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Hard coal — Determination of caking index

Houille — Détermination de l'indice d'agglutination

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Foreword

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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).

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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, *Solid mineral fuels,* Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 15585:2006), which has been technically revised. The main changes compared to the previous edition are as follows:

- Change to test precision (both for repeatability, *r*, and reproducibility, *R*) resulting from ILS conducted in 2016 and 2017.
- Additional information provided on anthracite sample packing and homogeneity test in Annex A and standard deviation formula and flowchart for standard anthracite sample taking in Annex B.

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

Caking index is a key parameter to identify the caking power of hard coal, which is a measurement of the agglutinating strength between the coal particles and inert constituents after heating without the contact of air. Coal caking characteristic is important and widely used in coking, gasification, liquefaction and combustion industries.

Hard coal — Determination of caking index

1 Scope

This document specifies a method of determination of caking index of hard coal. It is applicable to the evaluation of caking power of bituminous coal with random reflectance of vitrinite, R_r, greater than 0,6 % and less than or equal to 1,8 % (>0,6 % and ≤1,8 %).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

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

3.1
caking index
measure of the magnitude of the binding strength between coal particles or between coal particles and inert particles after coal is heated at 850 °C

4 Principle

A portion of prepared sample of coal of limited size range and the standard anthracite are mixed under defined conditions, and the mixture is carbonized rapidly. The crucible coke thus obtained is tested for strength in a drum conforming to certain specifications. The caking power of the test sample is expressed by the abrasive strength, i.e. resistance to breakage of the crucible coke.

5 Reagent and materials

5.1 Standard anthracite, having a moisture of less than 2,5 % in mass fraction, air-dried basis, an ash of less than 4 % in mass fraction, dry basis and a volatile matter of less than 8 % in mass fraction, dry, ash free basis. The size limits are 0,1 mm to 0,2 mm. The undersize content of 0,1 mm should not be more than 6 % in mass fraction, and the oversize content of 0,2 mm should not be more than 4 % in mass fraction.

NOTE Annexes A and B provide information on the extraction, preparation and testing of standard anthracite.

6 Apparatus

6.1 Balance, analytical balance with a resolution of at least 0,1 % relative of the test portion mass.

6.2 **Crucible**, porcelain, having the following dimensions (see Figure 1):

a) external diameter at the top: 40 mm ± 1,5 mm;

b) internal diameter at the base: 20 mm ± 1,5 mm;

c) external height: 40 mm ± 1,5 mm;

d) wall thickness: less than 2 mm.

