TECHNICAL SPECIFICATION

ISO/TS 20362

First edition
2022-08

Hard coal — Determination of plastometric indices — Automated Sapozhnikov penetration plastometer method

Houille — Détermination des indices plastométriques — Méthode automatisée du plastomètre à pénétration Sapozhnikov

Reference number
ISO/TS 20362:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
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, *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

Historically the determination of plastic layer indices has been performed by manual operation. Firstly, the thickness of the plastic layer is detected with a probe by hand, then curves of the upper and lower layer are manually established and the results calculated. This process is labour intensive and required technicians with vast experience.

In recent years, the automated type of determinator was developed to measure the plastic layer indices. Displacement curves are auto-established by computer. The intelligent manipulator automatically measures the thickness of plastic layer and establishes curves of upper and lower plastic layer. The result is reported by the system automatically.

The objective of this document is to provide an alternative method for determining the plastic layer indices with automated Sapozhnikov penetration plastometer.

Hard coal — Determination of plastometric indices — Automated Sapozhnikov penetration plastometer method

1 Scope

This document specifies a method for the determination of plastometric indices with an automated Sapozhnikov penetration plastometer. These indices are the maximum thickness of the plastic layer, *Y*, in mm, and the final contraction, *X*, in mm.

This document is applicable to hard coals with a determined ash level of less than 15 % as 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 13909-2, *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
maximum thickness of plastic layer
Y
maximum perpendicular thickness between the upper and lower plastic layer

3.2
final contraction value
plastometric shrinkage
X
distance between the height of the coal sample at the temperature of 250 °C (the zero line) and at 730 °C

3.3
zero line
original height of the coal sample paralleling with abscissa axis drawn at the 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 is automatically measured periodically throughout the test using a rounded end blunt probe. The manipulator arm lowers the probe through the paper tube created in the coal sample until a change in pressure is recorded. The volume changes are measured by displacement sensor and the displacement curve is auto-established by the computer. The curve representing changes of the upper and lower layer is generated by the least square method. The maximum thickness of the plastic layer is calculated by the maximum distance between both layers and final contraction is obtained by comparing volume at 250 °C and the end of a measurement automatically.

5 Materials

5.1 Cigarette rolling paper

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

5.2 Filter paper

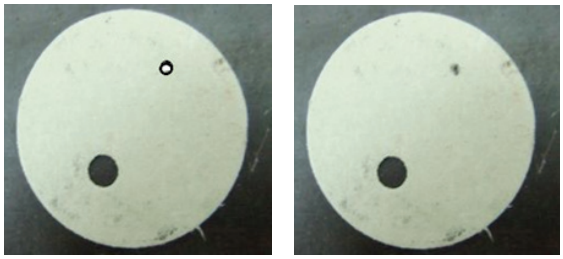
Qualitative filter paper, dimensions 60 mm wide and 190 mm to 200 mm long, used to line the inner wall of the steel retort.

5.3 Thin steel rod

The diameter of the thin steel rod is 3 mm. Cigarette rolling paper is wrapped around the rod to make a tube. The resultant paper tube is then placed into the steel retort and is surrounded by the coal after loading.

5.4 Refractory ceramic round pad

Heat resistant refractory ceramic pads with thickness of 1,0 mm and diameter of 59 mm for use on the top and bottom of the coal sample in the steel retort. The pads can be made by manual or mechanical means. Each base pad requires a hole to allow the thermocouple well 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 well to fit through and one to allow the paper tube to fit through. Figure 1 shows an example of these pads.



