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Hard Coal — Determination of plastometric indices — Manual method

Houille — Détermination des indices plastométriques — Méthode manuelle

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Foreword

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This document was prepared by Technical Committee ISO/TC 27, *Coal and coke,* Subcommittee SC 5, *Methods of analysis*.

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 determination of plastic layer indices has been performed in GB/T 479[5] and GOST 1186[6] for many years. First, a manual detection of the height of the upper and lower interfaces of the plastic layer is performed, then curves for the upper and lower layers are established and finally the maximum thickness of plastic matter is calculated.

Instrumental methods for a more rapid determination of plastic layer indices are now available. If such a method is to be used, it is important to demonstrate that the method is free from bias, when compared to a reference method. In addition, it should give levels of repeatability and reproducibility which are the same as, or better than, those quoted for the reference method (see Clause 11).

The objective of this document is to provide a reference method for determination of the plastometric indices.

Hard Coal — Determination of plastometric indices — Manual method

1 Scope

This document specifies a manual method for the determination of plastometric indices. These indices are the maximum thickness of the plastic layer (*Y*, mm) and the final contraction value (*X*, mm).

This document is applicable for hard coals with a determined ash level less than 15 % on a dry basis, as described in ISO 11722 and ISO 1171.

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*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

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

ISO 18283, *Coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 and the following apply.

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

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

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

3.1
displacement curve of the plastic layer
curve of displacement of the packed coal bed changing with the temperature during the determination of plastometric indices

3.2
maximum thickness of plastic layer
Y
maximum perpendicular distance between the upper and lower plastic layer

3.3
final contraction value
plastometric shrinkage
X
perpendicular distance from the end of the displacement curve at 730 °C to the zero line (at 250 °C)

Note 1 to entry: This distance is 3 times that of the actual displacement of the plastic layer.

3.4
zero line
original height curve of the packed coal bed paralleling the X-axis drawn at a temperature of 250 °C

4 Principle

The coal sample is heated unidirectionally from the base at a standard rate under constant pressure whilst the plastic layer develops. The plastic layer thickness (referred to as *Y'*) is manually measured periodically throughout the test using a rounded end blunt probe. The displacement curve of the plastic layer is recorded. The curves representing changes of the upper and lower layers are generated by the least squares method. The maximum thickness of the plastic layer (referred to as *Y*) is calculated by the maximum perpendicular distance between the upper and lower layers. The final contraction (referred to as *X*) is calculated by the perpendicular distance from the end of the displacement curve at 730 °C to the zero line (at 250 °C).

5 Reagent and materials

5.1 Cigarette rolling paper, (also known as blanks), which are small sheets, rolls, or leaves of paper sold for rolling cigarettes either by hand or with a rolling machine.

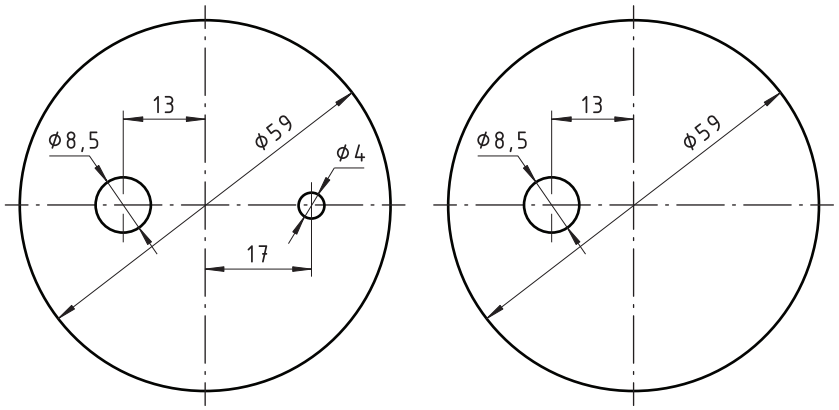
5.2 Filter paper, qualitative filter paper, dimensions of at least 60 mm wide and 190 mm to 200 mm long, used to line the inner wall of the retort (6.1.4).

NOTE The width of filter paper can be adjusted to keep bending the paper over the upper heat-resistant refractory round pad.

5.3 Thin steel rod, 3 mm in diameter and at least 150 mm long. Cigarette rolling paper is wrapped around the rod to make a tube. The resultant paper tube is then placed into the retort (6.1.4) and is surrounded by the coal after loading.