Dimensions in millimetres

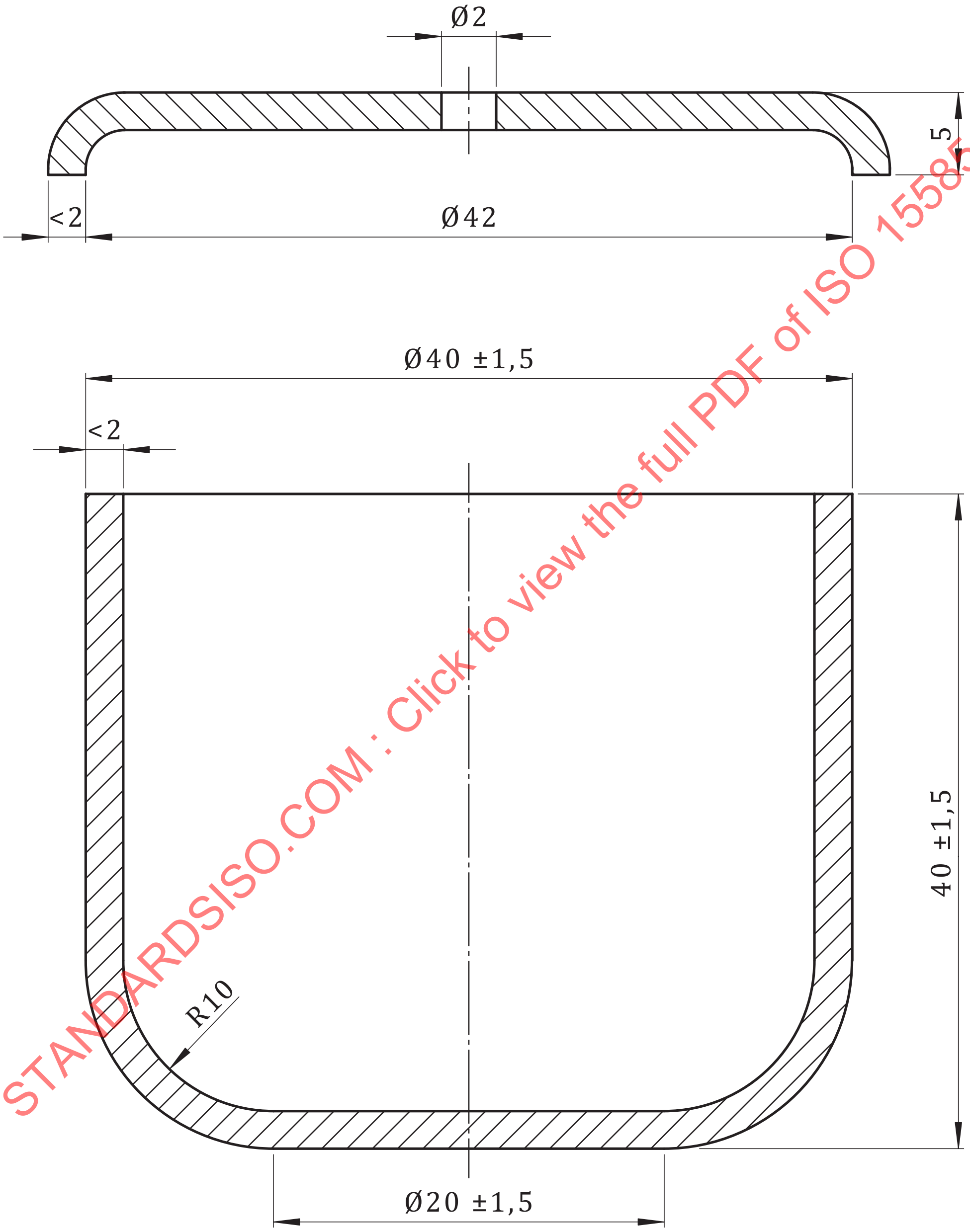

Figure 1 — Crucible and lid

6.3 **Lid**, porcelain, 1,5 mm to 2,0 mm thick, with a hole 2 mm in diameter in the centre (see Figure 1).

6.4 **Stirrer**, made of 1,0 mm to 1,5 mm diameter metal wire, having an 8 mm loop at one end (see Figure 2).

