INTERNATIONAL STANDARD

ISO 349

Second edition
2020-01

Hard coal — Audibert-Arnu dilatometer test

Houille — Essai au dilatomètre Audibert-Arnu

Reference number
ISO 349:2020(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 349:1975), which has been technically revised. The main changes are the following:

— Reformatting of the figures in the main document and annexes.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The Audibert-Arnu dilatometer test is used to determine the coking properties of hard coal or hard coal blends on the laboratory scale.

In principle, the test is not designed, nor can it be used, to indicate the pressures exerted by hard coals on the walls of industrial carbonization ovens.

Hard coal — Audibert-Arnu dilatometer test

1 Scope

This document specifies a method for determining the swelling properties of hard coal when heated under standard conditions in a dilatometer.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

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 1 to entry: See temperature T_1 in Figure 1.

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

Note 1 to entry: See temperature T_2 in Figure 1.

3.3
temperature of maximum dilatation
temperature at which the dilatometer piston reaches its highest point

Note 1 to entry: See temperature T_3 in Figure 1.

3.4
maximum contraction
maximum downward movement of the dilatometer piston, measured from the zero point

Note 1 to entry: Maximum contraction is expressed as a percentage of the initial test-piece length.

Note 2 to entry: See *a* in Figure 1.

3.5
maximum dilatation
maximum upward movement of the dilatometer piston after contraction, measured from the zero point

Note 1 to entry: Maximum dilatation is expressed as a percentage of the initial test-piece length.

Note 2 to entry: See *b* in Figure 1. The value can be either positive or negative.

3.6
repeatability
r
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

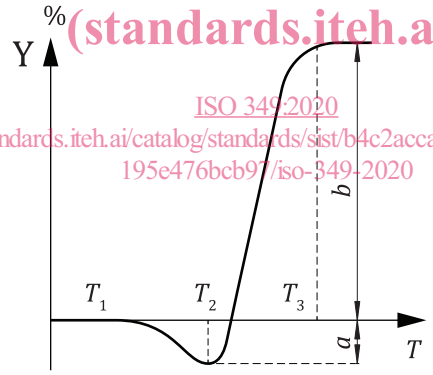
3.7
reproducibility
R
maximum acceptable difference between the means of two determinations which are carried out in each of two laboratories, on representative portions taken from the same gross sample, after the last stage of sample preparation

4 Principle

A pencil made of powdered (pulverised) coal formed under pressure is inserted into a precisely calibrated narrow tube and topped by a calibrated steel rod (piston) which slides in the bore of the tube.

The whole assembly including the tube, pencil and piston is heated at a constant and definite rate.

Upon taking regular readings of the displacement of the piston as a function of the temperature and expressing the displacements observed as percentages of the original length of the pencil, a curve of the type shown in Figure 1 can be plotted.



Key

T_1 temperature at which the piston has moved down 0,5 mm (or one division, if the scale is calibrated in percentage of the standard length of pencil) softening temperature or initial contraction temperature

T_2 temperature at which the piston reaches its lowest point: temperature of maximum contraction

T_3 temperature at which the piston reaches its highest point: temperature of maximum dilatation

Y piston displacement (or dilatation) in %

a maximum contraction of length of pencil in %.

b maximum dilatation of length of pencil, %.

Figure 1 — Piston displacement with temperature

Different types of dilatation curves are represented in Annex A.

NOTE The principal factors capable of distorting the results of this empirical test are the following:

a) Deterioration/oxidation of the coal, consequent on bad storage or faulty drying;

b) Deviation from the tolerances of

1) the internal dimensions of the dilatometer tube,

2) the clearance between tube and piston,

3) the mass of the piston,

4) the dimensions of the mould;

c) Deviation from the specified mean rate and regularity of heating;

d) Deviation from the specification for the preparation of the sample in respect of maximum particle size, or for the pencil in respect of its length after tamping.

5 Apparatus

5.1 Apparatus for preparing the coal pencil

5.1.1 Mould, polished internally, with accessories; see Figure B.1.

5.1.2 Gauge.

5.1.3 Ram, of which Figure B.2 shows an example.

5.1.4 Press, of which Figure B.3 shows an example.

5.2 Dilatometer and accessories

5.2.1 Dilatometer tubes and pistons, see Figure B.4.

5.3 Other apparatus

5.3.1 Electric furnace, example shown in Figure B.5, consisting of a block of metal resistant to oxidation and of sufficiently high melting point. A suggested material is aluminium bronze. The metal block is pierced by at least two holes of 15 mm diameter by 350 mm deep to take the dilatometer tubes and one hole 320 mm deep to take a temperature-measuring device, which intends to keep the thermojunction of the thermocouple in the middle position of the inserted coal pencil with 60 mm. The block is heated by a metallic winding, suitably insulated. Control gear permits the use of any selected rate of heating up to 5 °C/min over a temperature range of 300 °C to 550 °C.

The furnace shall be constructed so that the temperature conditions are uniform in the dilatometer tubes placed in their normal position in each hole.

