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Hard ~~Coal~~coal — Determination of plastic properties — Constant-torque Gieseler plastometer method

Houille — Détermination des propriétés plastiques — Méthode du plastomètre Gieseler à couple constant

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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.

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 document 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).

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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, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 10329:2017), which has been technically revised.

The main changes are as follows:

- ~~Updated~~ updated the scope of this document, it is only applicable to ~~Hard Coal~~ hard coal;
- ~~Clarified~~ clarified reporting requirements for the various ranges of dd/min;
- ~~editorial~~ editorial revisions.

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.

Hard ~~Coal~~coal — Determination of plastic properties — Constant-torque Gieseler plastometer method

1 Scope

This document specifies a method for obtaining a relative measure of the plastic behaviour of hard coal when heated under prescribed conditions. The method is used to obtain values of the plastic properties of coals and blends used in carbonization and in other situations where determination of plastic behaviour of coals is of practical importance.

NOTE The empirical nature of this test requires proper equipment calibration to produce fluidity readings which are a true indication of the relative plastic behaviour of the coal.

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 18283, *Coal and coke — Manual sampling*

ISO 13909-1, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

ISO 13909-3, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*

ISO 13909-4:2016, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain ~~terminological~~terminology databases for use in standardization at the following addresses:

— IEC Electropedia: available at <https://www.electropedia.org/>

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— ~~3.1~~ IEC Electropedia: available at <https://www.electropedia.org/>

3.1

dial division per minute

dd/min

measure of stirrer rotation rate, as used in the constant-torque Gieseler plastometer method

Note 1 to entry: — There are 100 dial divisions for each full 360° rotation of the stirrer. The fluidity result is expressed as total dial divisions turned by the stirrer in a 1 min time period, i.e. dd/min.

3.2 3.2**initial softening temperature**

temperature at which dial movement or electronic readout indicates a stirring shaft movement of one *dial division per minute* ~~(3.1)~~(3.1), with continued indication of movement of at least 1 dd/min thereafter

3.3 3.3**maximum fluidity temperature**

temperature at which stirring shaft rotation reaches the maximum rate

3.4 3.4**plastic range**

difference between the initial softening temperature and the solidification temperature

3.5 3.5**final fluidity temperature**

temperature at which the last 1 dd/min stirrer rotation rate is reached

3.6 3.6**solidification temperature**

temperature at which the stirring shaft stops

3.7 3.7**maximum fluidity**

maximum rate of rotation for the stirring shaft in *dial divisions per minute* ~~(3.1)~~(3.1)

3.8 3.8**jamming**

swelling up of coal into the retort tube during the test

Note-1-to-entry:-Jamming can produce a lower fluidity result than expected and can only be noted after visual inspection of the disassembled crucible and retort at the conclusion of the test.

3.9 3.9**breaking**

free spinning behaviour of coal, either by rotating at maximum motor speed or by abrupt changes in rotation

Note-1-to-entry:-Breaking occurs as a result of a molten ball of coal forming around the base of the stirrer, and makes reporting of the true *maximum fluidity* ~~(3.7)~~(3.7) of the coal difficult.

4 Principle

Measurements of the plastic properties of coals are made by applying a constant torque to a stirrer placed in a crucible into which the coal is charged. The crucible is immersed in a bath and the temperature increased uniformly. The rotation of the stirrer is recorded in relation to increase in temperature.

5 Apparatus

5.1 5.1 — Gieseler plastometer retort, composed of the following component parts (see **Figure 1**).**Figure 1**.

5.1.1 5.1.1 — Retort crucible, cylindrical, with $(21,4 \pm 0,1)$ mm inside diameter, and $(35,0 \pm 0,3)$ mm in depth with exterior threads for joining the crucible to the barrel.

The crucible shall have a $(2,38 \pm 0,02)$ mm diameter notch with an included angle of 70° in the centre of its inside base to serve as a seat for the stirrer.

5.1.2 ~~5.1.2~~—Retort crucible cover, with interior threads for joining the crucible cover to the crucible and exterior threads for joining the crucible cover to the barrel.

The inside diameter of the hole which accommodates the stirrer shall be $(9,5 \pm 0,1)$ mm.

5.1.3 ~~5.1.3~~—Guide sleeve, provided near the upper end of the stirrer to guide the latter within the barrel with a clearance of between 0,05 mm and 0,10 mm.

5.1.4 ~~5.1.4~~—Gas exit hole, provided on the barrel to afford an exit for the volatile products during a test, placed, for example, at the midpoint of the barrel; as an option, a tube may be fitted if desired.

5.1.5 ~~5.1.5~~—Barrel, $(121,0 \pm 2,5)$ mm long, having an inside diameter of $(9,5 \pm 0,1)$ mm.

The top end of the barrel shall be 12,7 mm in inside diameter to a depth sufficient to allow the fitting of a guide sleeve through which the axle of the stirrer passes when the apparatus is assembled.

5.1.6 ~~5.1.6~~—Steel stirrer, provided with a straight shaft $(3,95 \pm 0,05)$ mm in diameter and equipped with four rabble arms.

The lower end of the stirrer shall be tapered to a point having an included angle of 60° . The rabble arms on the stirrer shall be $(1,60 \pm 0,05)$ mm in diameter, $(6,4 \pm 0,05)$ mm in length, and shall be placed ~~so as to be~~ perpendicular to the shaft at 90° intervals around the shaft and $(3,2 \pm 0,05)$ mm apart centre to centre along the shaft. The middle two rabble arms shall be set at 180° to each other, and likewise, the remaining two arms at 180° to each other. The lowest rabble arm shall be set to give $(1,60 \pm 0,05)$ mm clearance between it and the bottom of the crucible when the stirrer is in place. The upper end of the stirrer shall be cut to fit into a slot on the lower end of the axle in the plastometer head.