Dimensions in millimetres

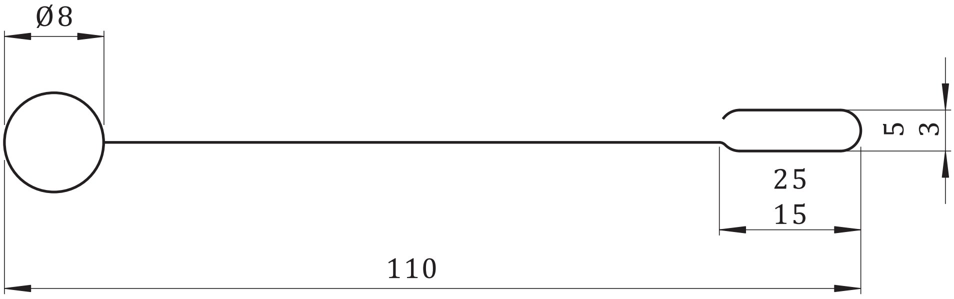

Figure 2 — Stirrer

6.5 Heat resistant weight, composed for example of Nichrome steel[1], with a mass of 110 g to 115 g (see Figure 3).

Dimensions in millimetres

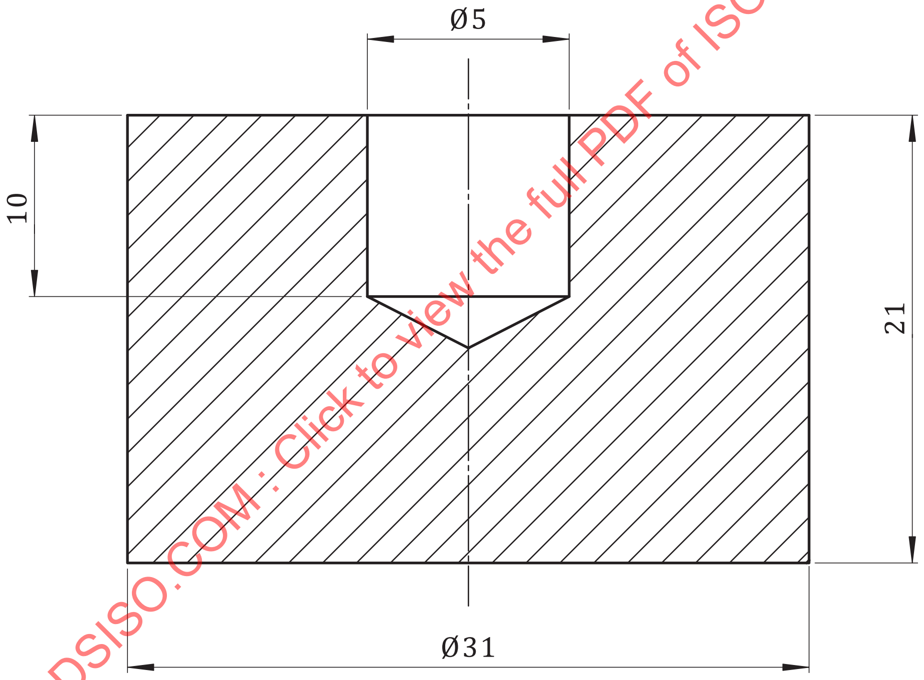

Figure 3 — Heat resistant weight

