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Hard coal — Determination of caking index

Houille — Détermination de l'indice d'agglutination



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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.

ISO 15585 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Method of analysis*.

Hard coal — Determination of caking index

1 Scope

This International Standard 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 % ($> 0,6 \% \leq 1,8 \%$).

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 562, *Hard coal and coke — Determination of volatile matter*

ISO 589, *Hard coal — Determination of total moisture*

ISO 1171, *Solid mineral fuels — Determination of ash*

3 Terms and definitions

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

5.1 Standard anthracite, having a moisture of less than 2,5 mass % air-dried basis, an ash of less than 4 mass % dry basis and a volatile matter of less than 8 mass % 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 mass % and the oversize content of 0,2 mm should not be more than 4 mass %.

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

6 Apparatus

6.1 **Balance**, capable of weighing to the nearest 0,01 g.

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

- a) external diameter at the top: 40 mm \pm 1,5 mm;
- b) internal diameter at the base: 20 mm \pm 1,5 mm;
- c) external height: 40 mm \pm 1,5 mm;
- d) wall thickness: less than 2 mm.

Dimensions in millimetres

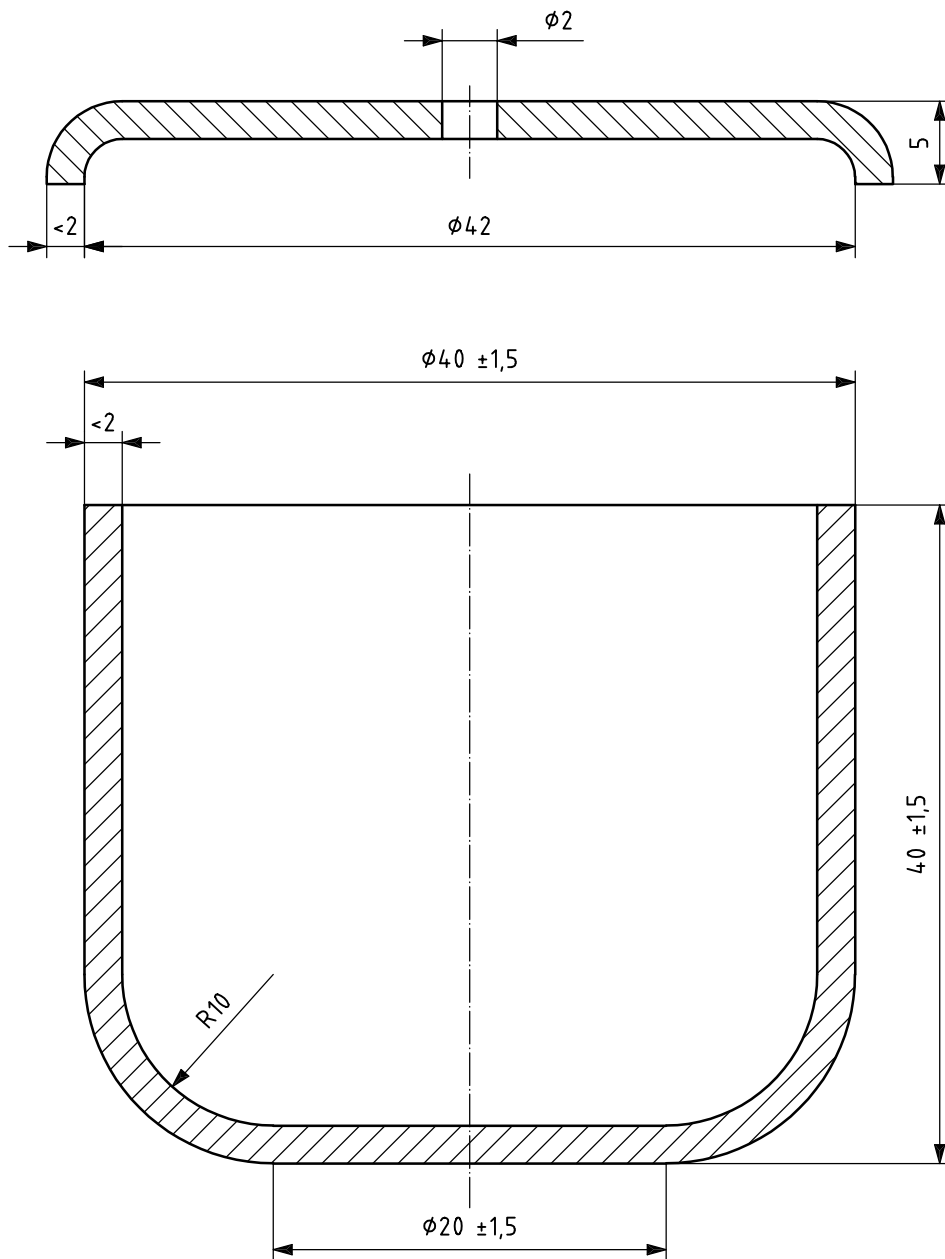


Figure 1 — Crucible and lid

- 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

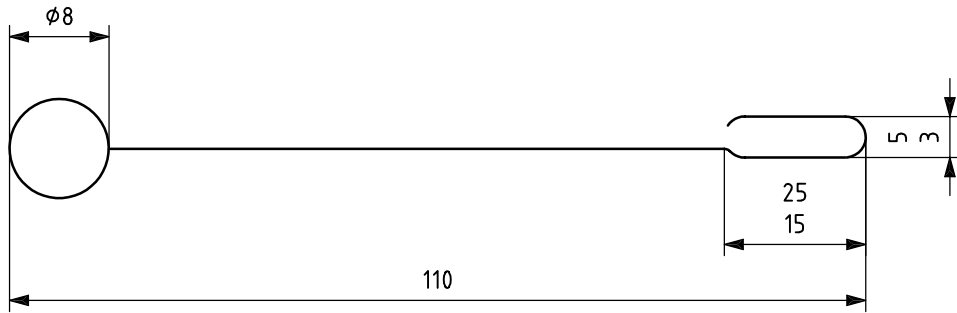


Figure 2 — Stirrer

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

Dimensions in millimetres

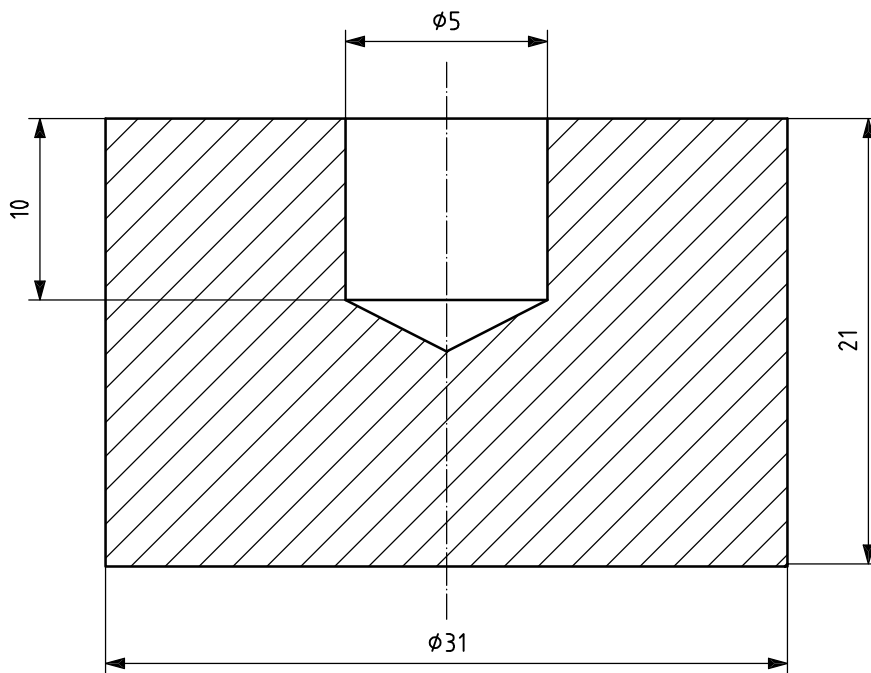
