
**Hard coal — Method for the measurement
of the swelling of hard coal using a
dilatometer**

*Houille — Méthode pour le mesurage du gonflement de la houille au
moyen d'un dilatomètre*

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 23873 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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Introduction

The dilatation of coal has been primarily measured by two different standard procedures: the Ruhr dilatometer and the Audibert-Arnu dilatometer. Both of the methods were very similar, having the same basic apparatus and heating rate but using different initial masses of coal arising from the method of pencil preparation.

This International Standard combines the two previous procedures by adjusting the equipment specification to define a single acceptable method.

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Hard coal — Method for the measurement of the swelling of hard coal using a dilatometer

1 Scope

This International Standard describes a method for the measurement of the swelling of hard coal using a dilatometer.

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.

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 and the following apply.

3.1

softening temperature

temperature of initial contraction

temperature at which the downward movement of the dilatometer piston is 0,5 mm

NOTE See temperature θ_1 in Figure 3.

3.2

temperature of maximum contraction

temperature at which the dilatometer piston reaches its lowest point

NOTE See temperature θ_2 in Figure 3.

3.3

temperature of maximum dilatation

temperature at which the dilatometer piston reaches its highest point

NOTE See temperature θ_3 in Figure 3.

3.4

maximum contraction

maximum downward movement of the dilatometer piston, measured from the zero point

NOTE 1 Maximum contraction is expressed as a percentage of the initial test-piece length.

NOTE 2 See c in Figures 3 and 4.

**3.5
maximum dilatation**

maximum upward movement of the dilatometer piston after contraction, measured from the zero point

NOTE 1 Maximum dilatation is expressed as a percentage of the initial test-piece length.

NOTE 2 See *d* in Figures 3 and 4. The value can be either positive or negative.

**3.6
repeatability**

maximum acceptable difference between two determinations which are carried out in the same laboratory, by the same operator with the same apparatus, on test pieces prepared from the same test sample and tested simultaneously in two different retorts during the same heating cycle or separately in the same retort during different heating cycles

**3.7
reproducibility**

maximum acceptable difference between the means of two determinations which are carried out in each of two laboratories, on representative portions taken from the same gross sample, after the last stage of sample preparation

4 Principle

A test piece, in the form of a pencil, prepared from powdered coal is heated at a constant rate in a steel retort positioned in a furnace, the temperature monitoring system having been previously calibrated using two reference metals of known melting points. The change in level of a piston resting upon the test piece is observed continuously, and a record is produced which is characteristic of the swelling properties of the coal.

5 Materials

The following materials are required for temperature calibration (7.1).

5.1 Graphite pencils, 30 mm long, base diameter 7,4 mm, top diameter 6,8 mm, with a small cylindrical reservoir drilled in the narrow end of each pencil.

5.2 Metal balls, of the following reference metals:

- a) **lead**, of analytical reagent grade, assay (Pb) 99,98 % minimum, melting point 327,0 °C;
- b) **zinc**, assay (Zn) 99,87 % minimum, melting point 419,3 °C.

5.3 Water-based blacking.

6 Apparatus

6.1 Mould and accessories

6.1.1 Mould (see Figure 1), made from steel, case-hardened after machining. The bore shall be polished after hardening and the bore and uniformity of taper (i.e 1 in 50) shall conform to the dimensions given in Table 1.

6.1.2 Mallet, plastics head, mass about 200 g.

6.1.3 Ram (see Figure 1).

6.1.4 Press (see Figure 1).

6.1.5 Load cell (see Figure 1), capable of registering a load of 0 to 15 kN.

6.1.6 Pencil gauge (see Figure 1).

Table 1 — Dimensions of mould

Distance from wide end	Bore (tolerance: -0,00, +0,005)
0	7,4
10	7,2
20	7,0
30	6,8
40	6,6
50	6,4
60	6,2
70	6,0

6.2 Dilatometer

A general arrangement of suitable dilatometer apparatus giving critical dimensions is shown in Figure 2.

6.3 Dilatometer furnace

A furnace capable of heating two or more retorts (6.6) to a temperature of 550 °C at a rate of 3 °C/min is used.

