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

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

Reference number
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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 10329 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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

1 Scope

This International Standard specifies a method for obtaining a relative measure of the plastic behaviour of coal when heated under prescribed conditions. The method may be 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 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 18283, *Hard 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*

3 Terms and definitions

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

3.1
dial division per minute
measure of stirrer rotation rate, as used in this method

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

3.3
maximum fluidity temperature
temperature at which stirring shaft rotation reaches the maximum rate

3.4
plastic range
difference between the initial softening temperature and the solidification temperature

3.5
final fluidity temperature
temperature at which the last 1 dd/min stirrer rotation rate is reached

3.6
solidification temperature
temperature at which the stirring shaft stops

3.7
maximum fluidity
maximum rate of rotation for the stirring shaft in dial divisions per minute

3.8
jamming
swelling up of coal into the retort tube during the test, which may 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
breaking
free spinning behaviour of coal, either by rotating at maximum motor speed or by abrupt changes in rotation, which occurs as a result of a molten ball of coal forming around the base of the stirrer, and which makes reporting of the true maximum fluidity 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 Gieseler plastometer retort, composed of the following component parts (see Figure 1).

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

The crucible shall have a (2,38 ± 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 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 ± 0,1) mm.

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 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; a tube may be fitted if desired.

5.1.5 Barrel, (121,0 ± 2,5) mm long, having an inside diameter of (9,5 ± 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 Steel stirrer, provided with a straight shaft (3,95 ± 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 (1,6 ± 0,05) mm in diameter, (6,4 ± 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 ± 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 (1,6 ± 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 should 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, 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 should be replaced or the stirrer discarded.

5.2 Plastometer head.

The plastometer head, shown schematically in Figure 3, shall consist of a fixed-speed motor (approximately 300 r/min to 500 r/min) connected directly to a magnetic clutch or hysteresis brake capable of adjustment over a reasonable range 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 degrees. 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 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 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 due to 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 Electric Furnace.

An electrically heated furnace shall be used (see Figure 4) with suitable controls so that a heating rate of (3,0 ± 0,1) °C/min, on an overall basis, with not more than (3,0 ± 1,0) °C for any given minute, can be maintained over a temperature range from 300 °C to 550 °C with a (15 ± 1) °C rise over any given 5 min period. The furnace shall contain a molten solder bath of approximately 50 percent lead and 50 percent 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 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, has a static weight of 9 kg together with a drop weight of 1 kg which is dropped twelve times from a height of 115 mm.

NOTE The static weight 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. 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 weight attached. A 25,4 mm (1 in) radius pulley and a 40 g weight 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 ± 5,1) g·cm [(40,0 ± 2,0) g·in]. All instruments should 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 is correct. If the torque values are different, this indicates a problem that must be repaired, otherwise lower fluidity values will be recorded.

NOTE A pulley/weight assembly is the only one capable of reliably evaluating the torque over a complete 360° rotation.

7 Sample

Collect a representative gross sample of coal 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 should 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 from 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 oversize material until no oversize material remains. The size reduction process should 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 sample passing a 425 µm sieve. Avoid delays in performing the analysis so far as possible because the plastic properties of coal may be significantly affected by deterioration and oxidation. Refrigeration or inert gases should be used to minimize oxidation of prepared samples.