The rabble arms shall be checked before each test to ensure that they are clean and free from distortion or other visual damage. It is essential that the dimensions of the rabble arms on the stirrer meet the requirements in this clause and in ~~Figure 2~~Figure 2 and it is important that these dimensions are checked on a regular basis, e.g. every 50 determinations. If after use, it is found by accurate measurement that any of the dimensions are outside the specified limits, the rabble arms shall be replaced or the stirrer discarded.

5.2 ~~5.2~~—Plastometer head

The plastometer head, shown schematically in ~~Figure 3~~Figure 3, shall consist of a fixed-speed motor (approximately 300 r/min) connected directly to a magnetic clutch or hysteresis brake capable of adjustment over a reasonable range on either side of a torque value of 101,6 g·cm (9,66 Nm). A dial drum, attached to the clutch brake output shaft, shall be graduated into 100 divisions for 360° . Each complete drum revolution or 100 dial divisions shall be recorded on a counter actuated by an electric eye or other suitable method. As an alternative, an electronic device capable of measuring rotation rates between 0,01 r/min and 300 r/min may be attached to the clutch or brake output shaft. Using this latter technique, rotation rates can be converted directly to dial divisions per minute (dd/min) and can be displayed or recorded once a minute on a suitable electronic readout or printer.

Note 1 to entry: NOTE Care is needed in the maintenance and lubrication of all bearings, particularly after the instrument has been used with a coal high in volatile matter. Using bearings with a closed race is undesirable because of the increased friction of the closed race compared to bearings with an open race. When using open race bearings, clean and replace on a regular basis.

5.3 ~~5.3~~—Electric furnace

An electrically heated furnace shall be used (see ~~Figure 4~~Figure 4) with suitable controls so that a heating rate of $(3,0 \pm 0,1)$ °C/min can be maintained over a temperature range of 300 °C to 550 °C. The furnace shall

contain a molten solder bath of approximately 50 % lead and 50 % tin composition. The temperature in the bath shall be measured with a suitable thermocouple in a protection tube of approximately 6 mm in outside diameter immersed in the bath so that the tube touches the outside wall of the crucible and the hot junction of the thermocouple is at the same height as the centre of the coal charge. A stirrer shall be used to agitate the solder.

5.4 ~~5.4~~ Loading device

The loading device shall be provided to pack the coal uniformly in the crucible under a total load of 10 kg and designed in such a manner that, after compression, the crucible and its contents can be removed easily from the device without disturbing the contents. A suitable device, such as shown in ~~Figure 5~~, ~~Figure 5~~, has a static mass of 9 kg together with a drop mass of 1 kg which is dropped 12 times from a height of 115 mm.

~~Note 1 to entry:~~ **NOTE** The static mass of 9 kg is the combined mass of the cage, packing head, dropping mass shaft and the static mass.

6 Calibration

Torque shall be checked with the string and pulley method shown in ~~Figure 6~~, ~~Figure 6~~. In the string and pulley method, a pulley is screwed onto the spindle and a string attached to this pulley is placed over a second vertical pulley, with the required mass attached. A 25,4 mm (1 in) radius pulley and a 40 g mass have been used for that purpose. With the plastometer motor turned on, the brake or clutch suspension, or the torque gauge or transducer, reads $(101,6 \pm 5,1)$ g·cm $[(40,0 \pm 2,0)$ g·in]. All instruments shall be checked in this manner as required, at a predetermined frequency based on the laboratory's experimentation on stability or torque setting.

Alternatively, torque can be checked with a suitable gauge or transducer provided it has been validated against the string and pulley method. If this method is used, the torque applied to the shaft is checked as a minimum at two positions at 90° to each other to check that the drive shaft alignment and bearings running are correct. If the torque values are different, this indicates a problem that shall be repaired, otherwise, lower fluidity values will be recorded.

~~Note 1 to entry:~~ **NOTE** A pulley/weight assembly is the only one capable of reliably evaluating the torque over a complete 360° rotation.

7 Sample

A representative gross sample of coal shall be collected in accordance with ISO 18283, ISO 13909--1, ISO 13909--2 and ISO 13909--3. Approximately 4 kg of coal crushed to pass a 4,75 mm sieve shall constitute the laboratory sample. This sample shall be representative of the batch being tested. Spread the laboratory sample on a tray and allow it to equilibrate with the laboratory atmosphere. Drying shall not be continued beyond this point so that the plastic properties of the coal are not altered by oxidation. The drying temperature shall not exceed 40 °C. After air drying to equilibration, divide the sample representatively to about 500 g. Representatively subsample one quarter of this 500 g portion, then crush this one quarter in successive steps to pass a 425 µm sieve in such a manner as to minimize the production of fines. This is done by alternatively screening and crushing the oversized material until no oversized material remains (the stage crushing process shall not violate Table 1 of ISO 13909--4:2016). The size reduction process shall aim at keeping the fines of less than 212 µm in diameter to less than 50 % of the final sample.

Thoroughly mix the sample, preferably by mechanical means, and withdraw not less than 5 g of this sample by increments. Test the coal for plastic properties by duplicate determinations within 8 h after preparing the