To verify this, heat the furnace at a rate of 5 °C/min. When the temperature reaches about 450 °C, make an exploration of the lower 180 mm of a dilatometer tube placed in the furnace, by comparing the readings on the normal temperature-measuring device and a thermometric probe placed in the tube. The difference between the probe temperatures and the mean temperature shown by the normal temperature-measuring device shall be less than

— ±2 °C in the lower 120 mm,

— ±5 °C from 120 to 180 mm.

This verification is not to be confused with the later calibration of the temperature-measuring device; it is intended to measure only the temperature variation along the tube.

The furnace shall be equipped with an adjustable scale for each hole. The scale shall be preferably engraved on a mirror in front of which the indicator pointer of the piston moves. It shall be at least

180 mm in length and calibrated in millimetres or in percentages of the standard length of the pencil (60 mm ± 0,25 mm, see 5.1).

The apparatus can also be equipped with an automatic heating regulator and a device for the automatic registration of the curve.

5.3.2 Temperature-measuring device, consisting of a thermocouple accurate to 0,1 °C.

5.3.3 Cleaning equipment, consisting of the following:

5.3.3.1 Auger, diameter approximately 7,8 mm;

5.3.3.2 Brass wire brush, the diameter of which shall slightly exceed 8 mm;

5.3.3.3 Reamer, consisting of a steel bar of semi-circular section of diameter 7,95 mm.

The total length of each of the cleaning components shall be 400 mm.

5.4 Calibration of apparatus

Calibrate the apparatus by comparing the temperature in a dilatometer tube in each hole with the temperature indicated by the temperature-measuring device in its normal position. Carry out the calibration at the desired rate of heating by using a thermocouple with wires of diameter approximately 0,6 mm, the thermojunction touching the wall of the tube 30 mm above the bottom. Correct the temperatures read during the test by the differences found during this calibration.

5.5 Inspection

5.5.1 Dilatometer

In order to inspect the wear of the tube and piston after a hundred determinations have been carried out in one tube, compare the results of the next four determinations using that tube with those obtained in a new tube. This comparison will thus be made successively on four coals.

Divide the difference in percentage dilatation between the two tubes by the “relative length” of the dilated pencil obtained with the new tube; the “relative length” is here expressed as the ratio of the length of the dilated pencil to its original length.

Average the figures so obtained from the four coals. If the average is greater than 3,5, irrespective of sign, discard the old tube (see Annex B). If the tube is still satisfactory, repeat this comparison after every subsequent 25 tests.

5.5.2 Mould

Check the wear of the mould periodically with the gauge, which can also be used to check new moulds.

If, when the gauge is inserted in the larger orifice of the mould,

a) two lines can be seen on the gauge, the mould is too small and shall be reamed out;

b) one line can be seen, the mould is satisfactory;

c) no line can be seen, the mould is worn and shall be replaced.

6 Preparation of sample

As certain types of hard coal are very susceptible to oxidation, it is necessary to minimize the contact with air after reducing the gross sample.

As a special precaution, therefore, store the test sample after reduction in an oxygen-free nitrogen atmosphere.

Care shall be taken to ensure that the test sample taken is truly representative, meaning that it is prepared and divided according to ISO 13909-4 or ISO 18283.

A suitable relationship between the mass of the test sample and the maximum particle size is shown in Table 1. Data in this table is taken from ISO 18283:2006, Table 3.

Table 1 — Relation between mass of test sample and maximum particle size

Maximum particle size mm	Minimum mass of test sample g
5	2 250
4	1 500
3	800
2	250
1,5	175

Reduce the maximum particle size to 1,5 mm. Mix and take a part sample of 50 g to 100 g. Crush to pass a 0,212 mm mesh sieve. Both crushing operations shall be controlled so as to produce the minimum of fines (see NOTE). Mix again and carry out the determination on a sample weighing 10,0 g. Moisten this sample with 1 ml of water and mix carefully to prepare coal-water paste. Too intensive mixing is liable to cause difficulty when the pencil is removed from the mould. For the same reason, it is essential that the preparation of the pencil shall be carried out without interruption.

Too fine grinding of the coal affects the result of the determination. The sample shall be crushed to obtain the following size analysis: through 0,2 mm: 100%; through 0,1 mm: 85% to 70%; through 0,06 mm: 70% to 55%.

7 Procedure

7.1 Preparation of the coal pencil

Place the mould on its support with the larger orifice upwards and set the funnel on the mould. Place the coal in the funnel and lightly tamp into the mould without moving the funnel, by means of a tamping pin. Place the mould assembly under the ram in order to tamp the sample by dropping the plunger three or four times until the mass of coal ceases to yield. Repeat this three or four times until the mould is filled.

In order to remove the coal pencil from the mould, remove the support and the funnel. Place the ejector guide at the end of the mould corresponding to the smaller diameter of the pencil. Place the guide tube at the other end of the mould and the receptacle in the guide tube. Then insert the ejector piston in the guide and push the coal pencil onto the receptacle by means of the press (see NOTE).

Then adjust the length of the pencil to 60 mm ± 0,25 mm by cutting away as much as necessary of the thick end with a fine blade.

NOTE Particularly when dealing with coals which are difficult to remove from the mould, it is recommended that the ejector piston be removed from time to time and cleaned, the inner surface of the mould being cleaned at the same time.