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Hard Coal — Determination of plastometric indices — Manual method

Houille — Détermination des indices plastométriques — Méthode manuelle

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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 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).

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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*.

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.

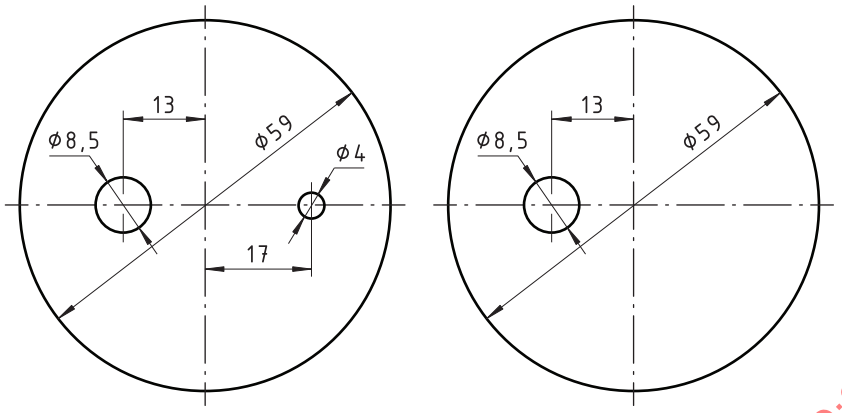
5.2 Filter paper, qualitative filter paper, dimensions of at least 60 mm wide and 190 mm to 200 mm long, used to line the inner wall of the retort (6.1.4).

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

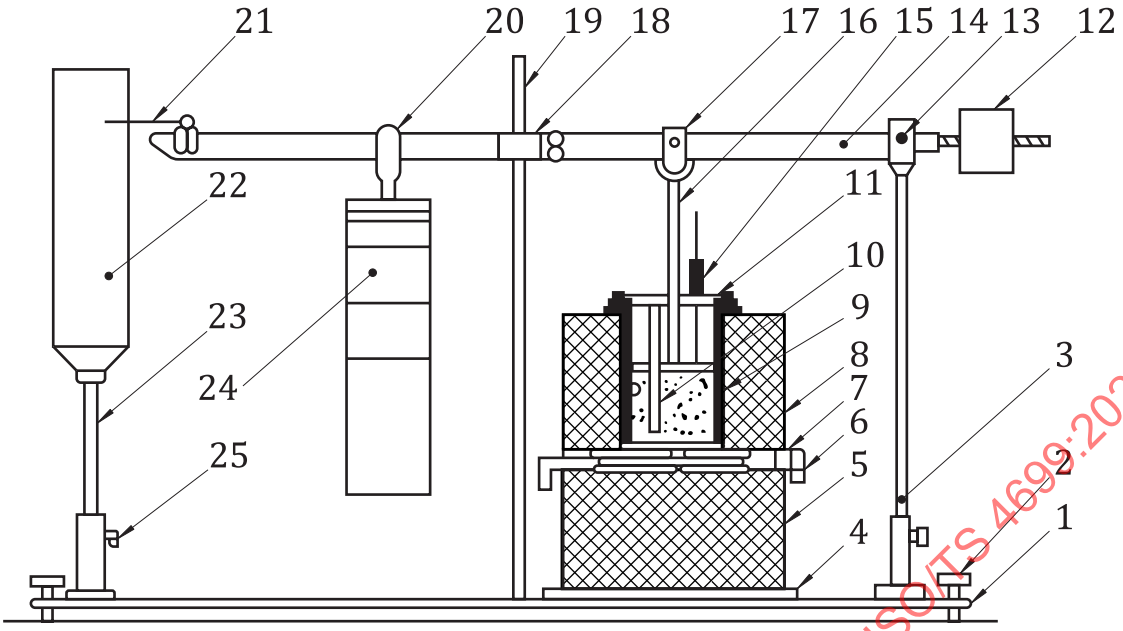
6 Apparatus

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		

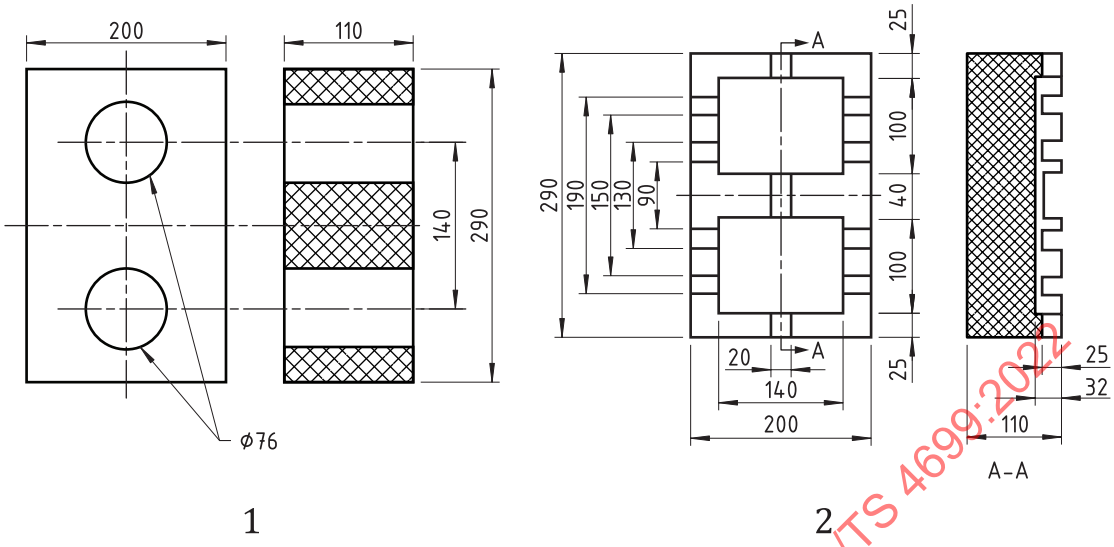
Figure 2 — Schematic of a plastometric apparatus with balance masses

