SLOVENSKI STANDARD
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Ļrni premog - Ugotavljanje lastnosti nabrekanja z dilatometrom

Hard coal -- Determination of the swelling properties using a dilatometer

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

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INTERNATIONAL STANDARD

ISO 8264

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Hard coal — Determination of the swelling properties using a dilatometer

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

Reference number
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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.*

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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.

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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.