6.6 Press, for compressing the mixture of coal and standard anthracite under a weight having a 6 kg mass (see Figure 4).

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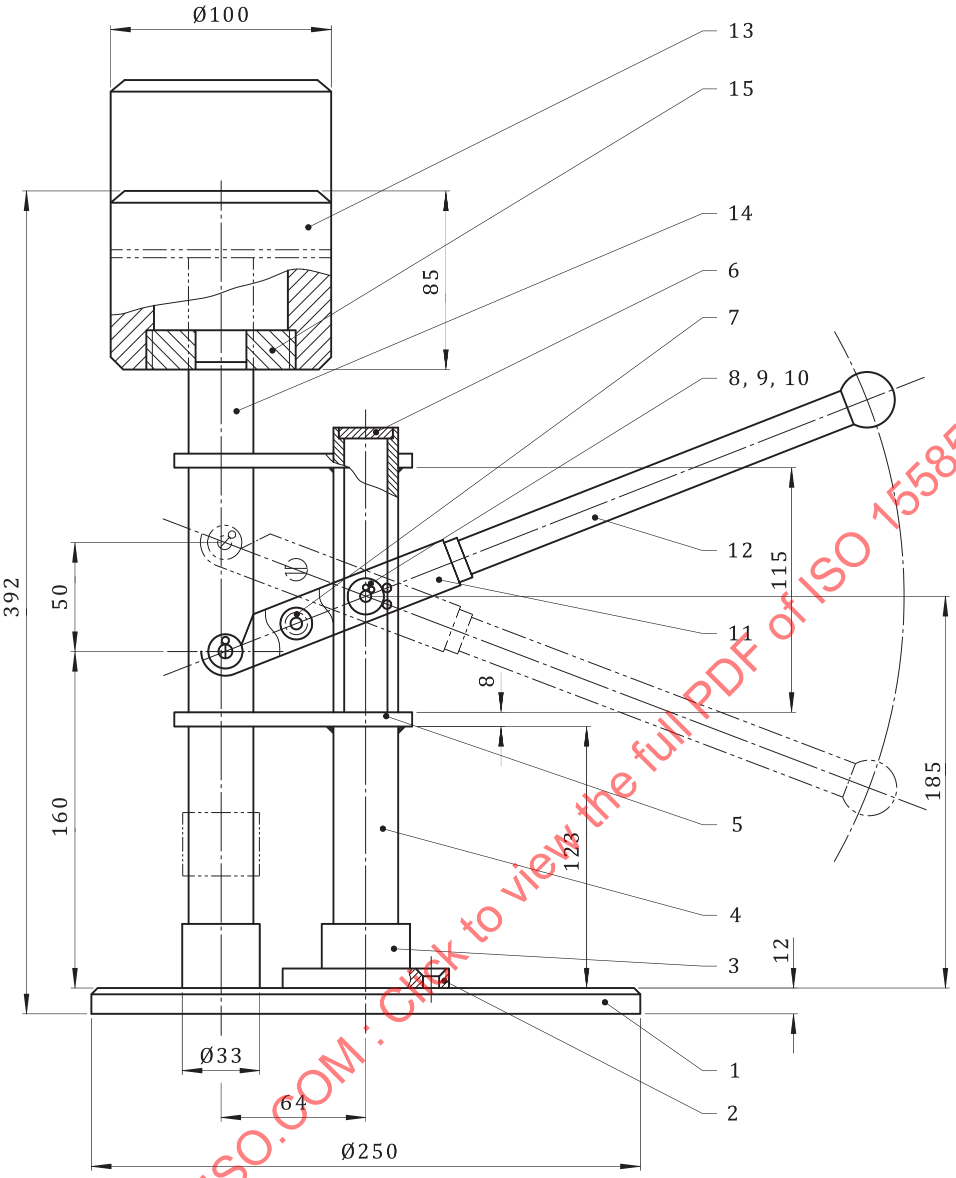
[1] Nichrome is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Dimensions in millimetres