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

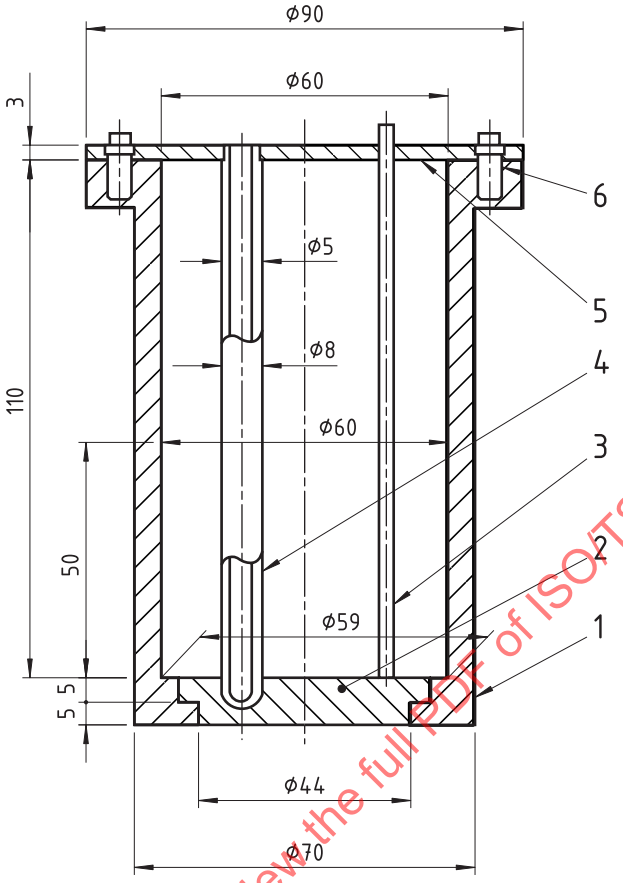
Component parts made with steel conforming to C45E4 in ISO 683-1 shall consist of 6.1.4.1 to 6.1.4.3.

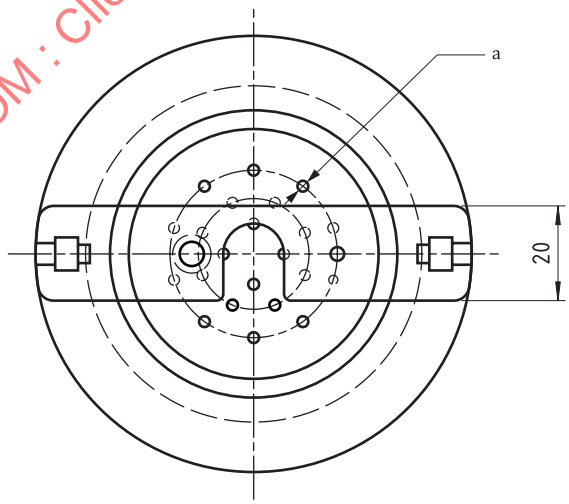
6.1.4.1 Retort body

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.

Dimensions in millimetres





Key

1 retort body
2 retort base
3 thin steel rod
4 thermocouple tube
5 steel clamping bar
6 screws for steel clamping bar

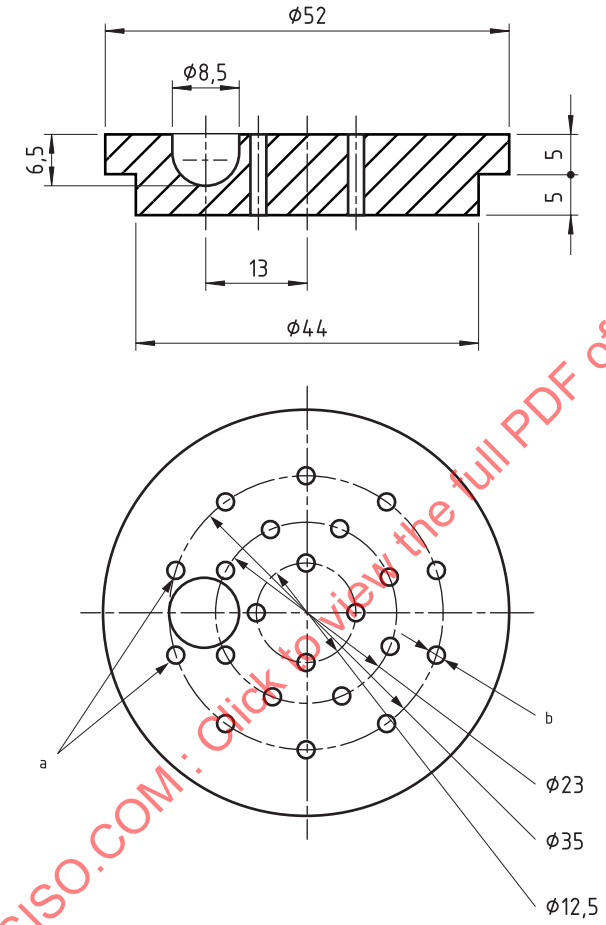
a 22 holes ϕ2.

Figure 4 — Retort body and other accessories

6.1.4.2 Retort base

Specifications and layout of gas ventilation holes are shown in Figure 5.

Dimensions in millimetres



a These two holes are symmetrically on the opposite sides of the centre line at a distance of 11,5 mm and other holes are equidistant.