5.4 Refractory round pad. Heat resistant refractory ceramic pads or other refractory and porous pads, with thickness of 0,5 mm to 1,0 mm and diameter of 59 mm for use on the top and bottom of the coal sample in the retort (6.1.4). The pads can be made by manual or mechanical means. Each base pad requires a hole to allow the thermocouple tube (6.1.6) to fit through and a mark corresponding the probe hole of the pressure plate. Each top pad requires two holes, one to allow the thermocouple tube to fit through and one to allow the paper tube to fit through (see Figure 1).

Dimensions in millimetres



Key

1 top pad

2 base pad

Figure 1 — Refractory round pad

5.5 Abrasive cloth. Emery cloth P80 grade is suitable for removing coke residue from the retort (6.1.4) and associated components.

5.6 Displacement curve recording paper. A type of standard millimetre coordinate paper, with a height the same as the height of rotary drum (6.1.8) and length is slightly longer than the perimeter of the rotary drum.

5.7 Ceramic cord and ceramic wool

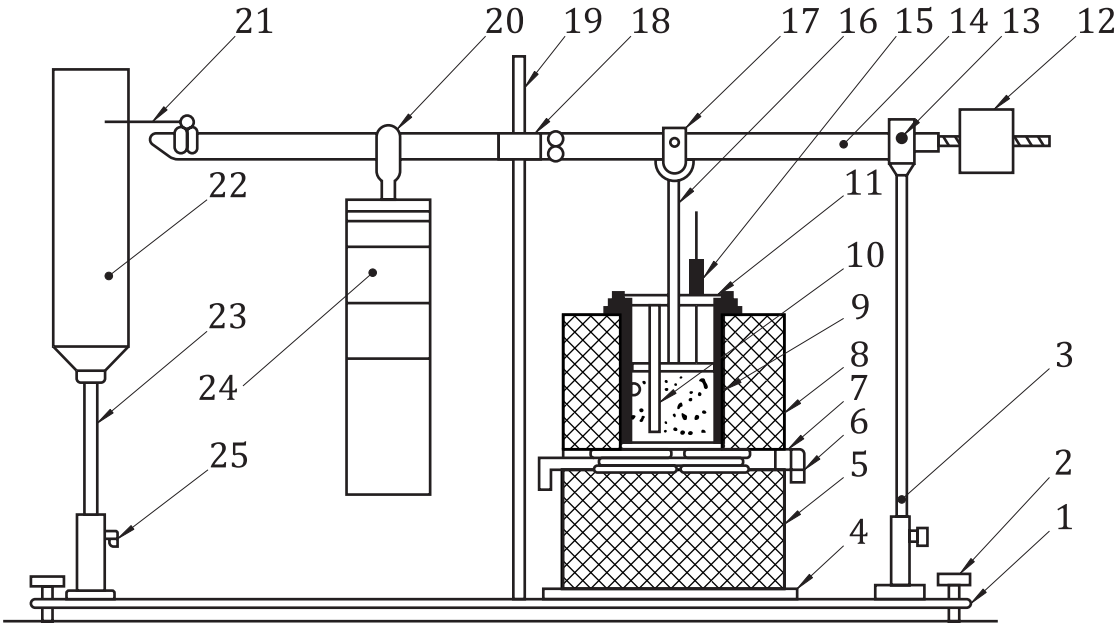
6 Apparatus

6.1 Plastometric apparatus

Typically, two types of manual plastometers are used in daily analysis of plastometric indices, one of which is with a balance mass (see Figure 2), the other is without a balance mass (the setup is the same as Figure 2 except without the balance masses).

The pressure applied to the cross section of the loaded coal sample during the measurement of the plastometric indices shall be $9{,}8 \times 10^4$ Pa (1 kg/cm^2).

The pressure cross section on the loaded coal sample should be checked when the apparatus is newly purchased, moved to a new location OR when major parts have been replaced. Annex B provides guidance on how to check the cross-sectional load on the coal sample.



Key

1	pedestal	14	lever
2	horizontal screws	15	probe
3	upright column	16	pressure plate
4	refractory pad	17	articulated joint
5	lower brick layer	18	direction control panel
6	connecting clamp	19	direction pillar
7	silicon carbide rod	20	masses hook
8	upper brick layer	21	recording pen
9	retort	22	rotary drum (with a clockwork)
10	thermocouple tube	23	rotary drum support
11	steel clamping bar	24	loaded masses
12	balance masses	25	set screw
13	connecting shaft		