A suitable furnace (for heating three retorts) is illustrated in Figure 2 and consists of a casing fitted with a base and a top cover. The cover supports a cylindrical block of copper-aluminium alloy CA 104, of 65 mm diameter and 460 mm long. The block has three holes of 380 mm minimum depth and 15,0 mm ± 0,1 mm diameter, drilled as shown in Figure 2. The top surface may be insulated by an appropriately shaped piece of board. The block is heated electrically by an insulated resistance winding, capable of raising the temperature of the block to 550 °C at a rate of 3 °C/min. The space between the block and the casing is filled with a thermal insulating material. A suitable temperature sensor is positioned in the third retort in such a way that the sensor tip lies centrally 30 mm above the internal base of the retort. The distance of 30 mm is established by using a graphite pencil (5.1) as a means of measurement.

The furnace shall comply with the following operating conditions.

Heat the furnace to 400 °C, measuring the temperature at the standard sensing point, i.e. at a position equivalent to that of the centre of a normally sited test piece 30 mm above the internal base of the sensing retort. Place a second temperature-measuring device (e.g. a probe), of similar precision, in one of the other retorts at the same height above its base. If the two devices differ, apply a correction. Heat the furnace at a rate of 3 °C/min and, when the temperature has reached about 450 °C, measure the temperature over the lower 250 mm of the retort using the probe. The difference between the corrected probe temperature and the mean temperature shown at the standard temperature-sensing position shall be not more than:

- 2 °C in the lower 120 mm;
- 5 °C from 120 mm to 180 mm;
- 10 °C from 180 mm to 250 mm.

The instrument used to measure the temperature can be either the temperature recorder described in 6.5 or another of at least equal precision.

6.4 Temperature controller

The temperature controller shall be a separate instrument from that used to record the rise of temperature during the test. It shall be of the automatic, programmed type capable of maintaining a mean rate of temperature rise of $3\text{ °C/min} \pm 0,05\text{ °C/min}$ between 250 °C and 550 °C with a variation of not more than $\pm 1\text{ °C}$ per 30 °C rise in any 10 min period, with a precision of $\pm 1\text{ °C}$.

6.5 Temperature recorder

A suitable means of producing a complete record of the temperature variation during the test.

6.6 Retort and piston

A cylindrical retort of cold-drawn seamless tube of steel fitted with a gastight threaded plug at its base and a collar at its top. When inserted in a hole in the furnace, the retort shall be supported only by the collar with the threaded plug clear of the bottom of the hole.

When new, the internal diameter of the retort shall be $8,00\text{ mm} \pm 0,05\text{ mm}$ and the external diameter shall be $14,5\text{ mm} \pm 0,1\text{ mm}$. Check the internal diameter with a suitable ball gauge when new, and again after 100, 150, 200, etc., tests. If the internal diameter of the lower third of the retort has increased at any point to more than $8,075\text{ mm}$, discard the retort.

The piston is machined from rod made of steel. Adjust the combined mass of the piston and pen assembly to $150\text{ g} \pm 5\text{ g}$ by machining cut-out portions from the piston. The difference between the diameter of the piston and the internal diameter of the retort shall be $0,2\text{ mm} \pm 0,05\text{ mm}$ on manufacture. If this difference exceeds $0,275\text{ mm}$ in use, the piston shall be replaced. The piston shall slide freely in the retort.

A stand shall be provided to allow the retorts and pistons to cool in a vertical position after removal from the furnace.

6.7 Means of recording piston movement

A suitable means of recording piston movement versus time on a chart shall be used. The horizontal scale (time) shall be such that, when converted to temperature (see 7.3.3), a range of 180 °C will occupy a length of at least 150 mm . On the vertical scale, 5 % expansion or contraction shall occupy at least 3 mm . This may be achieved either by a mechanical pen/chart system or a transducer/electrical signal recorder.

A simple mechanical system is illustrated in Figure 2. In this example, two tests are recorded simultaneously on opposite sides of the chart by means of pens clipped firmly to the tops of the pistons. The chart is fixed to a cylinder which is rotated at uniform speed by either a clockwork or a synchronous motor and is mounted on a stand which is clamped to the top of the dilatometer outer casing.

6.8 Cleaning instruments

6.8.1 Auger, of diameter approximately $7,8\text{ mm}$ and stem length 400 mm .

6.8.2 Wire brush, of diameter 8 mm and stem length 400 mm .

NOTE A wear-resistant steel reamer, of diameter $7,95\text{ mm}$ and stem length 400 mm , can also be used.

7 Procedure

7.1 Temperature calibration

Check that the standard temperature sensor is accurate by either of the following procedures. Carry out the operations for each retort in the furnace, other than the retort used for the standard temperature sensor.