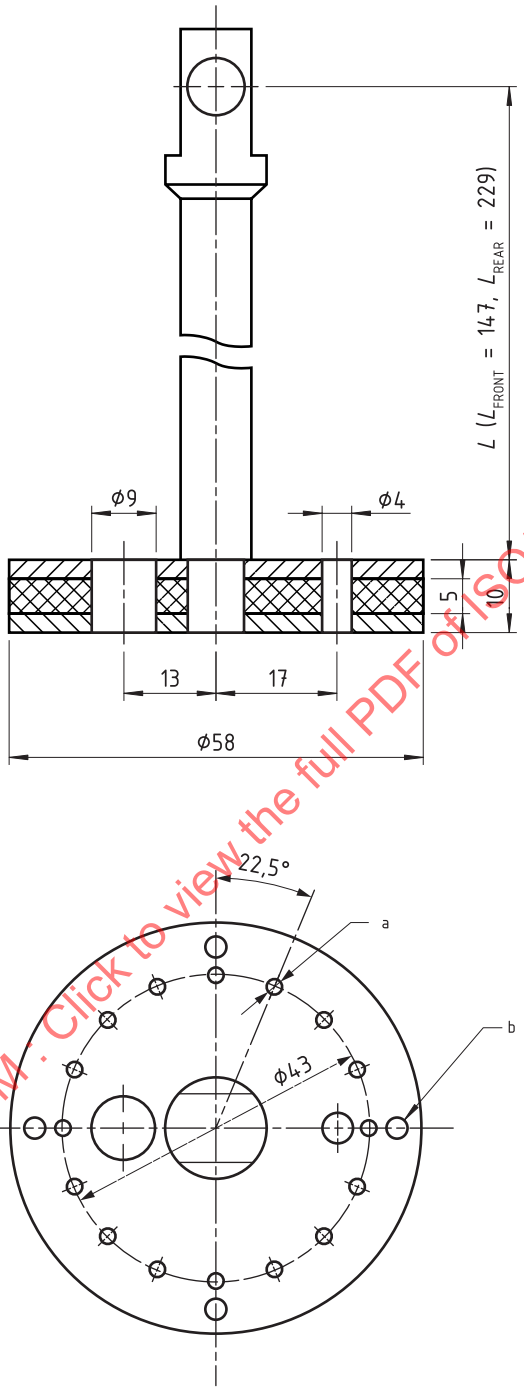
b 22 holes ϕ2.

Figure 5 — retort base

6.1.4.3 Pressure plate

There is a 5 mm thick refractory insulating fibre disc between the upper and lower pressure discs of the pressure plate. The dimensions and layout of 16 gas ventilation holes are shown in Figure 6.

Dimensions in millimetres



a 16 through holes $\phi 2$.

b 4 rivets.

Figure 6 — Pressure plate

6.1.5 Thermocouples

The thermocouple should be made from nickel-chromium and nickel-aluminium or nickel-chromium and nickel-silicon. The thermocouple and associated temperature measurement system shall be checked on a yearly basis to ensure accuracy of measurement.

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

6.1.6 Thermocouple tube

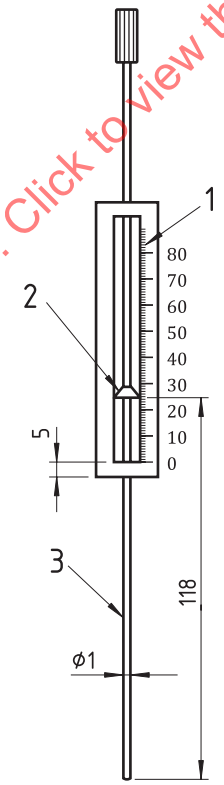
A hollow, stainless steel tube, closed at one end, that sits in the retort base (6.1.4.2), used to house the thermocouple. The dimensions are shown in Figure 4. Inspect the condition of the well frequently and after 100 tests to check that it has not corroded.

6.1.7 Probe

An example of probes is shown in Figure 7. The probe consists of a steel needle fixed with a rod on the top and a movable aluminium graduated scale. The steel needle with a rounded blunt end is 1 mm in diameter. The minimum division value of the graduated scale is 1 mm. The scale marks with a thickness of 0,1 mm to 0,2 mm should be straight and distinct.

The pointer should indicate the zero marks when measuring the bottom of the paper tube placed in a loaded retort before testing. Before each test, the probe shall be cleaned and checked to ensure it is straight. If it is not, it shall be replaced.

Dimensions in millimetres



Key

1 an aluminium graduated scale
2 pointer
3 steel needle

Figure 7 — An example of probes

6.1.8 Recording device

If a recording rotary drum with a clockwork is used, the rotary drum should operate at a smooth and constant speed to ensure the recording pen is capable of drawing a line with a length of 160 mm ± 2 mm over a period of 160 min. The rotating speed of the drum should be verified regularly to ensure it meets these requirements. During verification, the segment length drawn in the period of at least 80 min should be measured. Adjust the rotating speed of the drum to meet these requirements. Other types of recorders can also be used.

6.2 Programmed temperature controller

The furnace shall be heated electrically with automatic controls to ensure a heating rate of 8 °C/min until furnace temperature is 250 °C, after which the heating rate shall be 3,0 °C/min ± 0,1 °C/min from 250 °C to 730 °C.

The difference between the displayed temperature and the target temperature is not more than 5 °C from 350 °C to 600 °C and 10 °C for other periods. The temperature is measured with the thermocouple positioned in the thermocouple tube (6.1.6) in the retort (6.1.4).