Key

1	bottom plate
2	screw
3	round base
4	steel tube
5	connecting board
6	dead plate
7	back-up shaft
8	small shaft
9	dump
10	forelock
11	carriage
12	hand knob
13	press
14	lighting vertical shaft
15	plug

Figure 4 — Press used for compressing the mixture of anthracite and test coal

6.7 Electric furnace, with a zone of uniform temperature and a temperature control device capable of maintaining that zone at 850 °C ± 10 °C.

6.8 Drum, with a cover, driving shaft, transmission gear and electric motor for carrying out the abrasion test on coke.

The drum (see Figure 5) has a 200 mm internal diameter, is 70 mm deep and is made of 3 mm thick sheet iron. To the inside walls are welded two symmetrical sheet iron strips 70 mm long, 30 mm wide and 2 mm thick. To close the drum, the cover is seated on a felt or rubber gasket and is secured by two wing-nuts. The drum is rotated with the stub axle horizontal at (50 ± 0,5) r/min.

Dimensions in millimetres

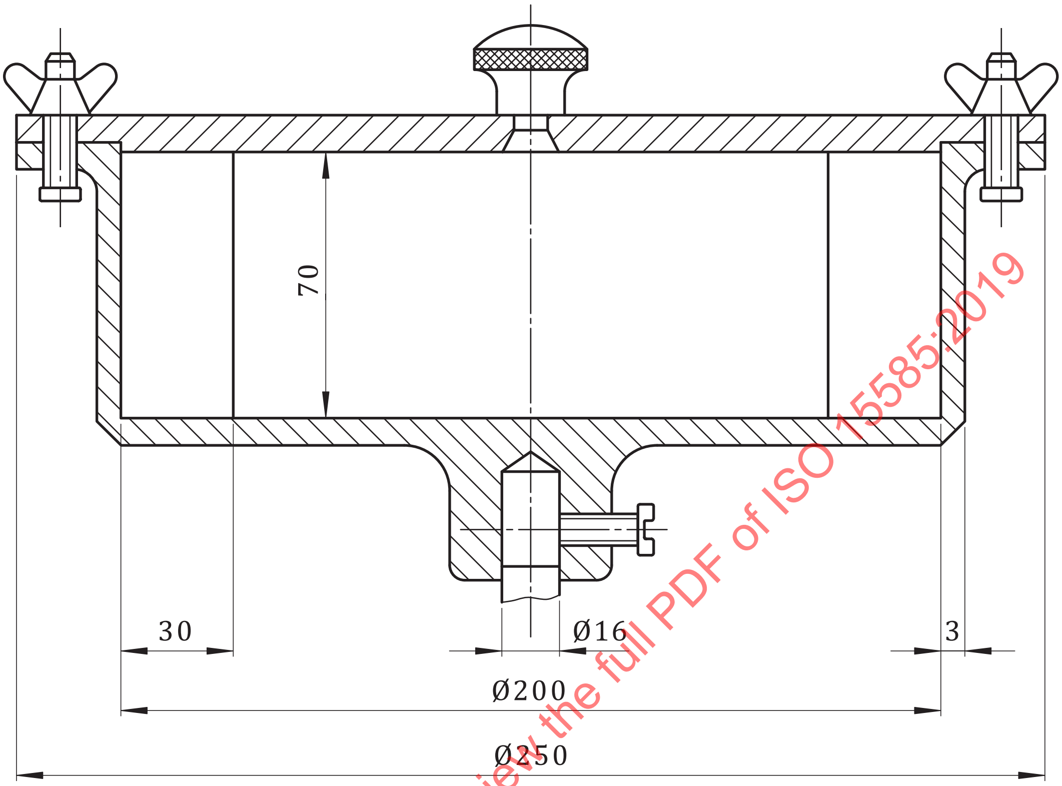


Figure 5 — Drum used for test

6.9 Laboratory sieve, made of thin brass sheet or stainless steel sheet, with 1 mm round holes.

6.10 Stopwatch.

6.11 Brush.

6.12 Long handled tongs or rod, suitable for moving the weight.

7 Preparation of test sample

7.1 Crush the sample of air-dried coal to pass a 0,2 mm test sieve. Take care to avoid an excessive production of particles below 0,1 mm. It is essential that 20 % to 40 % mass fraction of the total sample consists of particles between 0,1 mm and 0,2 mm.

7.2 The test sample shall be kept in a tightly closed container. Determine caking index of the sample on the same day after its preparation. Otherwise, refrigeration of prepared sample or maintaining it under inert gases or other anti- oxidation method should be used to minimize oxidation, and the time between preparation of the sample and carrying out the test shall not exceed 5 days.

7.3 Before commencing the determination, mix the analysis sample for at least 1 min.

8 Procedure

8.1 Carry out duplicate determinations on each sample of coal. These determinations shall not be carried out simultaneously, but for convenience, the second crucible may be heated in the furnace at the same time as the first.

8.2 Weigh a clean, dry crucible (6.2). Place in it 1,00 g of the coal and 5,00 g of the standard anthracite (5.1). Both weighed to an accuracy of 0,01 g. Mix carefully for 2 min with the stirrer (6.4), level off the surface and place the heat resistant weight (6.5) on it. Press the whole for at least 30 s under the weight having a 6 kg mass (6.6). Remove the crucible from the press and cover it with the lid (6.3), leaving the heat resistant weight in the crucible.

8.3 Raise the temperature of the furnace to about 850 °C and insert the crucible. Check the temperature on the floor of the furnace at the side of the crucible and ensure that the temperature of 850 °C ± 10 °C is regained within 6 min from the insertion of the crucible, then keep the temperature of 850 °C ± 10 °C, otherwise the run is invalid. After a total heating time of 15 min (including temperature recovery time of 6 min), remove the crucible from the furnace and allow it to cool on a heat resistant plate for 45 min.

8.4 After cooling, remove the weight from the crucible by using the tongs or rod (6.12). Brush back into the crucible any particles of coke adhering to the heat resistant weight and weigh the crucible plus contents to an accuracy of 0,01 g.

8.5 Transfer the contents of the crucible to the drum and fit the cover. Start the stopwatch and rotate the drum for 5 min at a speed of (50 ± 0.5) r/min. Remove the coke from the drum and sieve it through the 1 mm round hole sieve (6.9). Transfer the coke remaining on the sieve back to the crucible and reweigh. Return the coke from the crucible to the drum and repeat the abrasion procedure, sieving and reweighing of the oversize exactly as described above. Carry out a second abrasion test under the same conditions, sieve and weigh the final oversize. All weightings shall be made to an accuracy of 0,01 g.