- a) Coat the lower 30 mm of the internal wall and the screw thread and the sealing plug of the retort, as well as the lower face of the piston (6.6), with a thin layer of blacking (5.3) prior to testing in order to prevent the molten reference metals adhering to the steel construction material. Dry by gentle warming. Place a lead ball [5.2 a)] in the recess at the narrow end of a graphite pencil (5.1). Place the pencil in a retort, replace the screw plug and assemble the piston and recording mechanism. Insert the retort assembly into the furnace at a temperature of approximately 280 °C and determine the melting point of the lead using the procedure described in 7.3.3, replacing the test piece (7.2.2) by a prepared graphite pencil. Repeat this procedure using a zinc ball [5.2 b)]. Before re-using graphite pencils, heat the narrow end of each pencil in a bunsen flame for a few seconds and shake the molten metal from the cylindrical reservoir.
- b) Place an independently calibrated reference-temperature-measuring device in the retort, with the measurement point 30 mm from the internal base of the retort. Starting at 300 °C, raise the temperature of the furnace to 550 °C using the normal mechanism. Record the temperature in both the standard sensing retort and the retort under test at 50 °C intervals.

If the difference between the standard and indicated temperatures is less than 7 °C, establish a factor to correct the indicated temperatures. If the difference is greater than 7 °C, check the sensor/indicator system by, for example, direct potentiometric calibration against a standard electromotive force (e.m.f).

Repeat the calibration after 200 tests or after 3 months use, whichever occurs first, or if any component is replaced.

7.2 Preparation of test sample and test pieces

7.2.1 Test sample

7.2.1.1 General

Two alternative methods of preparing the test sample are described. If the determination is to be carried out immediately after preparation of the test sample, direct size reduction (7.2.1.2) may be used. If there is likely to be a delay between size reduction and testing, or if a laboratory sample with an upper particle size of 600 µm is required for other tests, the method described in 7.2.1.3 shall be used. In all cases, the production of an excessive amount of fines shall be avoided.

Check the reduction equipment regularly to ensure that the size distribution of the test sample conforms to that specified in 7.2.1.2. Each of the different coal types analysed by the laboratory shall be checked in this manner. The portion used to check the distribution shall not be reconstituted for dilation testing.

NOTE Low-speed disc mills are suitable for carrying out such reductions.

7.2.1.2 Direct size reduction

Air dry the sample of coal and reduce to an upper particle size of 212 µm to yield a 225 g test sample, as described in ISO 13909-4, avoiding oxidation. The size distribution of the test sample shall comply with the following:

- passing 212 µm test sieve: 100 %;
- passing 125 µm test sieve: 70 % to 60 %;
- pass 63 µm test sieve: 40 % to 30 %.

Commence the determination as soon as possible after the preparation of the test sample.

7.2.1.3 Size reduction via a 600 µm laboratory sample

If coal of maximum particle size 600 µm is required for other analyses, air dry the sample and reduce to an upper particle size of 600 µm, avoiding an excessive amount of fines, to yield a 225 g laboratory sample. For preparation of the dilatometer test pieces crush, a 20 g sub-sample to yield a test sample with a maximum particle size of 212 µm and a size distribution as in 7.2.1.2.

Commence the final reduction as soon as possible after reduction to 600 µm, and the determination as soon as possible after reduction to 212 µm.

7.2.1.4 Storage of sample

If necessary, store the test sample in an inert atmosphere in a sealed glass phial.

7.2.2 Test piece

Place 10,0 g of the test sample (7.2.1) in a small glass beaker; add 1,0 ml of water and mix thoroughly for 2 min to 5 min using a glass stirring rod or metal spatula.

The quantity of the water should be such that the coal just holds together when pressed between the fingers. If it is difficult to obtain a homogenous mixture, this procedure can be modified by adding the coal in increments, mixing after each addition, or by adding a small drop of detergent to the water or by adding a little more water.

Mount the mould (6.1.1), with its plug in position and resting on the base holder, on a firm surface. Place approximately 0,5 g of the moistened sample in the mould and place the ram (6.1.3) on top of the coal. Consolidate the sample by three or four sharp blows from the mallet (6.1.2). Add at least five further increments to the mould and consolidate to fill the barrel of the mould. After the last portion has been inserted and consolidated, compress the test piece further in the screw press (6.1.4) using the load cell (6.1.5) as a support. Continuously apply a load up to a maximum of 15 kN. Release the load as soon as 15 kN is reached.