8 Procedure

8.1 Adjust the hysteresis brake to (101,6 ± 5,0) g·cm.

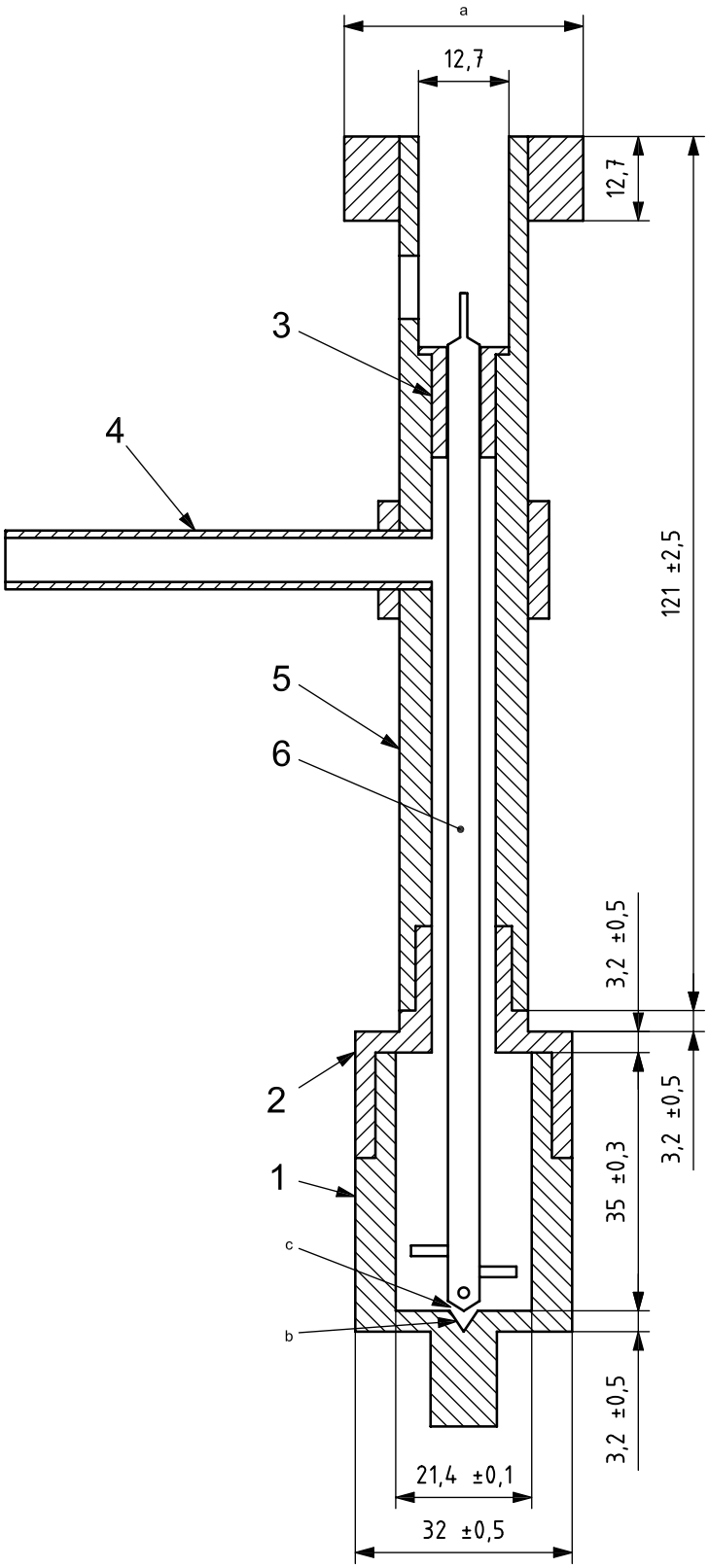
8.2 Mount the crucible, with the stirrer in place, in the loading device shown in Figure 5 but with the 10 kg weight removed and packing tamper raised. Charge 5,0 g of the prepared sample of coal into the crucible. Rotate the stirrer carefully with the fingers in order to fill the voids under the rabble arms. Lower the packing tamper weight, making sure that the total weight is transmitted to the coal charge. Compress the charge by dropping the 1 kg weight twelve times from a height of 115 mm.

NOTE For coals that are difficult to pack, it is permissible to add 1 to 3 drops of toluene or lubricant oil on the shaft of the stirrer near the surface of the coal before the weight is added. Very dry coal can benefit from the addition of several drops of water and thorough mixing prior to packing.

8.3 Remove the crucible from the loading device, screw on the crucible cover taking extreme care not to disturb the position of the stirrer in the coal. Screw the crucible and stirrer into the retort assembly. The stirrer should be centred in the guide sleeve. Screw the retort assembly onto the plastometer head, making sure that the top end of the stirrer fits into the slotted end of the axle. To prevent binding, ensure that there is a space of about 1 mm between the top of the stirrer and the top of the slot.