8.6 If the value of caking index obtained as per above procedure and calculated by Formula (1) is less than 18, a further test should be carried out with the ratio of test sample to standard anthracite changed to 3:3. Weigh 3 g of test sample and 3 g of standard anthracite. The remaining procedure is the same as that above but the caking index value is calculated by Formula (2).

9 Expression of results

9.1 If the ratio of test sample to standard anthracite is 1:5, the caking index, G, is given by Formula (1).

$$G = 10 + \frac{30m_1 + 70m_2}{m} \tag{1}$$

where

m is the total mass of crucible coke after carbonization, expressed in grams;

m_1 is the mass of coke remaining on the sieve after the first drum test, expressed in grams;

m_2 is the mass of coke remaining on the sieve after the second drum test, expressed in grams.

9.2 If the ratio of test sample to standard anthracite is 3:3, calculation of the result is as given in Formula (2).

$$G = \frac{30m_1 + 70m_2}{5m} \tag{2}$$

9.3 Calculate the result of each determination to one decimal place. Report the result as the mean of duplicate determinations, to the nearest integer. If the duplicate results differ by more than the repeatability figure given in Table 1, disregard the results and repeat the complete test.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus within a short interval of time on representative portions taken from the same analysis sample, shall not differ by more than the values of the repeatability limit, *r*, shown in Table 1.

10.2 Reproducibility limit

The mean of the results of duplicate determinations, carried out in each of two laboratories, on the representative portions taken from the same sample at the last stage of sample preparation, shall not differ by more than the values of the reproducibility limit, *R*, shown in Table 1.

Table 1 — Precision of the method

Cake index *G*	Repeatability limit *r*	Reproducibility limit *R*
<18	1	4
≥18	3	6

11 Test report

The test report shall include the following information:

a) identification of the sample tested;

b) the method used by reference to this document, i.e. ISO 15585:2019;

c) the date of the determination;

d) results and the calculation basis in which they are expressed.

Annex A
(informative)

Extraction and preparation of standard anthracite for caking index determination

A.1 Source of standard anthracite

Two definite seams of the Ruqigou mine, China, are designated for the extraction of the raw coal for preparing the standard anthracite.

Any anthracite that conforms to the specifications given in 5.1 and meets the requirements of Annex B may be used for the preparation of the standard anthracite.

A.2 Preparation of standard anthracite

A detailed procedure should be followed for the preparation of the standard anthracite (including picking out any dirt band and shale and other contaminants from the raw coal, drying, crushing with a specified crusher, and sieving with a specific sieve).

A.3 Quality checks and verification

A regular examination of the prepared standard anthracite should be followed for checking the moisture, ash and volatile matter content in accordance with ISO 11722, ISO 1171 and ISO 562, respectively; the size analysis in accordance with ISO 1953; the content of undersize and the comparison with the reference anthracite by performing a caking index determination of appropriate bituminous coal samples.

Any standard anthracite that is commercially available should be marked with the limits of ash, volatile matter and undersize content and the lot number and the verification certificate should be attached.

A.4 Sample packing

The sample was divided into the required mass by using riffling or other division methods described in ISO 13909-4 or ISO 18283, and then packaging the anthracite in a clean and sealed container, and ensure maintaining of integrity (no breakage) during transportation. The net quality of each minimum packing unit is (1 000 ± 10) g or (500 ± 5) g. The verification certificate should be attached in each minimum packing unit.

A.5 Homogeneity test

Extract five packing units randomly, and perform duplicate determination of the caking index of a bituminous coal sample with G value in range 70 to 90. Calculate the variance within packing units and variance between packing units, and run a F-test.

If F-test shows the samples are not well distributed, all the samples in packing units should be unpacked and remixed. Determine the causes and re-process the samples to form the smallest packing units. Repeat the homogeneity test as per the above method until the samples are well distributed.

Annex B

(informative)

Quality test for standard anthracite used for caking index determination

B.1 Reference anthracite sample

Any reference anthracite sample should be renewed every 3 years.

B.1.1 Method of preparation

Four new reference samples are extracted and prepared in accordance with Annex A. Each new reference sample should have a mass of 4 kg and be divided into two equal portions, one of which is used for the test and the other should be preserved by the preparing laboratory.