Remove the base and plug from the mould barrel. Trim the wide end of the test piece free from irregularities. This is conveniently carried out by scraping the end of the test piece using a metal straight-edge of an appropriate width to fit into the recess at the base of the mould barrel. A piece of hacksaw blade suitably ground with a square edge is satisfactory for this purpose.

Expel the formed test piece by suspending the mould on the carrier arm of the press, and screwing the plunger down onto the compressed sample surface. Reduce the length of the test piece to 60 mm ± 0,5 mm by removing material from the narrow end, using, for example, a sharp knife, so that the test piece conforms to the length of a gauge 60 mm long (6.1.6). Two test pieces are required.

Record the mass of the prepared coal pencil. Use this mass to monitor control of factors affecting pencil preparation including addition of water, internal mould dimensions, test sample size distribution, pencil compaction and pencil trimming.¹⁾

7.3 Determination

7.3.1 Number of tests

Carry out the determination in duplicate using test pieces prepared from the same test sample and tested either in two retorts in the same furnace during a single heating cycle, or in the same retort during independent furnace-heating cycles.

1) ACARP Project Report, *Coal Dilatation*, Reference C17054, John J. Kelly, Australia.

7.3.2 Inspection of apparatus

It is essential that the test is carried out with the retorts freely suspended in the furnace and with the dilatometer piston and retort scrupulously clean. Clean as described in 7.3.4 after each test.

7.3.3 Determination of dilatation

Place a test piece (7.2.2) narrow end uppermost in the dilatometer retort and insert the piston into the retort so that it rests on the test piece.

Stabilize the furnace temperature at 300 °C or 30 °C below the expected softening temperature, if this is known.

Insert the retort containing the test piece and piston in the appropriate hole in the furnace block and allow 10 min for the system to regain the stabilized charging temperature. Attach the recording mechanism to the piston during this period and adjust at the zero location.

A slight offset from zero is recommended to facilitate subsequent reading of the chart.

Commence the heating programme and, after a further period of 10 min, start the recorder and index the furnace temperature, as indicated by the temperature recorder, upon the chart.

Conclude the test when no further dilatation has occurred for 5 min or when no dilatation has occurred up to 500 °C. In the 5 min interval following completion of the dilatation process, or at about 500 °C when no dilatation has occurred, make a second indexing of furnace temperature from the temperature recorder on the dilatation chart. If, in the period between the indexed temperatures, the temperature recorder shows a rate of temperature rise which differs from 3 °C/min, i.e. $30\text{ °C} \pm 1\text{ °C}$ in any 10 min interval, the test is not valid.

At the end of the test, detach the recording mechanism and withdraw the retort and piston assembly from the furnace. The piston may be removed from the retort and independently cooled by suspending it in air in a suitable stand.

7.3.4 Cleaning of the furnace, retort and piston

7.3.4.1 Furnace

Check that each retort can be freely suspended from the collar. If not, clean out the holes in the furnace block.

7.3.4.2 Retort and plug

Remove the plug, crush the semi-coke and remove as much of it as possible with the auger (6.8.1). Complete the cleaning of the retort using the wire brush (6.8.2) and a reamer, if necessary. Clean the plug end with very fine emery paper, taking care, as far as is possible, not to abrade the metal. Solvents, such as pyridine or dimethylformamide can be used for this purpose, provided all necessary requirements of national health and safety regulations are observed.

7.3.4.3 Piston

Clean the piston, including its base, with wire wool and very fine emery paper, taking care not to round the edges. Check that the piston slides freely in the cleaned retort; the required tolerances between the piston and the retort are given in 6.6.

8 Expression of results

Report the following five basic parameters obtained from the charts after completion of the test (see Figures 3 and 4);

- softening temperature, θ_1 (3.1), in degrees Celsius;
- temperature of maximum contraction, θ_2 (3.2), in degrees Celsius; resolidification;

- maximum contraction, *c* (3.4), expressed as a percentage;
- maximum dilatation, *d* (3.5), expressed as a percentage.

The dilatation is positive if the final line of maximum dilatation is above the zero line [see Figure 4 a)], and negative if it is below it [see Figure 4 b)]. If the dilatometer curve does not rise after the initial contraction, the dilatation behaviour is noted as “contraction only” [see Figure 4 c)]. If the final trace of the curve is not truly horizontal but slopes downward [see Figure 4 d)], report the contraction as the value observed at 500 °C.

