece during the normal thermal cycling of the test (see clause **7**). The device shall heat or cool the test piece contained in its holder (see **5.3**) and in any surrounding protection tube.

The variation in temperature along the length of the test piece within the device shall be determined using differential thermocouples positioned as shown in Figure 4, and shall not exceed the value given in Table 1 during thermal cycling.

5.7 *Temperature programmer and power control unit*, for temperature control of the heating and cooling device (see **5.6**), incorporating a thermocouple (see **5.5**) positioned in the uniform temperature zone of the device. This apparatus shall be such that for tests at a constant rate of change of temperature, deviation from a smooth rate of change shall not exceed the value given in Table 1, as determined by the thermocouple in contact with the test piece. For tests at a series of steady temperatures, variations in temperature shall not exceed the value given in Table 1 of the mean temperature.

5.8 *Expansion measuring device*, either a micrometer, dial gauge, linear displacement transducer, or an interferometer. The device shall have a capability of measuring displacements of the push-rod relative to the specimen holder according to the level set in Table 1 (see clause **6**), and individual measurements shall be repeatable to this accuracy.

Where the apparatus is designed for differential measurements, the measuring device shall directly record the differential movement of the two push rods.

NOTE 1 The difference between separately measured push rod displacements is not acceptable as a measurement.
NOTE 2 Ideally, the expansion measuring device should have its temperature controlled to ± 1 K, using a thermostatic device such as a water jacket operating at a temperature near to, but preferably a little above room temperature. Checks should also be made to ensure that any electronic amplifiers or recording devices used such as a transducer amplifier and recording voltmeter, have outputs that are insensitive to room temperature change. If such changes in output exceed the equivalent of a displacement of 10^{-3} mm for a 10 K change in room temperature, then the room temperature should ideally be controlled to ± 1 K.

5.9 *Data recording unit*, providing for simultaneous recording of test piece temperature and displacement measuring device output. The sensitivity for temperature measurements shall be set according to Table 1.

NOTE This is equivalent to 1 µV for accuracy A or 5 µV for accuracy B with type R or type S thermocouples and 4 µV for accuracy A or 20 µV for accuracy B with type K thermocouples (see **5.5**).

The thermocouple (see **5.5**) shall have a reference cold junction which may be the temperature of the thermostatically controlled measurement unit, a separate thermostatic unit, or electronic compensation in the recording device as appropriate.

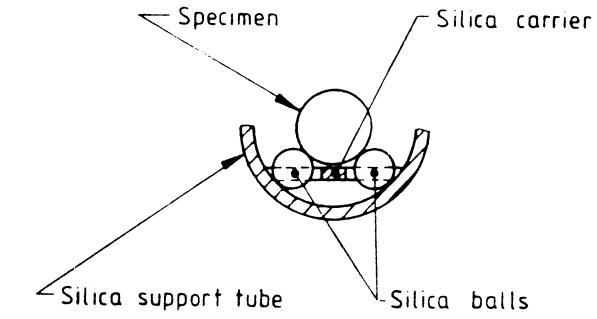
The sensitivity for displacement measurement shall be as described in **5.8**.

6 Test pieces

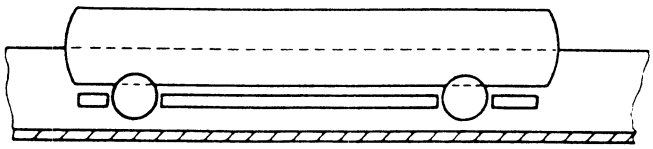
Materials for testing should be sampled in accordance with the guidance given in ENV 1006.

The dimensions of the test pieces are dependent on the design of the apparatus (see clause **5**), and in particular the temperature homogeneity of the heating or cooling device (see **5.6**).

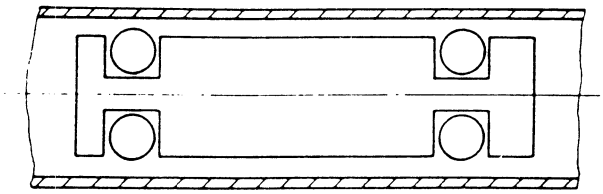
The length of the test piece shall be similar to that of any certified reference material used to calibrate the apparatus as in Annex A. For the differential type of apparatus, the length of the test piece shall be within ± 0,2 mm of the length of the piece of reference material.



a)



b)



c)

NOTE This shows:
a) the cross section;
b) the side view; and
c) the top view of carrier without the test piece.

The carrier may also comprise a series of short silica rods to retain the position of the silica balls.