6.3 Exhaust device

This device is used to exhaust fumes generated during measurement.

6.4 Mass measurement device

The mass measurement device is a top loading balance with a resolution of 0,1 % or less relative to the test portion mass.

6.5 Spirit level gauge

The spirit level gauge is used to measure if the pressure lever is horizontal (level).

6.6 Callipers

Callipers are used to measure the internal height of the retort body, and the depth from the retort lip to the top of the pressure plate. These readings are required to confirm the height of the coal charge after loading the retort.

6.7 Double roll crusher

A double roll crusher can crush the sample to pass through a 1,5 mm round hole sieve.

NOTE If other methods are used to crush the sample to pass 1,5 mm round hole sieve, it is important that they achieve a similar effect of double rolls crusher and do not generate excessive fines.

6.8 1,5 mm round hole sieve

This 1,5 mm round hole sieve shall be in accordance with the requirements of ISO 3310-2.

6.9 Sampling shovel

The sampling shovel is rectangle, 45 mm long and 30 mm wide.

7 Sample preparation

7.1 Collect a representative gross sample of coal with the method specified in ISO 18283, ISO 13909-1, ISO 13909-2, and ISO 13909-3. The laboratory sample shall be prepared by the method specified in ISO13909-4 or ISO 18283.

1,5 kg of coal crushed to pass a 4 mm square holes sieve or no less than 0,7 kg of coal crushed to pass a 3 mm square holes sieve as described in ISO 3310-1 shall constitute the laboratory sample. This sample should be representative of the batch being tested.

For the purpose of the coal classification, the mass fraction of ash on a dry basis shall be less than 10 %. Hard coals with an ash level of more than 15 % can refer to this document. The ash level may be declared in the test report. Details of any beneficiation to a value of ash level can also be noted in the test report.

7.2 Spread the laboratory sample on a tray and allow it to equilibrate with the laboratory atmosphere. Drying (other than air drying) should not be continued beyond this point to avoid the effect of oxidation on the plastometric indices of the coal sample. The drying temperature should not exceed 40 °C. After the laboratory sample has been air dried to equilibration, stage crush the laboratory sample with a double roll crusher (6.7) to pass a 1,5 mm round hole sieve (6.8), ensuring that a minimum of fines is produced. If the amount passing 0,212 mm is more than 30 %, it should be noted in the test report. The bulk density of the coal sample is affected by the particle size distribution when loaded into the steel retort and subsequently affects both the contraction, *X*, and the maximum thickness of plastic layer, *Y*, reported.

7.3 Thoroughly mix the stage crushed sample, preferably by mechanical means and divide a portion of 500 g stored into an airtight container. Test the coal sample for plastometric indices by duplicate determinations on the same day, as soon as practical after preparing the sample passing the 1,5 mm round hole sieve. Avoid delays as far as possible because the plastometric indices of coal sample can be significantly affected by deterioration and oxidation. If the coal sample cannot be tested in time, refrigeration or inert gases should be used to minimize oxidation of prepared coal samples.

8 Preparation for testing

8.1 Cleaning retort

Remove coke residual on the retort and its base, thermocouple tube and pressure plate with abrasive cloth (5.5) to make it clean. Each hole on these components should be clear and not blocked.

8.2 Preparation of paper tube

Wind cigarette rolling paper around a thin steel rod (5.3) to make a paper tube with diameter of 3 mm and at least 60 mm long. Coat a thin layer of glue on the edge of the end part of the rolling cigarette paper to avoid unrolling of the paper tube. Fold up the bottom of the paper tube about 2 mm to 3 mm to seal the tube. The top end should be closely attached to the thin steel rod to keep coal from flowing into the paper tube.

8.3 Preparation of refractory round pad

Prepare refractory round pads in accordance with the requirements of 5.4.

8.4 Loading retort

8.4.1 Fit the retort base into the retort body. The recess in the retort base should be aligned with the hole for resting the thermocouple tube in the steel clamping bar, making sure that the marked grooves line up and the two surfaces are well connected and flat.

8.4.2 Using callipers (6.6), measure and record the internal distance from the retort base to the top of the retort body, *H*, Figure 8.

8.4.3 Fit the base refractory round pad (5.4) into the retort body and gently push it to the base. The hole on it should be aligned with the recess in the retort base. Line the inside wall of the retort body close to retort bottom with the filter paper (5.2). Insert the thermocouple tube locating its base into the recess in the retort base. Position the paper tube (8.2) with the thin steel rod (5.3) on the mark of the base refractory round pad corresponding with the probe hole of pressure plate. The thermocouple tube and the paper tube/thin steel rod are vertically fixed by the steel clamping bar. Hold them in a vertical position during the entire coal-inflow period.

8.4.4 Thoroughly mix the stage crushed sample (7.3), preferably by mechanical means and divide to collect two sample portions of 100 g ± 0,1 g.

If the manual division method is used, the subsequent method may be carried out. Load all prepared samples on a dividing plate. Thoroughly mix the samples and spread them to form a rectangle with a uniform thickness of 10 mm. Then divide this sample rectangle into equal squares of 30 mm × 30 mm. Take two alternating increments by sampling shovel (6.9) in accordance with the flattened-heap method specified in ISO 13909-4 or ISO 18283, with 100 g ± 0,1 g for each.

8.4.5 Load the coal sample into the retort assembly in four separate equal (about 25 g) test portions by cone quartering. Take one of the portions and slide it into the retort. Use the flattening rod to flatten coal samples loaded into the retort. Take another portion on the diagonal direction and repeat the above operation of first portion. After each portion is added, level the coal surface to ensure even distribution. The coal sample shall not be tamped during this process. Ensure that no coal enters the thermocouple tube.