If the maximum dilatation, *d*, is greater than 300 %, report it as $d > 300$.

Average the results of acceptable duplicate determinations (see 9.1), and round to the nearest whole number for temperature, contraction and dilatation.

9 Precision

When the method specified in this International Standard is operated satisfactorily, the numerical values for repeatability (3.6) and reproducibility (3.7) shall not exceed those given in Table 2.

Fractional dilatation tolerances shall be rounded to the next higher whole number.

9.1 Repeatability

The results of duplicate determinations carried out in the same laboratory, by the same operator using the same dilatometer on test pieces prepared from the same test sample, shall not differ by more than the values shown in Table 2.

9.2 Reproducibility

The means of acceptable duplicate determinations carried out in two different laboratories on test pieces prepared from representative samples, which are taken from the same gross sample after reduction to a maximum coal particle size of 2,8 mm, shall not differ by more than the values shown in Table 2. If differences persistently greater than these limits are observed, the need for further investigation into the methods of sampling and testing is indicated.

Table 2 — Precision

Property	Repeatability	Reproducibility
Temperature	7 °C	15 °C
Parameters		
Contraction, <i>c</i>	5 units	8 units
Negative dilatation, <i>d</i>	5 units	8 units
Positive dilatation, <i>d</i>	$5 \left(1 + \frac{d}{100} \right)$	$5 \left(2 + \frac{d}{100} \right)$