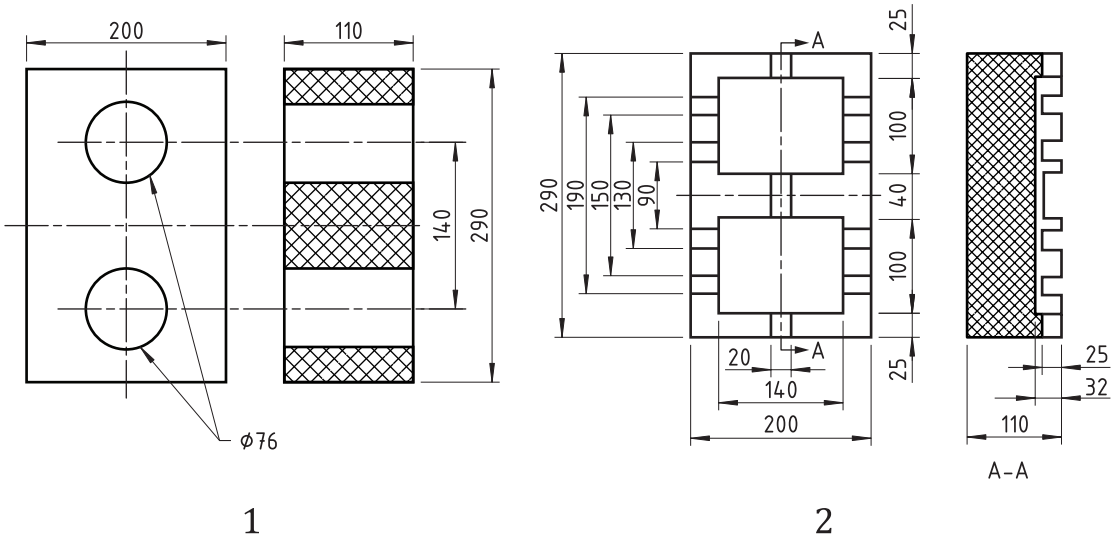
Figure 2 — Schematic of a plastometric apparatus with balance masses

6.1.1 Electric Furnace

The furnace shall consist of two layers of rectangular furnace firebricks, each measuring 200 mm × 290 mm × 110 mm. The lower layer has a longitudinal groove to allow for visual inspection, and four latitudinal grooves that support the four heating elements. The upper firebrick layer sits over the lower firebrick layer and has two cylindrical holes that accommodate the retorts (6.1.4). The upper firebricks (Figure 3) surface shall be flat and very carefully positioned, to ensure the alignment of the rolling paper tube, relative to the probe.

NOTE Typically the furnace firebrick has a refractoriness of 1 670 °C to 1 710 °C, in which the content of Al_2O_3 is not less than 40 %, and appearance porosity is not more than 26 %. Other refractory bricks can be used provided the furnace can achieve these temperature specifications

Dimensions in millimetres



Key

1 upper firebrick layer

2 lower firebrick layer

Figure 3 — Sketch of electric furnace fire bricks

6.1.2 Heating elements

There are four silicon carbide elements each protected by a quartz glass tube of 200 mm × 20 mm. The difference of resistance between the two series elements under each retort (6.1.4) is not more than 0,5 Ω. The elements shall have a resistance of 6 Ω to 8 Ω with an active length of 150 mm and diameter of 8 mm, providing a limiting incandescence temperature of up to 1 200 °C to 1 400 °C. The length of the cold end should be 60 mm long and diameter of 16 mm. The incandescence intensity of the heating elements decreases at a distance of 15 mm from the cold end. The resistance of the heating elements shall be checked at time intervals to ensure compliance with these temperature specifications.

NOTE Heating elements made from different materials can be used provided they can achieve these temperature specifications.

6.1.3 Pressure lever assembly

The pressure lever assembly consists of lever, loaded masses and horizontal adjustor as shown in Figure 2.

6.1.4 Retort

Component parts made with steel conforming to C45E4 in ISO 683-1 shall consist of 6.1.4.1 to 6.1.4.3.

6.1.4.1 Retort body

Specifications are shown in Figure 4. The external diameter is 70 mm. The height from the inside base of the retort bottom to the top of the retort body shall be 110 mm. The retort body shall be tapered, the internal diameter at the bottom shall be 59 mm and the internal diameter, at a height 50 mm from the bottom and top, shall be 60 mm. The inner wall of the body should be smooth without scratches and/or dents. The internal diameter of the working range of the retort body shall be measured for conformance to specifications every 50 determinations. To check the diameter, measure six points (every 10 mm from bottom) on the retort body. The variations between the average results of 6 points

and the average diameter (59,5 mm) should be within 0,5 mm. The gap between the retort base and retort body should also not be more than 0,5 mm.