INTERNATIONAL STANDARD

ISO 8264

First edition
1989-11-15

Hard coal — Determination of the swelling properties using a dilatometer

Houille — Détermination des propriétés de gonflement à l'aide d'un dilatomètre

INTERNATIONAL ISO

Reference number
ISO 8264 : 1989 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8264 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels.*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Introduction

The Audibert-Arnu dilatometer test was adopted as ISO Recommendation ISO/R 349 : 1963 which was transformed into an International Standard ISO 349 : 1975. When reviewed in 1980, it was generally acknowledged that similar tests, using slightly different equipment and techniques, were used in various countries. One test in widespread use was that which measures the swelling properties of hard coal using the Ruhr dilatometer.

A thorough survey of the construction and operation of this instrument was made between 1973 and 1978 by a working group in the United Kingdom. Eleven laboratories participated in the work, including two which operated the Audibert-Arnu dilatometer as described in ISO 349. In the course of considerable inter-laboratory testing, the results indicated that values of contraction and dilatation found with the Audibert-Arnu dilatometer were higher and lower respectively than those found with the modified Ruhr dilatometer (the version described in this International Standard).

These differences were attributed to the fact that the excess material from the tapered test piece is removed from the wider end in the Audibert version of the dilatometer test and from the narrower end in the Ruhr version. The latter procedure ensures a test piece of greater and more uniform volume.

It is not intended that ISO 349 be withdrawn immediately, however it is suggested that the test be gradually phased out and replaced by that described in this International Standard, a test which has been tried and proven, particularly in the United Kingdom and the Federal Republic of Germany, and shown to be reliable and suitable for measuring the swelling properties of all types of hard coal.

This page intentionally left blank

Hard coal — Determination of the swelling properties using a dilatometer

1 Scope

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 428 : 1983, *Wrought copper-aluminium alloys — Chemical composition and forms of wrought products.*

ISO 683-1 : 1987, *Heat-treatable steels, alloy steels and free-cutting steels — Part 1: Direct-hardening unalloyed and low-alloyed wrought steel in form of different black products.*

ISO 1988 : 1975, *Hard coal — Sampling.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 softening temperature; temperature of initial contraction: The temperature at which the downward movement of the dilatometer piston is 0,5 mm.

NOTE — See θ_1 in figure 3.

3.2 temperature of maximum contraction: The temperature at which the dilatometer piston reaches its lowest point.

NOTE — See θ_2 in figure 3.

3.3 resolidification temperature; temperature of maximum dilatation: The temperature at which the dilatometer piston reaches its highest point.

NOTE — See θ_3 in figure 3.

3.4 maximum contraction: The maximum downward movement of the dilatometer piston, measured from the zero point and expressed as a percentage of the initial test piece length.

NOTE — See *c* in figures 3 and 4.

3.5 maximum dilatation: The maximum upward movement of the dilatometer piston after contraction, measured from the zero point and expressed as a percentage of the initial test piece length.

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

3.6 repeatability: The 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: The 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, 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.

Table 1 — Dimensions of mould for dilatometer test

Dimensions in millimetres

Distance from wide end	Bore (tolerance: − 0,00, + 0,05)
0	7,4
10	7,2
20	7,0
30	6,8
40	6,6
50	6,4
60	6,2
70	6,0

NOTE — Information on suitable gauges for this purpose may be obtained from the British Coal Corporation, Coal Research Establishment, Stoke Orchard, Cheltenham, United Kingdom.

6.1.2 Mallet, plastics head, mass about 200 g.

6.1.3 Ram (d) (see figure 1).

6.1.4 Press (see figure 1).

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

6.1.6 Test piece gauge (i) (see figure 1).

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 K/min. The furnace shall comply with the following operating conditions.

Heat the furnace at a rate of 3 K/min, and measure 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 a retort. When the temperature has reached about 450 °C, measure the temperature over the lower 250 mm of the retort. The difference between the probe temperature and the mean temperature shown at the standard temperature sensing position shall be not more than:

2 K in the lower 120 mm;

5 K from 120 mm to 180 mm;

10 K from 180 mm to 250 mm.

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

A suitable furnace (for heating three retorts) is illustrated in figure 2 and consists of a casting fitted with a base and a top cover. The cover supports in a centre hole a cylindrical block of copper-aluminium alloy (CuAl10Ni5Fe4), complying with ISO 428, as manufactured (i.e. not annealed), 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 K/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.

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 K/min ± 0,05 K/min between 250 °C and 550 °C with a variation of not more than ± 1 K per 30 K rise in any 10 min period, with a precision of ± 1 K.

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, type 28 Mn6 complying with ISO 683-1, fitted with a gas-tight 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 mm ± 0,05 mm and the external diameter shall be 14,5 mm ± 0,1 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 mm, discard the retort.

The piston is machined from rod made of steel, type C 55 complying with ISO 683-1. Adjust the combined mass of the piston and pen assembly to 150 g ± 5 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 mm ± 0,05 mm on manufacture. If this difference exceeds 0,275 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, approximately 7,8 mm diameter and stem length 400 mm.

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

NOTE — A wear-resistant steel reamer, 7,95 mm diameter and stem length 400 mm may also be used.

7 Procedure

7.1 Temperature calibration

Carry out the following operations for each position in the furnace, other than the position used for the temperature sensor.

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 [5.2a)] 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 (see 7.2.2) by a prepared graphite pencil. Repeat this procedure using a zinc ball [5.2b)].

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.

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

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

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.

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 1988, 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 %

passing 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 a maximum particle size of 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 g of the test sample (7.2.1) in a small glass beaker; add approximately 1 ml of water and mix thoroughly for 2 min to 5 min using a glass stirring rod. The quantity of the water shall be such that the coal just holds together when pressed between the fingers.

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) by applying continuously a load 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 on to 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 (see figure 1).

Two test pieces are required.

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.

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 retor 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 30 °C below the expected softening temperature or, if this is unknown, at the appropriate temperature given in table 2 or, if the volatile matter content is unknown, at 265 °C.

Table 2 — Furnace temperature for dilatometer test

Volatile matter content of coal (dry, ash-free basis) % (m/m)	Stabilized charging temperature °C
38,1 and above	265
28,1 to 38,0	295
18,1 to 28,0	325
18,0 and below	355

Insert the retort containing the test piece and piston in the appropriate hole in the furnace 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.

NOTE — 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 K/min, i.e. 30 °C ± 1 °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 in air in a suitable stand.

7.3.4 Cleaning of the furnace, retort and piston

Check and clean the equipment at the end of each test as follows.

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 temperature, θ_3 (3.3), in degrees Celsius;

— maximum contraction, c (3.4), as a percentage;

— maximum dilatation, d (3.5), as a percentage.

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

If the maximum dilatation, d, is greater than 300 %, report 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 3.

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 3.

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 3. If differences persistently greater than these limits are observed, the need for further investigation into the methods of sampling and testing is indicated.

Table 3 — Precision of dilatometer test

Property	Repeatability	Reproducibility
Temperature parameters	7 K	15 K
Contraction, c	5 units	8 units
Negative dilatation, d	5 units	8 units
Positive dilatation, d	$5\left(1 + \frac{d}{100}\right)$	$5\left(2 + \frac{d}{100}\right)$

10 Test report

The test report shall contain the following information:

a) the reference of the method used;

b) the results and the methods of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.