Such an arrangement may also be made from alumina.

Figure 3 — Silica ball test piece support and carrier

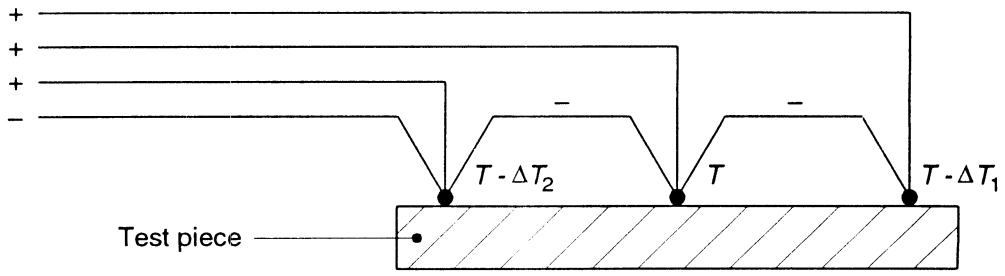


Figure 4 — Example of arrangement of differential thermocouples to monitor temperature distribution in the test piece, where ΔT_1, ΔT_2 are differential temperatures (see Table 1)

NOTE 1 When the test specimen comprises coarse-grained or heavily textured materials, care should be taken that the test specimen is fully representative of the material or component for which data are required.

The cross section of the test piece may be any suitable shape consistent with the design of the apparatus. For materials of thermal conductivity less than 10^{-1} W(m·K)$^{-1}$, one dimension shall be less than 5 mm.

NOTE 2 This minimizes transverse temperature gradient through test pieces during heating or cooling.

For use with apparatus with a flat test piece holder, end plate and push rod end (see **5.3**), the ends of the test piece shall be ground to a radius of curvature between 1 mm and 20 mm, so as to ensure point contact between specimen and apparatus (see Figure 5), except where a vertical measuring device is used with a free-standing test piece, where the lower end of the test piece in contact with the reference base may be ground flat, perpendicular to the test piece axis. In this case the test piece shall be mechanically stable on the reference base.

For use with apparatus with a round-ended test piece holder end plate and push rod, the test piece shall have ends ground flat and parallel (see Figure 5). The test piece end surfaces shall be visually square to the measurement axis. For the differential method, the reference test-piece shall be any suitable certified material, or one selected from these given in Annex C and appropriate for the temperature range over which measurements are to be made.

7 Procedure

Measure the length of the test piece at room temperature, using either vernier callipers in accordance with ISO 6906, a micrometer in accordance with ISO 3611, or other suitable measuring device, to an accuracy of either:

a) ± 0,05 mm for lengths of 10 mm or above

b) ± 0,02 mm for lengths of below 10 mm.

Ensure that the apparatus is assembled as specified in clause **5** and has been recently calibrated in accordance with either Annex A or Annex B. Insert the test piece in the apparatus, and raise the temperature with the heating device (see **5.6**), using one of the two alternative heating rates given below.

1) A constant rate of heating and cooling, specified, and of a maximum of 2 K/min for accuracy A and 5 K/min for accuracy B, to the maximum test temperature.

2) a rate of heating using step-wise temperature increments to the maximum temperature, and the same steps in the fall to ambient temperature. The size of the temperature increments shall be by agreement, covering suitable ranges for which data are required. At the end of each temperature step, the apparatus shall be maintained at constant temperature until no change in apparent length occurs over a period of 15 min.