8.4.6 Carefully remove the steel clamping bar while ensuring that the thermocouple tube and thin steel rod, wrapped with cigarette rolling paper, are not disturbed. Place the top refractory round pad (5.4) onto the coal surface aligning the two pre-cut holes with thermocouple tube and thin steel rod. Fold the excess filter paper over the top pad. The coal sample in the retort should now be sheathed in filter paper and refractory round paper. Position the pressure plate by aligning the holes with thermocouple tube and the thin steel rod. Remount the steel clamping bar and secure it with the two bolts to fix the thermocouple tube. The paper tube should be visible above the surface of the pressure plate.

8.4.7 Using callipers (6.6), measure and record the distance from the upper surface of the pressure plate to the top of the retort, *a*, in four different quadrants and the thickness of a base pad, a top pad, and the pressure plate, b, for each retort (see Figure 8).

8.5 Adjusting the rotary drum (if applicable)

If a rotary drum (6.1.8) is used, roll it up with the displacement curve recording paper (5.6) lining up the start and end horizontal line before connecting with each other. Adjust the height of the rotary drum or recording pen to keep the two displacement curves of the front and rear retorts synchronously recorded. Check the distance from the connecting shaft centre (see key 13 in Figure 2) to the tip of the recording pen and adjust it to 600 mm.

8.6 Determining the height of the coal sample in the retort

The height of the coal sample in the retort before heating shall be manually measured and calculated with Formula (1):

$$h = H - (a + b) \tag{1}$$

where

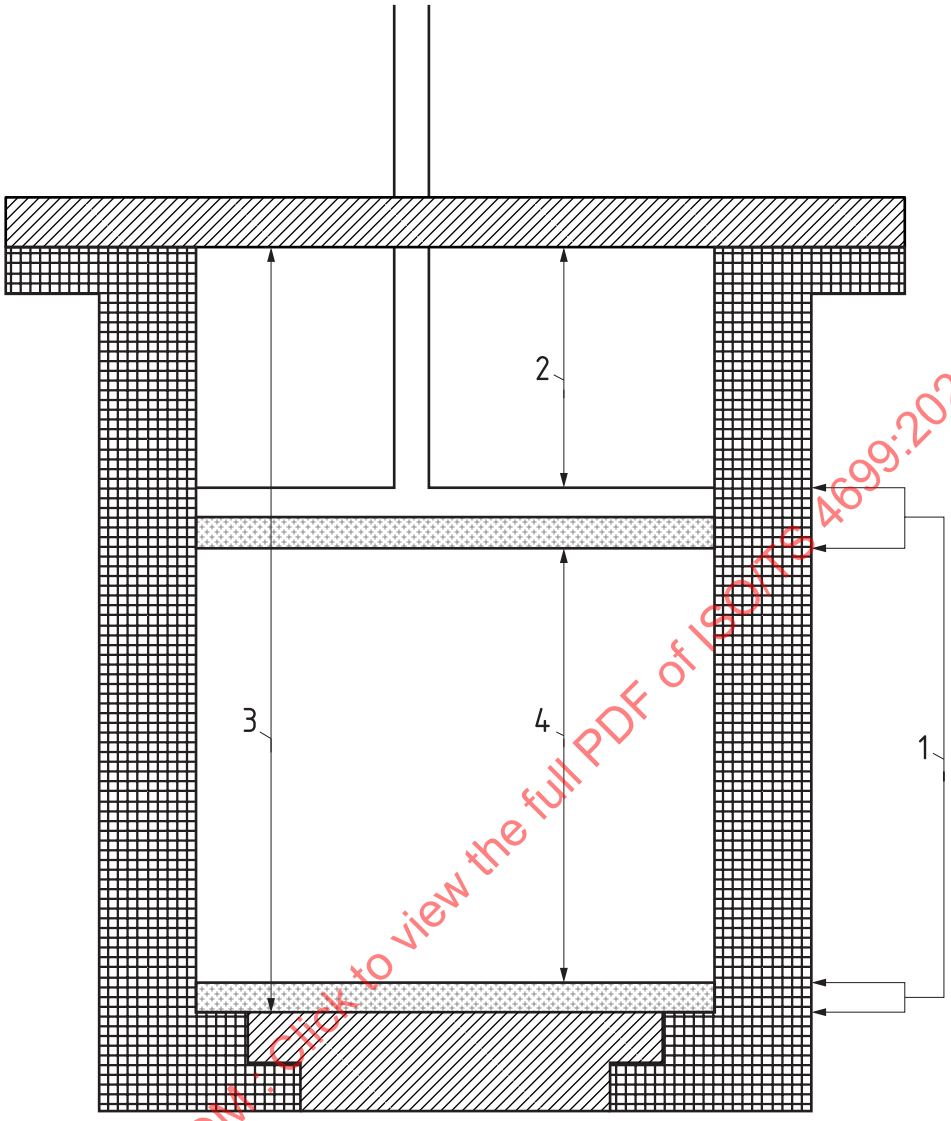
h is the height of the coal sample inside the retort, in mm;

H is the inner depth of the retort body, in mm (measured before coal sample is loaded);

a is the distance between the upper surface of the pressure plate and the top of the retort body, in mm (reported as the mean value from four different measures around the retort body);

b is the total thickness of the pressure plate and the two refractory round pads (measured with callipers), in mm.

The difference of the height of the coal sample inside the retort between duplicate tests shall not be more than 1 mm. Otherwise reload the retort with coal sample in accordance with the requirements of item 8.4. Record *h* and its average of duplicate tests.