8.4 Lower the assembled apparatus into the furnace until the bottom of the crucible plug is immersed to a depth of 75 mm in the molten solder bath, which is maintained at a temperature of 300 °C. Place the thermocouple in the bath in accordance with Clause 5.3. The heating control shall be such that the bath will re-attain the initial temperature in (10 ± 2) min after immersion of the retort. Thereafter, heat the retort at a rate of (3,0 ± 0,1) °C/min on the overall basis. When the movement of the drum or readout from the electronic sensor has reached 1 dd/min, take readings of temperature and dial movement at 1 min intervals. Continue readings until the dial shows no further movement.

Dimensions in millimetres



Key

1	crucible	4	gas exit tube	a	Diameter to fit head coupling.
2	crucible cover	5	barrel	b	70° included angle.
3	guide sleeve	6	rabble arm stirrer	c	1,6 mm clearance lower arm to crucible.

Figure 1 — Retort assembly

Dimensions in millimetres

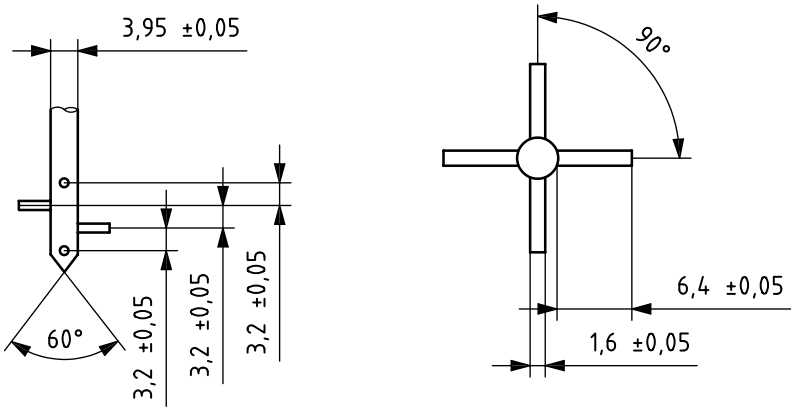
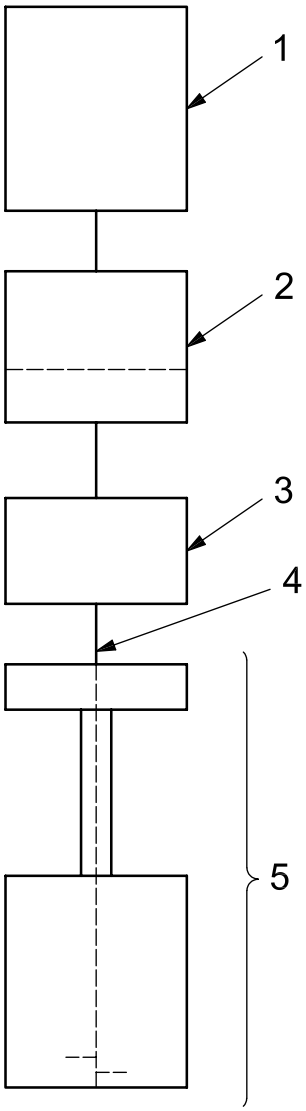