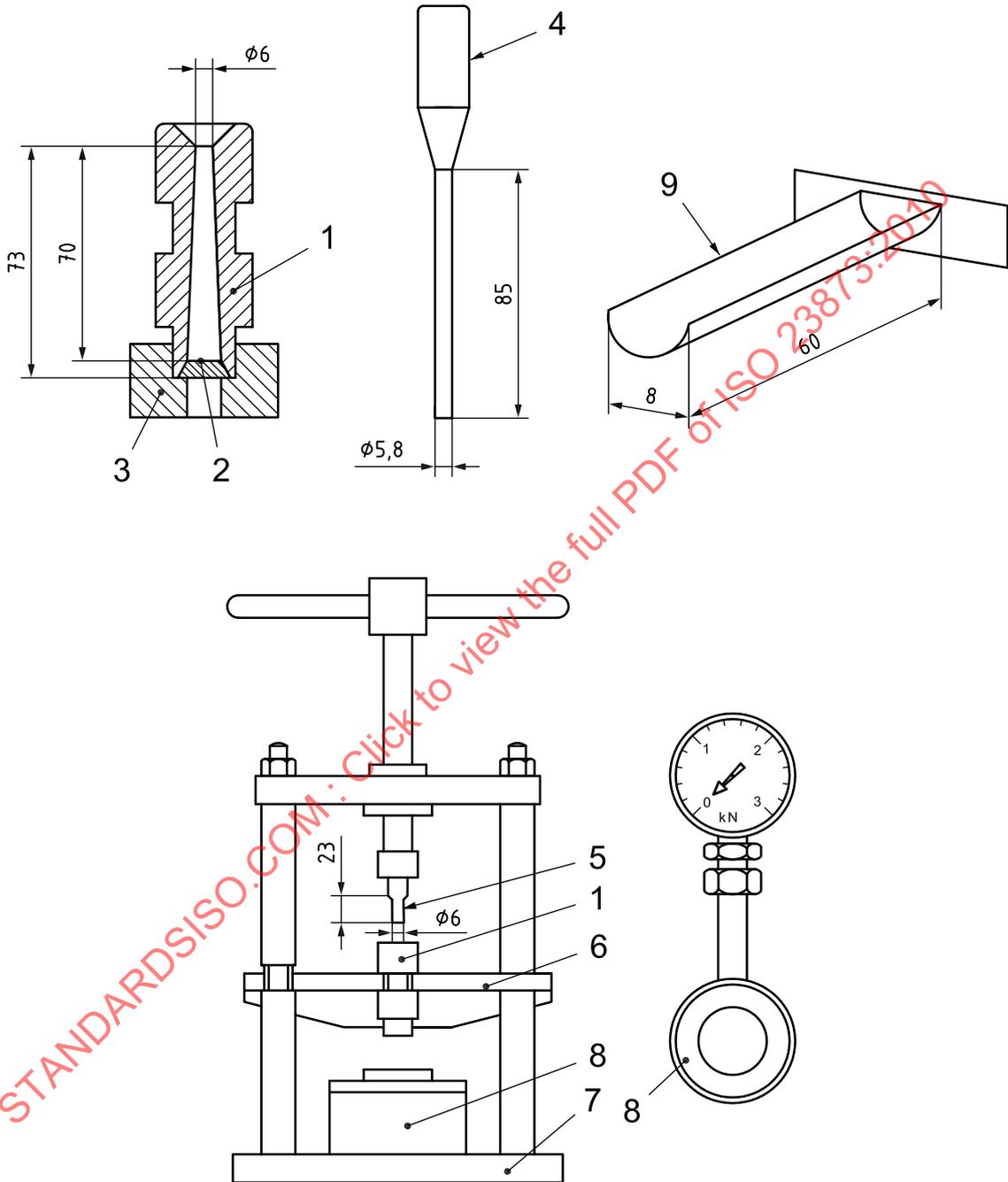
10 Test report

The test report shall contain the following information:

- a) a reference to this International Standard, ISO 23873:2010;
- b) the method used;

- c) the date of the test;
- d) the results and the methods of expression used.

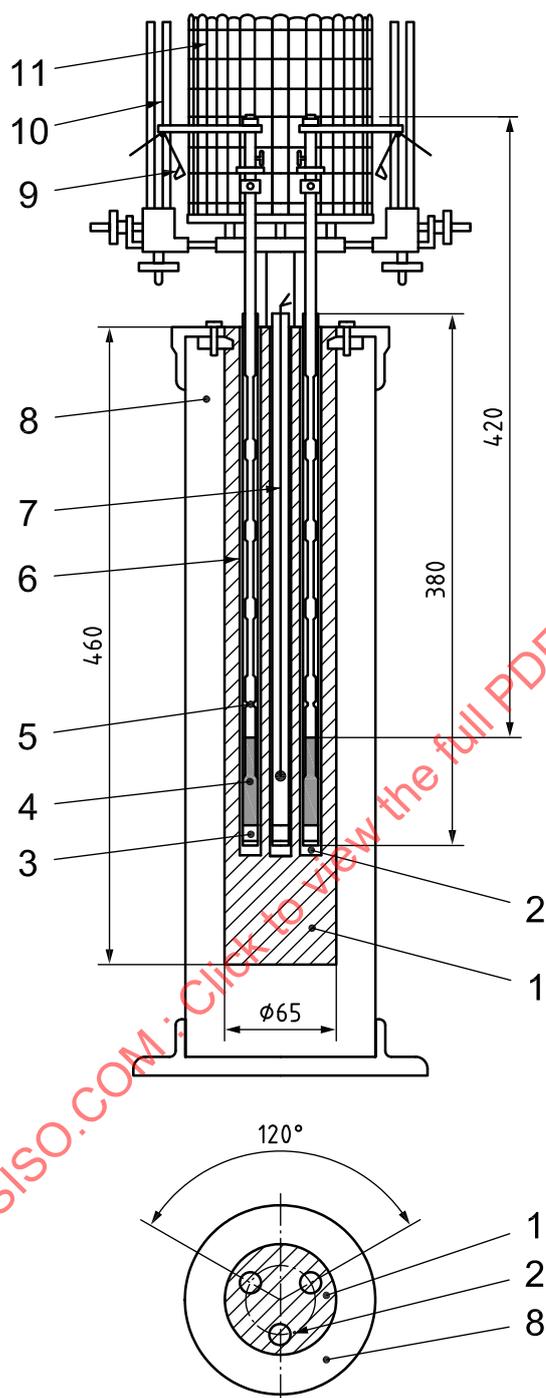
Dimensions in millimetres



Key

- | | | | |
|---|----------------|---|---------------------|
| 1 | mould cylinder | 6 | press mould-support |
| 2 | mould plug | 7 | press base-plate |
| 3 | mould base | 8 | load cell |
| 4 | ram | 9 | pencil gauge |
| 5 | press plunger | | |

Figure 1 — Mould, press and accessories



Key

- | | |
|---|------------------------------|
| 1 heated block | 7 thermocouple |
| 2 holes for retorts ($\phi 15,0 \pm 0,1$) | 8 refractory insulation |
| 3 threaded plug for retort | 9 pen mechanism |
| 4 test piece | 10 recording-drum stand |
| 5 piston | 11 recording drum with chart |
| 6 retort | |

Figure 2 — Typical dilatometer apparatus