Key

1 *b*, total thickness of the pressure plate and the two round refractory ceramic pads (measured with callipers), in mm

2 *a*, distance between the upper surface of the pressure plate and the top of the retort body, in mm (reported as the mean value from four different measurements around the retort body)

3 *H*, inner depth of the retort body, in mm (measured before coal sample is loaded)

4 *h*, height of the coal sample inside the retort, in mm

Figure 8 — Loaded retort

9 Test procedure

9.1 Place the loaded retorts into the furnace. Secure the pressure plate rod in its respective notched connecting arm. Attach the connecting arm with the bolts.

9.2 Hitch the masses. Check each lever arm with a spirit level gauge (6.5). Adjust the lever adjusting mechanism to ensure each lever is horizontal. Gently rotate and remove each thin steel rod ensuring that the position of each cigarette rolling paper tube is unchanged and remains vertical. The retort should be reloaded with coal sample in accordance with the requirements of item 8.4 if the paper tube moves out of the retort.

9.3 Put the graduated scale of the probe (6.1.7) on the steel clamping bar to measure the position of the bottom of the paper tube in the loaded retort. The pointer of the probe should be at the zero point of the scale marks, otherwise, reload the coal sample into the retort in accordance with the requirements of item 8.4.

9.4 Insert the thermocouples into the thermocouple tubes, making sure they are seated correctly.

9.5 Turn on the programmed temperature controller (6.2) and heat the furnace. The heating rate under the two loaded retorts is controlled at 8 °C/min until the furnace temperature is 250 °C after which the heating rate shall be 3,0 °C/min ± 0,1 °C/min from 250 °C to 730 °C.

9.6 Record the time and temperature during the testing. The initial testing time starts when the furnace temperature reaches 250 °C, then record the furnace temperature every 10 min. The difference between the displayed temperature and the target temperature shall not be more than 5 °C between 350 °C to 600 °C and not more than 10 °C for other periods. Otherwise, this run is invalid. The temperature is measured with the thermocouple positioned in the thermocouple tube in the retort.

9.7 If a rotary drum is used, adjust the tip of the recording pen to touch the recording paper on the drum and fix its position. Rotate the rotary drum in a circle to draw a zero line, then fix the tip of the recording pen on the starting point and start recording the displacement curve.

9.8 When measuring the upper plastic layer level, put the graduated scale of the probe (6.1.7) on the steel clamping bar and insert the steel needle into the paper tube carefully through the special holes on the steel clamping bar and pressure plate. Gently push the probe down until the end of the steel needle touches the surface of the plastic layer, which is the upper plastic level, causing a resistance felt by hand. Record the reading on the probe, which is the distance from the upper layer to the bottom of the retort. Record the reading of the upper plastic layer and probing time.

9.9 When measuring the bottom plastic layer level, probe the upper level of the plastic layer with the steel needle and then gently penetrate through the plastic layer until a significant increase in resistance is felt by hand. This is the surface of the semi-coke layer the bottom level of the plastic layer. The steel needle should be carefully and slowly penetrated into or pulled out of the plastic layer. Gently rotate and simultaneously remove the steel needle from the plastic layer to avoid removing any of the plastic layer or artificial release of gases stored in the plastic layer, which can damage the shape of the displacement curve and influence the position of the plastic levels.

NOTE 1 For the normal coal samples which do not generate a plastic layer with great fluidity, the upper level of the plastic layer can be probed when the displacement curve of the plastic layer descends about 5 mm, and the bottom level of plastic layer can be probed when the value of the upper level of the plastic layer reaches 10 mm. Measuring for the depth of plastic layer can be stopped when the furnace temperature reaches 650 °C.

NOTE 2 For coal samples with a mountain-type displacement curve or ones that generate a plastic layer with great fluidity, the measurement of the plastic layer level can be ceased after the maximum thickness of plastic layer is measured and the upper layer and bottom layer are successively probed 2 to 4 times each. Then, immediately block the probing hole on the pressure plate with ceramic cord or ceramic wool to avoid plastic mass overflow.

9.10 The frequency of measuring the upper plastic level and bottom plastic layer is dependent upon the shape of the displacement curve listed in 10.2 and the peculiarity of the plastic layer, which is specified below:

a) for a smooth descending type [Figure 10 a)], a tiny wave type [Figure 10 d)] or mountain type [Figure 10 f)]. Measure the top of the plastic layer every 5 min and the bottom of the plastic layer every 10 min.

b) for a wave type [Figure 10 c)] or zigzag type [Figure 10 e)]. Measure the upper level of the plastic layer at the peak. Measure the upper level and the bottom level of the plastic at the valley, and the bottom of the plastic layer at the valley is only measured every 8 min to 10 min. If displacement

curve ups and downs are frequent, the above measurements could be performed in the interval of one or two fluctuations.

c) for a mixture of types a) and b) [i.e. Figure 10 g) or Figure 10 h)]. The measurements for the levels of the upper plastic layer and bottom plastic layer should be performed according to the specific type at the time following the above provisions.

d) for a smooth ramp-down type [Figure 10 b), *Y* value is under 7 mm]. The levels of the upper layer and bottom layer are not conspicuous. The probe always tends to penetrate through the whole plastic layer to reach the bottom of the retort. In this case, stop the measurement for 20 min to 25 min to allow recovery of the plastic layer, then the probe should measure the top of the plastic layer and the bottom of the plastic layer at the frequency of no more than once every 15 min.

For coal samples that generate a plastic layer with a great fluidity during testing, the measurement of the bottom level of the plastic layer may begin later, then measure it in the interval of 7 min to 8 min. After the maximum thickness of the plastic layer is measured, the probing hole on the pressure plate is immediately blocked with ceramic cord or ceramic wool (around the temperature of 620 °C) to avoid plastic mass overflow.