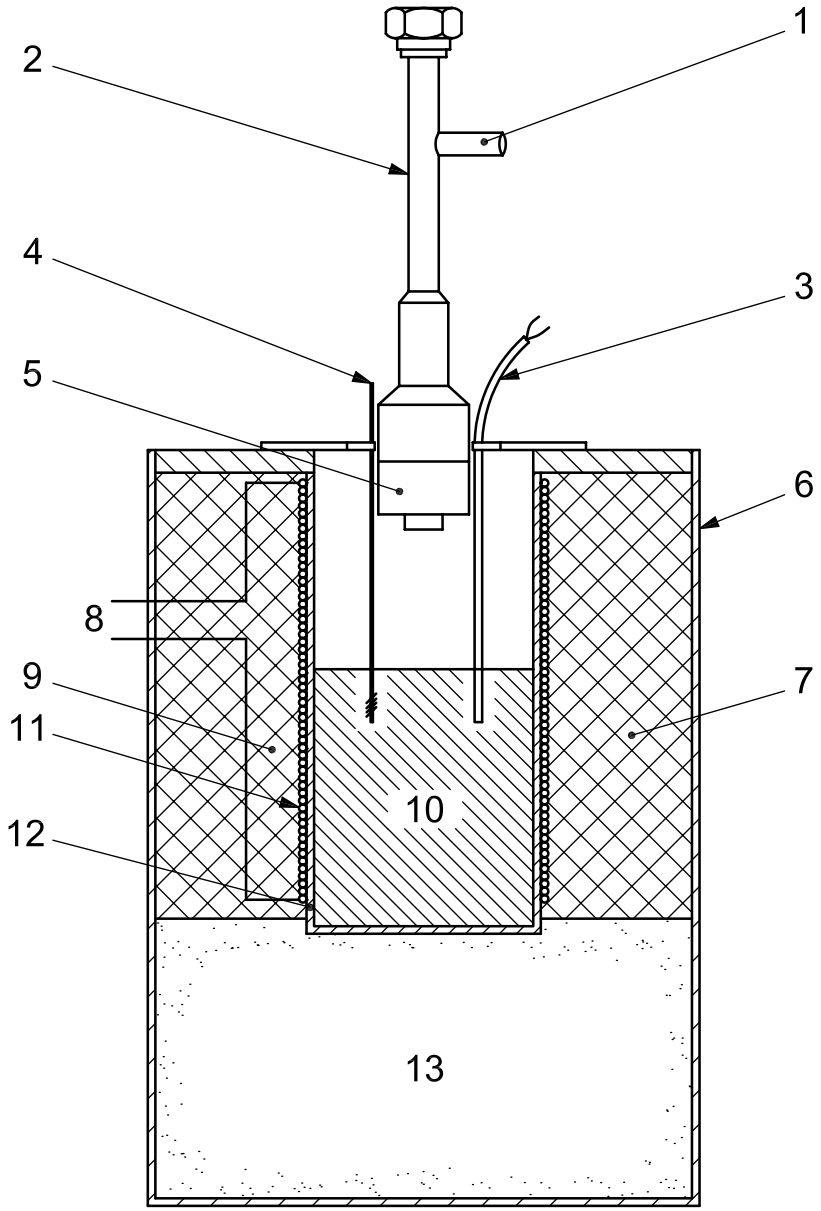
Figure 2 — Stirrer rabble arms



Key

1	motor	3	dial drum or electronic sensor	5	retort assembly (see Figure 1)
2	clutch or brake	4	spindle		

Figure 3 — Schematic representation of plastometer head

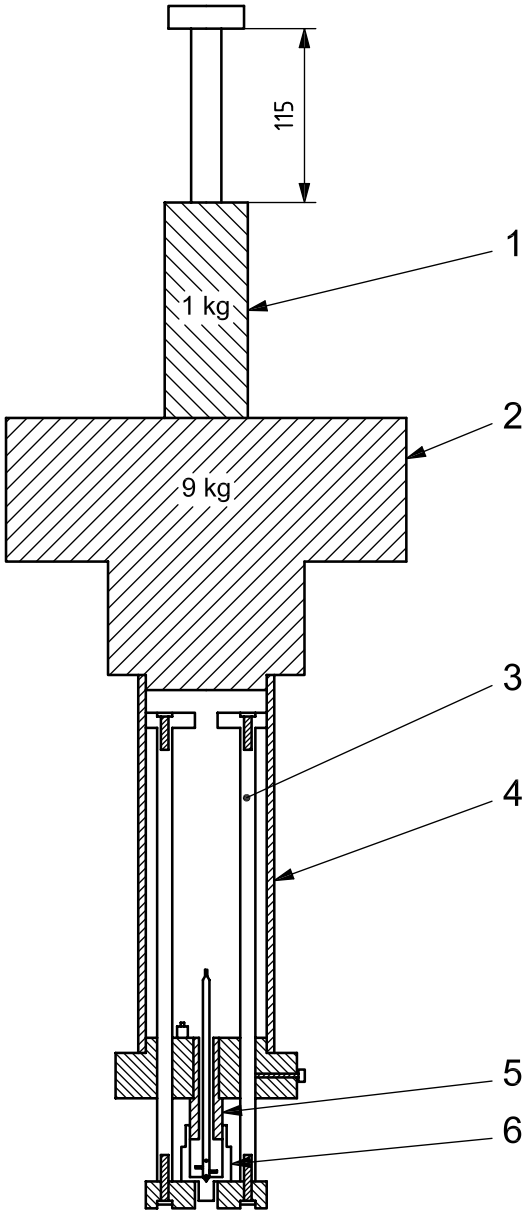


Key

1 gas exhaust tube
2 retort barrel
3 sheathed thermocouple
4 bath mixer
5 retort crucible
6 housing
7 insulation
8 exit to heating rate controller
9 heater winding
10 solder bath
11 mica insulating layer
12 low-carbon (mild) steel bath vessel
13 refractory block