B.1.2 Analysis of test samples

The following should be determined on the test samples:

a) Caking index, G, of appropriate bituminous coals;

b) Moisture in the analysis sample, ash and volatile matter, in accordance with the specifications in 5.1;

c) Size limits, in accordance with the specifications in 5.1.

B.1.3 Scheme of test caking index

Extract one previous reference sample and four new reference samples. Each of which is tested with eight bituminous coals (with values of G ranging from 20 to 90 spaced at intervals of approximately 10) by performing the determination of the caking index, G. The determination of each bituminous coal is repeated six times. The average results of each of the eight bituminous coals on four new reference samples are compared by statistical analysis (based on the S and the Grubbs tests) to the results of the tests on the previous reference sample. No significant differences should be indicated.

B.1.4 Selection of new reference anthracite

The averaged values of G obtained using the new reference samples are compared with those obtained using the previous reference sample. At least two of the four new anthracite samples should conform to the relevant requirements; otherwise, all of them should be discarded. The qualified samples are mixed and taken as the new reference sample.

If in the first renewal of the reference anthracite, the value of G is lower than (within required limit) that of the previous reference sample, the value of G in the next renewal should be higher than that of the previous reference sample, so as to prevent a consistent decrease or increase in the following renewals.

B.1.5 Preservation of reference anthracite sample

The reference sample is divided into approximately 200 g portions and kept in a dry, cool dark place.

B.2 The standard anthracite product for commercial availability

Each 200 kg constitutes one test lot.

B.2.1 Method of sample-taking

One increment is obtained for each 2 kg portion by taking an appropriate amount of material with a long scoop from five points in each portion to make a total mass of approximately 150 g. The 100 increments taken from one lot (200 kg) are mixed and reduced to approximately 1 kg, which constitutes the sample that is sent for testing (see Figure B.1).

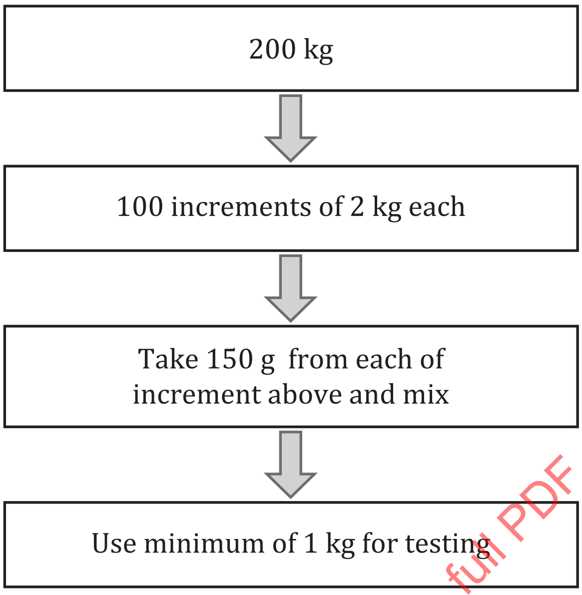


Figure B.1 — Sample-taking process

B.2.2 Analysis of test samples

The following should be determined from the standard anthracite test samples:

a) caking index, *G*, of bituminous coal;

b) Moisture in the analysis sample, ash and volatile matter, should conform to the following:

M_{ad} = 1,5 % to 2,5 % mass fraction;

A_d = 1,5 % to 4,0 % mass fraction;

V_{daf} = 6,5 % to 8,0 % mass fraction.

c) size limits, in accordance with the specifications in 5.1.

B.2.3 Scheme of test caking index

Determinations on each bituminous coal are repeated six times using the commercially available tested standard anthracite and the reference standard anthracite. The averages of the values of *G* for four coal samples (with values of *G* ranging from 20 to 90 at intervals of approximately 20) are determined using the tested and the reference standard anthracite. There should be no significant difference between them after statistical analysis using the T-test. The tolerance of the differences may be 2,5 S_C, to 3,0 S_C, where S_C is the collective standard deviation which is given in Formula B.1.

$$S_c = \sqrt{\frac{(n_1 - 1)S_1^2 + (n_2 - 1)S_2^2}{(n_1 + n_2 - 2)}} \tag{B.1}$$