If the plastic mass overflows the pressure plate during the testing process, stop the run and reload coal sample into the retort according to the requirements of 8.4. Additionally, the gaps between pressure plate and the inner wall of retort, and the pressure plate and thermocouple tube, should be sealed tightly using ceramic cord of 2 mm to 3 mm in diameter.

If a large amount of coal gas is discharged from the bottom of the retort during the process of testing, blow the heaters with air occasionally to avoid gas lock in the heating space and carbon black settling on the rods.

9.11 Stop the test when the furnace temperature reaches 730 °C. Remove the recording pen from the drum. Shut down the electricity and demount the masses and cool the furnace.

9.12 The furnace shall be cooled to room temperature before the next determination can be performed.

9.13 If the plastic mass flows over the pressure plate or the plastic layer level in the cigarette rolling paper tube rises abruptly, the run is invalid.

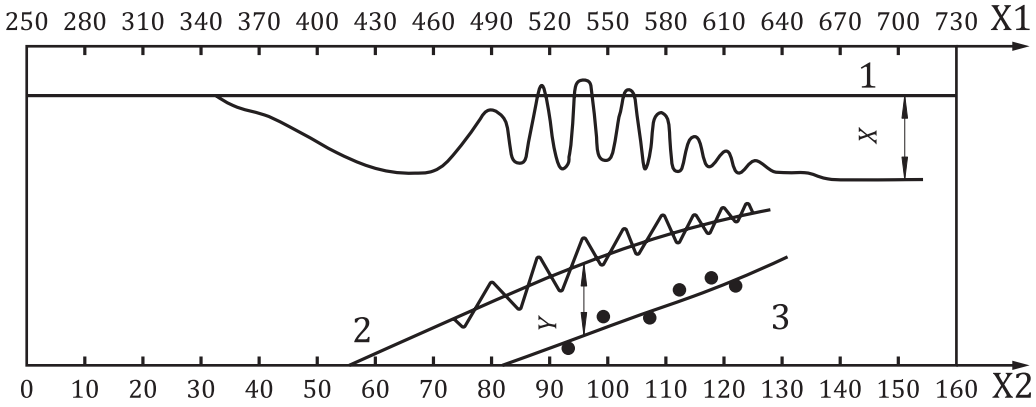
10 Expression of result

10.1 Processing of plastometric graph

The plastometric graph is processed as shown in Figure 9.

The temperature is marked horizontally above the displacement curve. Time (in min) is marked horizontally below the displacement curve as the X-axis and the distance to the bottom of the retort on the left is marked as the Y-axis (in mm).

The "upper plastic level curve" and "bottom plastic level curve" are created by the least squares method based on dots which represent the data of the upper and bottom plastic level locations, respectively, and the corresponding "time". The maximum thickness of plastic layer is calculated by the maximum perpendicular distance, *Y*, between both layers and final contraction, *X*, is obtained by the perpendicular distance from the end of the displacement curve at 730 °C to the zero line (at 250 °C).



Key

X1 temperature, in °C
X2 time, in min
1 zero line
2 upper plastic level
3 bottom plastic level

Figure 9 — Schematic diagram of plastic layer curve

10.2 Displacement curve types

Displacement curve types include:

a) smooth descending type, see Figure 10 a);

b) smooth ramp-down type, see Figure 10 b);

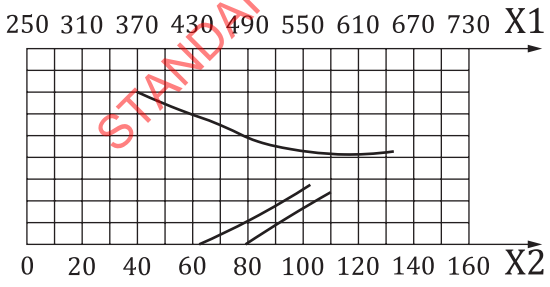
c) wave type, see Figure 10 c);

d) tiny-wave type, see Figure 10 d);

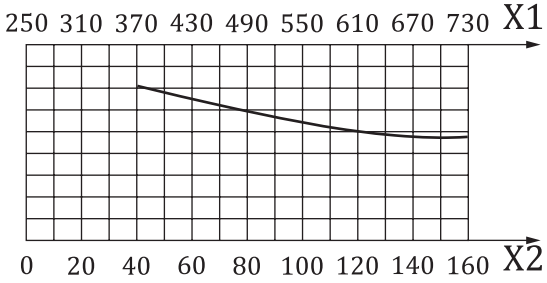
e) zigzag type, see Figure 10 e);

f) mountain type, see Figure 10 f);