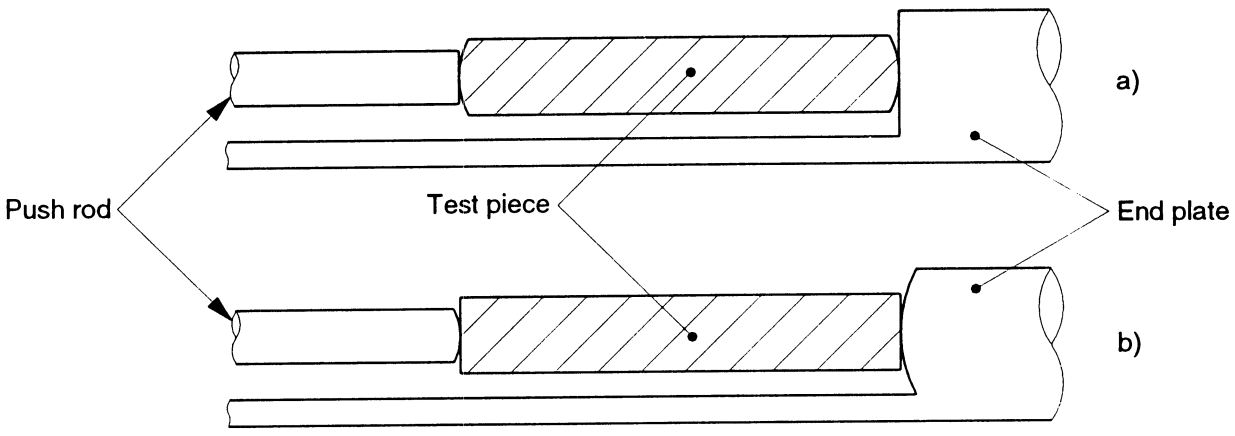


Figure 5 — Test pieces with a) rounded ends for use with flat end plate and push rod surfaces and b) flat square ends for use with rounded end plate and push rod surfaces in a conventional horizontal dilatometer

NOTE Calibration of the equipment (see Annex A or Annex B) should be made under the same conditions as are used for measurement, and with reference materials of similar length.

Whichever heating rate is chosen, take readings of the length of the test piece either continuously or at regular intervals, depending on the type of temperature recording apparatus (see **5.9**). If the constant rate of heating is used, take readings at a maximum temperature interval of 10 K.

8 Calculation of results

Calculate the mean linear thermal expansion coefficient in accordance with equation 1 (direct measurements) or equation 2 (differential measurements).

$$\bar{\alpha} = \frac{S\Delta X\,\delta}{L_o \Delta T} + \bar{\alpha}_A \qquad (1)$$

$$\bar{\alpha} = \frac{S\Delta X - \delta}{L_o \delta T} = \frac{L_{REF}}{L_o}\,\bar{\alpha}_{REF} \qquad (2)$$

where

$\bar{\alpha}$ is the mean thermal expansion coefficient of the test piece, over the temperature range ΔT

S is the measurement sensitivity of the displacement recording system (see **A.1**)

NOTE $S = 1$ for a direct-reading dial gauge, or a calibrated number of mm/volt for a displacement transducer or mm/fringe for a interferometer.

ΔX is the recorded displacement (in mm, volts or fringes)

L_o is the initial length in mm of the test piece at room temperature

L_{REF} is the initial length in mm of the reference specimen at room temperature

ΔT is the temperature interval in K from temperature T_1 to temperature T_2 for which the change of length is measured

δ is the base-line displacement (mm) inherent in the recording system when the apparatus temperature changes by ΔT from T_1 to T_2

$\bar{\alpha}_A$ is the mean thermal expansion coefficient correction for the apparatus over the temperature range used (see **A.3**)

$\bar{\alpha}_{REF}$ is the mean thermal expansion coefficient of the reference specimen in the differential method

9 Reporting of results

The calculated mean linear expansion coefficients shall be reported for at least two thermal cycles and for temperature ranges as required, and curves plotted of thermal expansion expressed as a percentage or as ppm against temperature (i.e. $\Delta L/L$ against temperature). Graphical curves shall be presented for both heating and cooling cycles. The net offset recorded over the heating and cooling cycles shall be reported.

NOTE 1 Any features identifiable as phase changes or softening under the load of the measuring device should also be reported, together with significant differences between the shapes of the heating and cooling curves.

The mean thermal expansion coefficient over the required temperature range shall be reported to the nearest $0.1 \times 10^{-6}\ K^{-1}$ for accuracy A and $0.5 \times 10^{-6}\ K^{-1}$ for accuracy B.

NOTE 2 The temperature ranges used to prepare the results of mean expansion coefficient shall be by agreement. It is customary to employ 20 °C or 25 °C as the temperature near to room temperature, while the other temperature may be in multiples of 50 °C or 100 °C.

10 Test report

The results shall be reported in accordance with EN 45001 and the test report shall include the following information:

a) the name and address of the testing establishment;

b) the date of the test, unique identification of report and of each page, customer name and address and signatory;

c) a reference to this European Standard, i.e. Determined in accordance with EN 821-1: accuracy A (or B, as appropriate);

d) a description of the apparatus, including the name of the manufacturer and the material of construction for the test piece holder and push rod (see **5.2**);

e) the method of calibration;

f) the description of the test material; material type, manufacturing code, batch number, date of receipt;

g) a description of the test piece, including its dimensions, preparation and orientation of length if cut from a component (see clause **6**);

h) the heating and cooling rate used, or if stepped temperature increments are used, the number of and temperature interval of the steps (see clause **7**);

i) the maximum temperature reached by the test piece;

j) the atmosphere during testing;

k) the mean linear thermal expansion coefficients, over stated temperature ranges determined from two or more thermal cycles (see clause **9**);

l) graphs of thermal expansion against temperature for each thermal cycle given in clause **10** k);

m) comments about the test or the test results (see clause **9**).