Key

1 top pad

2 base pad

Figure 1 — Refractory ceramic round pad

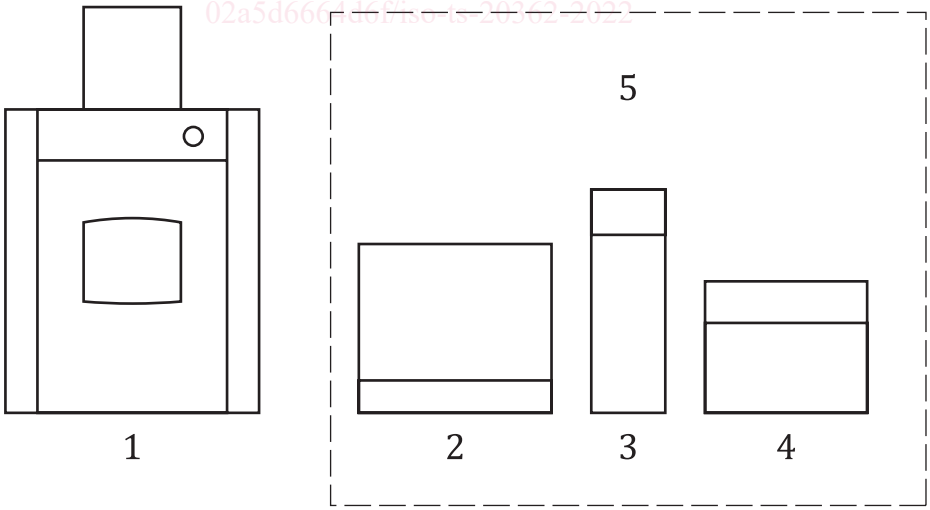
5.5 Abrasive cloth

Emery Cloth P80 grade is suitable for removing coke residue from steel retort and associated components.

6 Apparatus

6.1 Automated Sapozhnikov penetration plastometer

For determining plastometric indices with automated Sapozhnikov penetration plastometer, commercially available, consisting of determinator and the computer system. The computer system includes a computer, monitor, keyboard and printer (see Figure 2).



Key

1 determinator

2 monitor

3 computer

4 printer

5 computer system

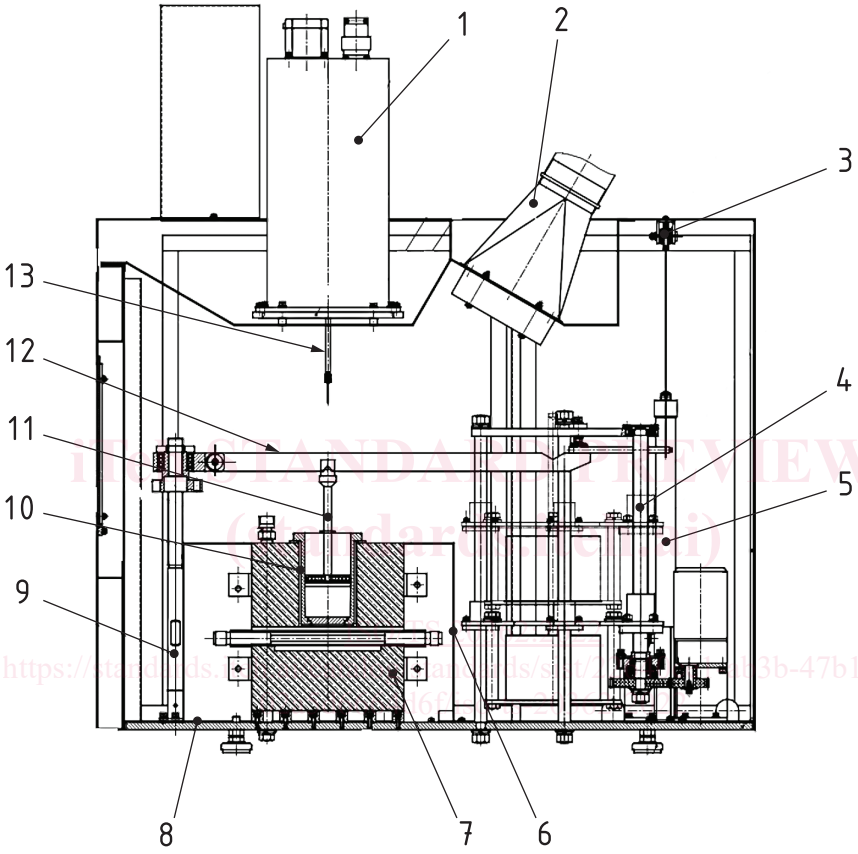
Figure 2 — Sketch of automated Sapozhnikov penetration plastometer

6.2 Determinator

The determinator shall consist of the following components as shown in Figure 3 and Figure 4.