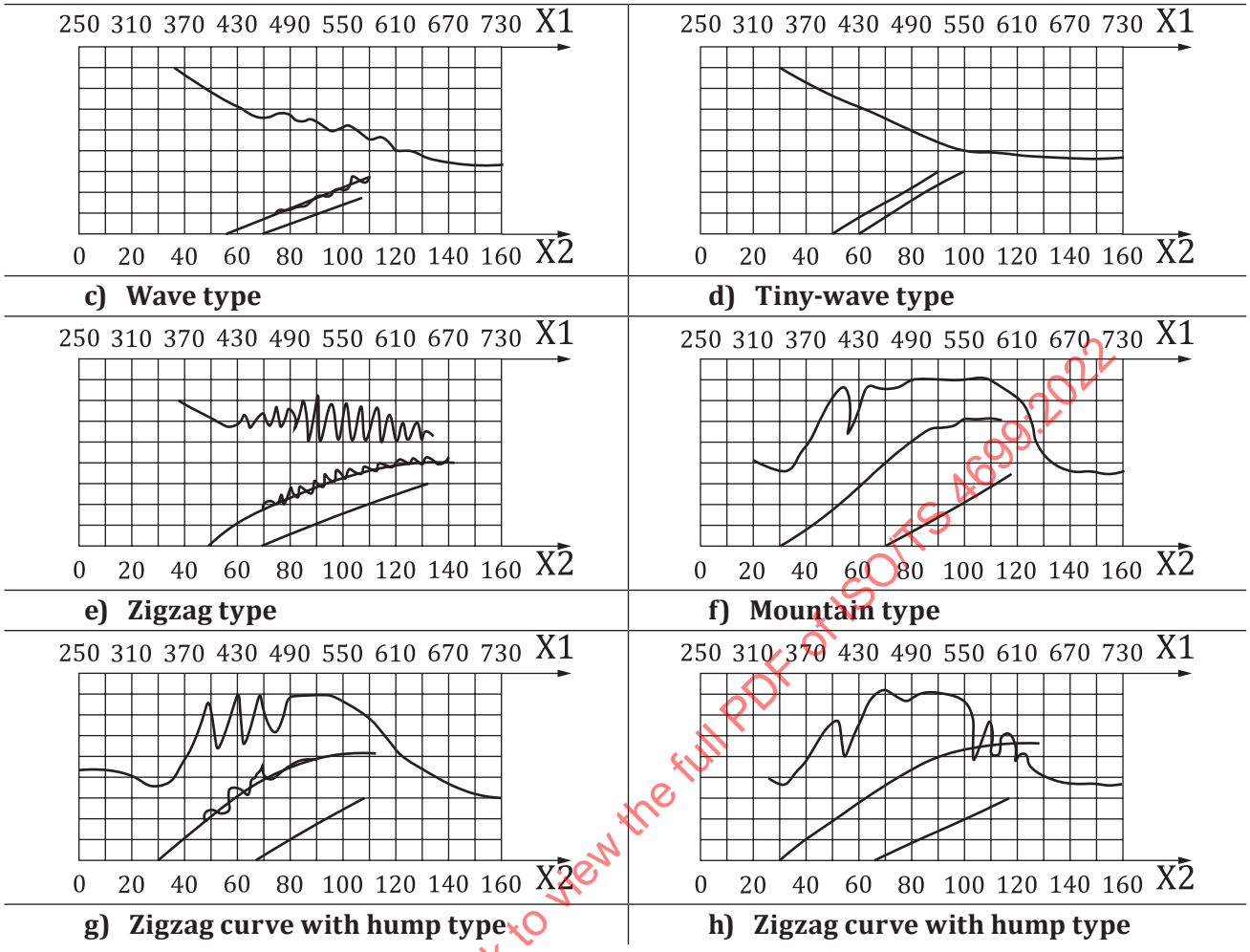
g) zigzag curve with hump type, see Figure 10 g) and 10 h).



a) Smooth descending type



b) Smooth ramp-down type



Key

X1 temperature, in °C

X2 time, in min

Figure 10 — Displacement curve types

10.3 Calculation and record

10.3.1 All tests shall be made in duplicate and the mean values reported to the nearest 0,5 mm.

10.3.2 The record should include at least the following:

— associated plastometric graphs for each determination with displacement curve type;

— final contraction value *X;*

— plastic layer thickness *Y'*;

— maximum thickness of plastic layer *Y* calculated;

— height of coal sample *h*;

— sample size distribution;

— sample moisture;

— if requested, report the technical characteristics of coke block identified by the method of Annex A.

11 Precision

11.1 Repeatability limit

NOTE Repeatability values are not included in this document and will be added in a later version after completion of an interlaboratory study.

11.2 Reproducibility limit

NOTE Reproducibility values are not included in this document and will be added in a later version after completion of an interlaboratory study.

12 Test report

The test report shall include the following information:

a) identification of the sample tested;

b) a reference to this document, i.e. ISO/TS 4699:2022;

c) the date of the determination;

d) the results and the method of expression used.

Annex A
(informative)

Visual assessmentfor the technical characteristics of the coke block

A.1 Cracks

The identification of cracks shall be subject to an inspection of the bottom surface (heating side) of the coke block. It is generally represented by three characterized types: a) no cracks, b) some cracks and c) many cracks, which are distinguished by the number of monomer coke blocks, i.e. the number of segments on the bottom surface of the coke block created by the crack lines.

a) No cracks, the number of monomer coke block is 1;

b) some cracks, the number of monomer coke blocks are 2 to 6;

c) many cracks, the number of monomer coke blocks are more than 6.

When a small portion of a crack is incomplete, the line of the crack can be extended to clearly delineate the area. As an example, in Figure A.1, the number of monomer coke blocks is eight, and the dotted lines are extensions along the crack lines.

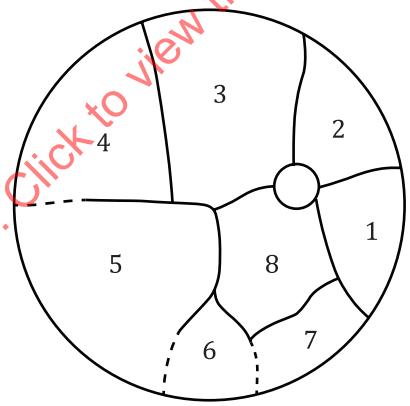


Figure A.1 — Schematic diagram of eight monomer coke blocks and cracks

A.2 Porosity

Porosity refers to the condition of the coke block profile, and is characterized by:

a) small pores;

b) small pores with large pores;

c) many large pores.