Annex A (normative) Calibration of apparatus (direct-measuring instruments)

A.1 Measurement sensitivity

The sensitivity of the measurement device, S, requires to be calibrated if it is not based on a direct-reading dial gauge or micrometer. This may be done either by using direct mechanical movement produced by using a certified micrometer of sufficient accuracy, or by use of certified thermal expansion reference materials as test pieces (see Annex C).

Calibration tests shall be made for mechanical movement in both directions, and the mean value of sensitivity taken. Any non-linearity shall be reported, and the apparatus shall be calibrated over the same range of displacement as that to be used for any series of tests.

NOTE This procedure applies to linear displacement transducer measuring devices in particular.

Where two certified thermal expansion reference materials (see Annex C) are available, the sensitivity is determined by use of equation 1 in clause **8** for the two materials:

$$\bar{\alpha}_1 = \frac{S\Delta x_1 - \delta}{L_{01}\,\Delta T} + \bar{\alpha}_A \qquad (3)$$

$$\bar{\alpha}_2 = \frac{S\Delta x_2 - \delta}{L_{02}\Delta T} + \bar{\alpha}_A \qquad (4)$$

where

$\bar{\alpha}_1$, $\bar{\alpha}_2$ are the mean thermal expansion coefficients of the two reference materials, over the temperature range ΔT

L_{01}, L_{02} are the initial lengths in mm of the two reference material pieces at room temperature

Δx_1, Δx_2 are the recorded displacements (in mm, volts or fringes), over the temperature range ΔT

ΔT is the temperature interval for which the changes in length are measured

S is the measured sensitivity of the displacement recording system

δ is the base-line shift (see **A.2**), over the temperature range ΔT

$\bar{\alpha}_A$ is the mean thermal expansion coefficient correction for the apparatus (see **A.3**)

$$\text{thus: } S = \frac{(\bar{\alpha}_1 - \bar{\alpha}_2)\,\Delta T}{\left(\frac{\Delta x_1}{L_{01}} - \frac{\Delta x_2}{L_{02}}\right)}, \text{ when } L_{01} = L_{02} \qquad (5)$$

A.2 Measurement of base line shift

This calibration is carried out by using a test piece made of the same material as the push rod, or by moving the push rod to come into contact with the end plate. A test is carried out according to the procedure given in clause **7**, and the displacement recorded is then due to different temperature distributions along the lengths of the push rod and the test piece holder.

NOTE For silica apparatus the base-line shift is usually quite small, but for alumina apparatus it can be larger because of the higher thermal conductivity and higher thermal expansion. The base-line shift may be either positive or negative. The above analysis assumes that at a maximum heating rate of 2 K/min (accuracy A) or 5 K/min (accuracy B), the base-line shift is consistent and is due solely to the apparatus, not to any thermal lag in the test piece.

A.3 Measurement of apparatus expansion correction

The expansion of the apparatus δ_A will depend on the materials of construction chosen (see **5.2**).

NOTE This is particularly the case with alumina materials fabricated by extrusion.

Calibration shall be carried out by using a certified reference material of similar thermal expansion to that of the apparatus, and carrying out the test in accordance with the procedure given in clause **7**. The apparatus expansion correction is then obtained by inserting calibrated and experimental data for the reference material in equation 1.

A.4 Frequency of calibration

The apparatus shall be re-calibrated whenever any of the following circumstances occur:

a) instabilities in or replacement of electrical or electronic measuring or reading units;

b) replacement of any mechanical part of the apparatus;

c) use of the equipment at very high temperatures (greater than 800 °C for fused silica, greater than 1 400 °C for alumina);

d) use of the equipment for the measurement of materials which react with the measurement device;

e) use of the equipment under a strongly reducing atmosphere, or in contact with carbon.

NOTE In addition to these requirements, calibrations should be made at regular intervals where none of these circumstances apply.

Annex B (normative) Calibration of apparatus (differential type)

B.1 Introduction

Differential-type apparatus is normally operated with a reference material of known expansion characteristics and of length similar to that of the test piece. In such cases the procedure given in Annex A is not required and is replaced by that given in **B.2**.