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

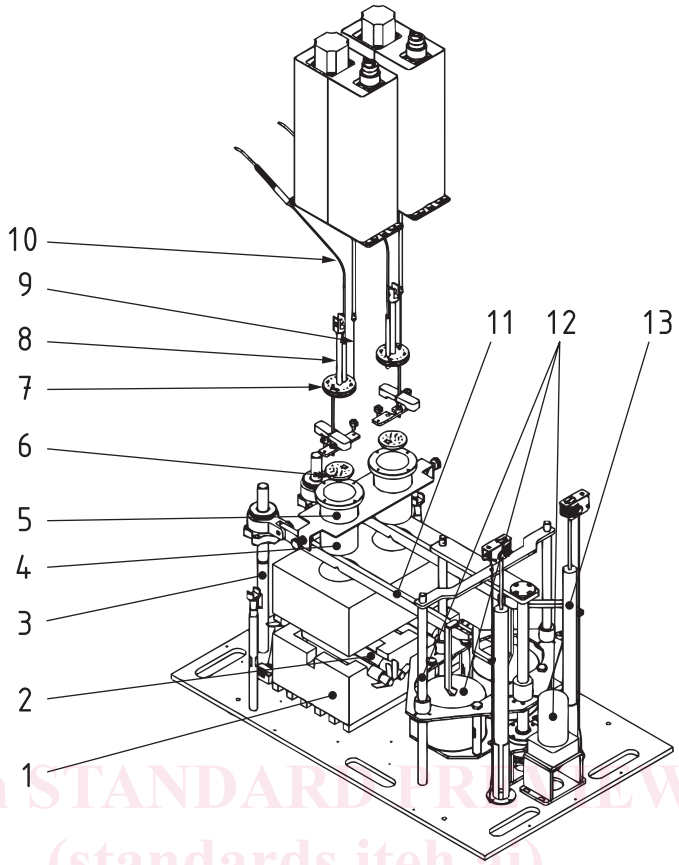
The pressure cross section on the loaded coal sample should be checked when the apparatus is newly purchased, moved to a new location or when major parts have been replaced. Annex B provides guidance on how to check the pressure on the cross section of the loaded coal sample.



Key

1	probe manipulator	8	base
2	exhaust	9	levelling assembly
3	pulley	10	steel retort
4	weight lifting device	11	pressure plate
5	displacement transducer	12	connecting arm
6	protective cover	13	probe and probe rod
7	brick stacks		

Figure 3 — Overview of a typical determinator



Key

1 electric furnace
2 heating elements
3 levelling assembly
4 steel retort
5 retort body
6 retort base
7 pressure plate
8 thermocouple well
9 probe
10 thermocouples
11 pressure lever assembly
12 weights elevating device
13 displacement sensor

Figure 4 — Exploded view of a typical furnace assembly

6.2.1 Electric furnace

The furnace shall consist of two layers of rectangular furnace brick, 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 brick layer sits over the lower brick layer and has two cylindrical holes that accommodate the steel retorts. The upper brick (see Figure 4) surface shall be flat and very carefully positioned according to manufacturer's specification, to ensure the alignment of the rolling paper tube, relative to the probe.

NOTE 1 Typically the furnace brick has the refractoriness of 1 670 °C~1 710 °C, in which the contents of Al_2O_3 are 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.

The furnace shall be heated electrically with automatic controls to ensure a heating rate of 3,0 °C/min ± 0,1 °C/min is maintained from 250 °C to 730 °C and a heating rate of about 8 °C/min is maintained before 250 °C.

NOTE 2 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 well in the steel retort.

6.2.2 Heating elements

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

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

6.2.3 Steel retort

Component parts made with steel according to ISO C45E4[5] specifications shall consist of 6.2.3.1 to 6.2.3.3.

6.2.3.1 Retort body

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 base, 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 average diameter (59,5 mm) should be within 0,5 mm. The gap between the retort base and the retort body should also not be more than 0,5 mm. Specifications and layout of air holes are shown in Figure 5.