Figure 4 — Schematic view of retort and furnace

Dimensions in millimetres

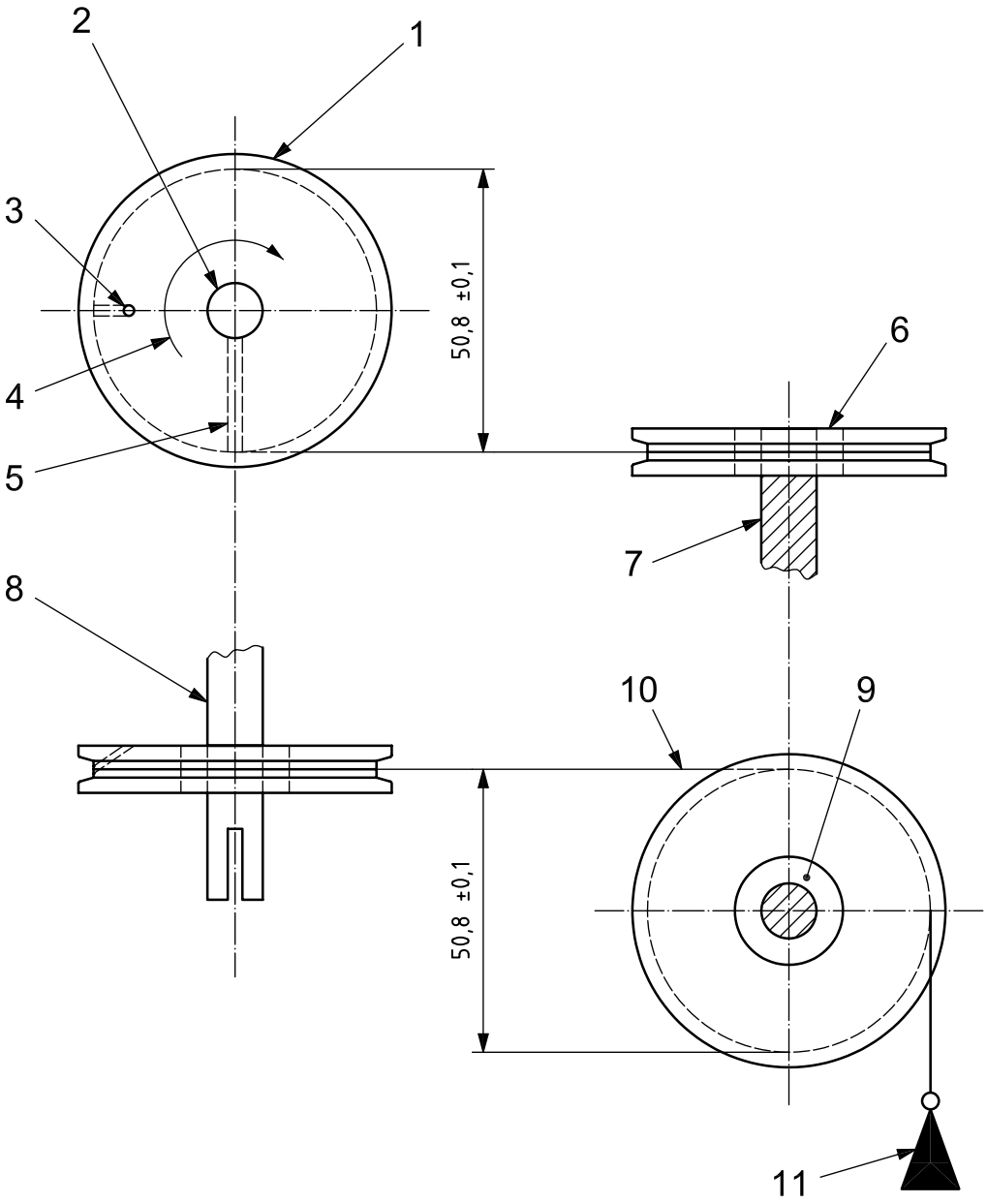


Key

1 dropped weight (1 kg)
2 static weight (9 kg)
3 slide rod
4 cage
5 packing head to fit inside crucible (clearance 0,5 mm)
6 crucible with charge and stirrer