B.2 Procedure

Carry out the calibration by making a thermal expansion cycle, using as a test piece a second certified reference material (**A.1**) with a significantly different thermal expansion characteristics to that of the one normally used. The output recorded then corresponds to that due to the difference in expansion, as given by the equation:

$$S\Delta x_1 = (L_{01}\bar{a}_1 - L_{02}\bar{a}_2)\,\Delta T + \delta \qquad (6)$$

where the symbols have the same meaning as in **A.1** and $\bar{a}_1 > \bar{a}_2$.

Reverse the specimen positions and carry out a further thermal expansion cycle. The recorded output is then:

$$S\Delta x_2 = (L_{02}\bar{a}_2 - L_{01}\overline{a_1})\,\Delta T + \delta \qquad (7)$$

with Δx_2 negative.

The apparatus sensitivity S is given by:

$$S\,(\Delta x_1 - \Delta x_2) = 2(L_{01}\overline{a_1} - L_{02}\bar{a}_2)\,\Delta T \qquad (8)$$

and the baseline shift δ is given by:

$$\delta = 0{,}5S\,(\Delta x_1 + \Delta x_2) \qquad (9)$$

NOTE 1 The term δ is included because of the possibility of unequal responses from the two push rods.

NOTE 2 Corrections for base-line shift (δ) should be small. The values calculated using the above procedure may be checked by bringing the push-rods into contact with the end plate.

B.3 Frequency of calibration

Re-calibrate the apparatus whenever any of the circumstances given in **A.4** occur. Replace the reference test piece at regular intervals, or whenever used under conditions likely to cause degradation or changes in expansion coefficient.

Annex C (informative) Thermal expansion reference data

C.1 Materials

The preferred calibration method for dilatometers is the use of certified reference materials. However, if such materials are not available it is acceptable to use the following materials in high purity form (0.01 % impurities).

a) Low expansion:	silicon, single or polycrystalline, to 700 °C in air, 1 000 °C in inert conditions
b) Medium expansion:	tungsten, polycrystalline, to 300 °C in air, 1 500 °C in inert conditions
c) High expansion:	platinum, to 1 300 °C in air or inert conditions
d) Very high expansion:	copper, to 300 °C in air or 800 °C in inert conditions

Each of these materials is crystallographically cubic and in the annealed form should exhibit the same thermal expansion characteristics as those reported below.

NOTE Crystallographically anisotropic materials, such as alumina or sapphire, or unstable materials, such as fused silica, require certification.

C.2 Data

This table of data has been extracted from various referenced sources. In many cases the values given have been calculated from polynomial curve-fitting equations. The accuracy is considered to be typically ± 1 %, which is probably better than the accuracy and repeatability exhibited by mechanical dilatometers. The expansion data are given as

$$\frac{\Delta L}{L}$$

referenced at 20 °C.

The mean expansion coefficient may be calculated by dividing by the temperature interval from 20 °C or any other reference temperature.

Curve-fitting equations for expansion as a function of temperature may be found in a number of reference sources and may be useful for computer-based calibrations.

Table 2 — Thermal expansion reference data

Temperature °C	$\Delta L/L_{20}$, parts per million, from 20 °C to temperature			
	Silicon (1)[a]	Tungsten (2, 3)	Platinum (4, 5)	Copper (6, 7)
– 223	– 219	– 864	—	– 3 185
– 173	– 240	– 762		– 2 815
– 73	– 190	– 398	—	– 1 480
– 23	– 101	– 188	—	– 704
0	– 49	– 87	—	– 328
20	0	0	0	0
25	12	22	36	83
50	82	132	268	501
100	228	356	722	1 356
150	392	584	1 185	2 233
200	562	814	1 654	3 124
250	741	1 048	2 130	4 035
300	923	1 283	2 611	4 963
350	1 120	1 520	3 097	5 909
400	1 318	1 760	3 590	6 872
450	1 519	2 000	4 088	7 855
500	1 727	2 243	4 592	8 857
550	1 932	2 487	5 102	9 880
600	2 142	2 732	5 619	10 923
700	(2 576)	3 229	6 677	13 077
800	—	3 730	7 767	15 327
900	—	4 245	8 894	17 686
1 000		4 769	10 057	—
1 100	—	5 304	11 258	—
1 200	—	5 853	12 499	—
1 300	—	6 417	13 782	—
1 400	—	6 997	15 114	—
1 500	—	7 595	16 506	—
1 600	—	8 210	17 975	—

[a] NOTE The numbers refer to the documents listed in Annex D.

Annex D (informative)
Bibliography

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List of references

See national foreword.

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