Figure 5 — Typical loading device

Dimensions in millimetres



Key

1 drive pulley
2 centre hole to fit slotted axle
3 attachment point for monofilament pulley line
4 direction of rotation
5 set screw
6 transfer pulley (aluminium)
7 transfer pulley support rod
8 slotted axle
9 sealed low-friction bearing
10 3 kg test monofilament pulley line
11 attached weight (40 ± 0,1) g

[a] All angles between the monofilament line and the centre-lines are 90°.

Figure 6 — Pulley arrangement for calibrating torque

9 Cleaning of the apparatus

9.1 Clean the stirrer and crucible of all carbonaceous residue after each test.

9.2 Ream out the gas exit tube, if present, to maintain initial internal diameter.

9.3 Clean and lightly oil, or replace, the ball bearings after every 100 tests.

9.4 Check the thermocouple sheath after every 100 tests to see that it is not corroded.

10 Calculation and expression of results

10.1 From the observed counter and dial readings, the corresponding movement of the dial drum shall be calculated in dd/min. Bituminous coals show a wide range of fluidities. For this reason, it is convenient to plot dd/min as ordinates on a logarithmic scale against temperature as abscissa on an arithmetic scale.

10.2 All tests shall be made in duplicate and the mean values reported.

10.3 The report shall include the following:

a) characteristic temperatures rounded to the nearest 1 °C: initial softening; maximum fluidity, final fluidity, solidification, plastic range;

b) maximum fluidity (dd/min) (mean value), rounded off and reported as follows:

   1) to the nearest 1 dd/min between 0 dd/min and 100 dd/min;

   2) to the nearest 10 dd/min between 100 dd/min and 1 000 dd/min;

   3) to the nearest 100 dd/min between 1 000 dd/min and 10 000 dd/min;

   4) to the nearest 1 000 dd/min for values over 10 000 dd/min;

c) the maximum fluidity, if desired, as a logarithm (base 10) value, reported to two decimal places, whenever the maximum fluidity is greater than 1,0 dd/min;

d) a reference to this International Standard (i.e. ISO 10329);

e) dates on which the coal was sampled and on which analysis was performed;

f) a statement that maximum fluidity cannot be determined and the phenomenon that occurred during the test, if maximum fluidity cannot be determined because jamming or breaking has occurred.

11 Precision of the method

Table 1 — Precision data

Characteristics determined	Maximum acceptance range of duplicate determinations	
	Repeatability (Same laboratory) $\log_{10}$ dd/min	**Reproducibility (Different laboratory)** $\log_{10}$ dd/min
Maximum fluidity		
$<$ 20 dd/min	0,3	0,7
20 dd/min to 10 000 dd/min	0,1	0,3
$>$ 10 000 dd/min	0,2	0,4
Temperature points	7 °C	20 °C

11.1 Repeatability

For duplicate tests carried out at different times in the same laboratory, by the same operator with the same apparatus, on representative portions taken from the same sample after the last stage of the reduction process, all characteristic temperature points shall agree within 7°C. Likewise, the logarithms of maximum rates of dial drum movement of duplicate tests shall agree with the repeatability values given above in Table 1.

If the difference between duplicate determinations is greater than the repeatability values given above in Table 1, a second set of duplicate determinations shall be carried out. If the difference between the second set of duplicate determinations is greater than the repeatability values given above in Table 1, then the average of all four determinations should be reported.

If jamming or breaking is suspected to have occurred during the first or second set of duplicates, then a third set of duplicate determinations must be analysed to confirm the occurrence of either phenomenon.

11.2 Reproducibility

Due to the inherent variance of test equipment, the values shown for reproducibility are indicative only. Experience with national Gieseler standards indicates that it is very difficult to assign tight limits.

ICS 73.040

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