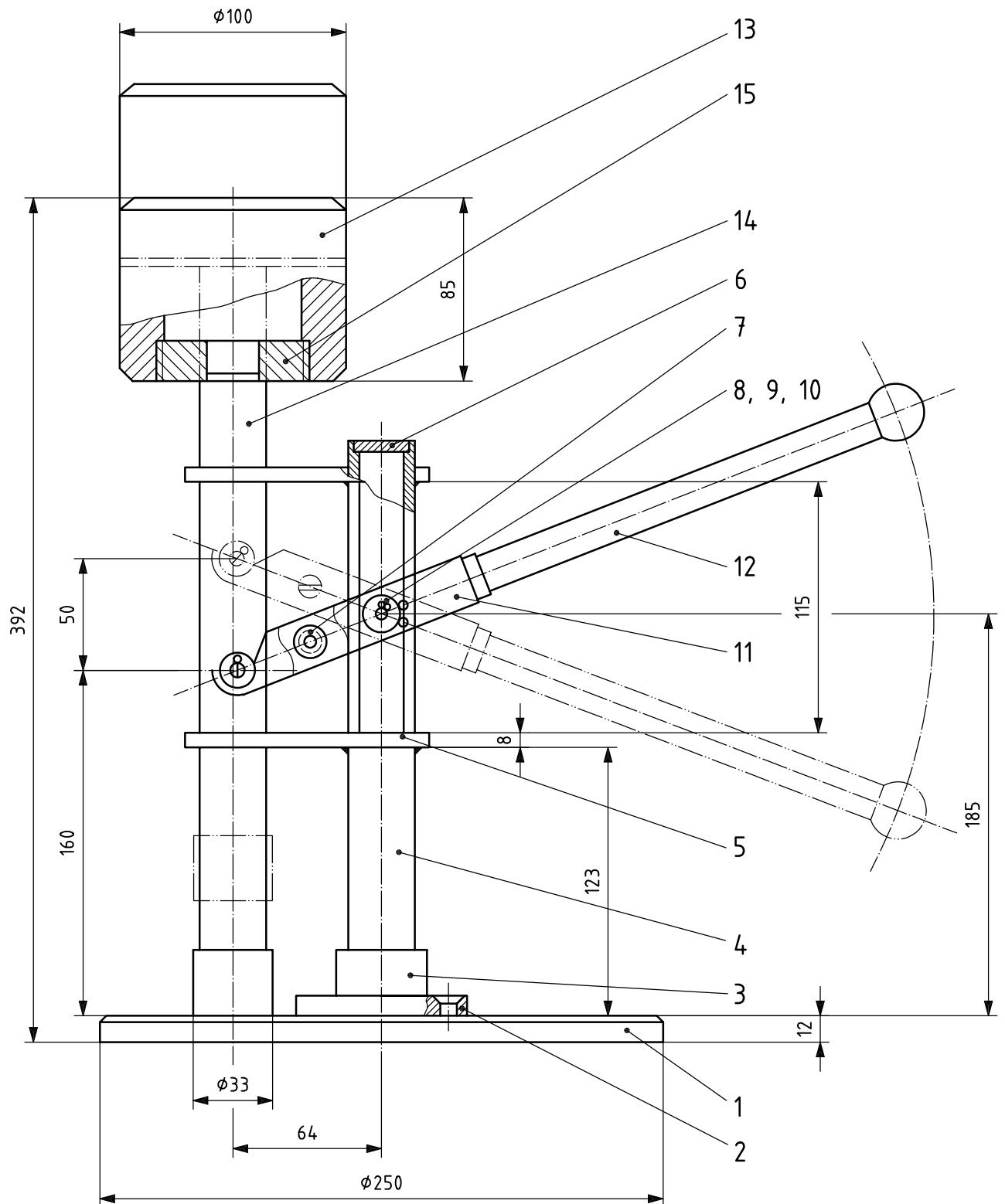


Figure 3 — Steel weight

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

1) Nichrome is an example of a suitable product available commercially. This information is given for the convenience of users of ISO 15585 and does not constitute an endorsement by ISO of this product.

Dimensions in millimetres



Key

- | | | |
|--------------------|-----------------|----------------------------|
| 1 bottom plate | 6 dead plate | 11 carriage |
| 2 screw | 7 back-up shaft | 12 hand knob |
| 3 round base | 8 small shaft | 13 press |
| 4 steel tube | 9 dump | 14 lighting vertical shaft |
| 5 connecting board | 10 forelock | 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 $850\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

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 $50 \pm 2\text{ rev/min}$.

Dimensions in millimetres

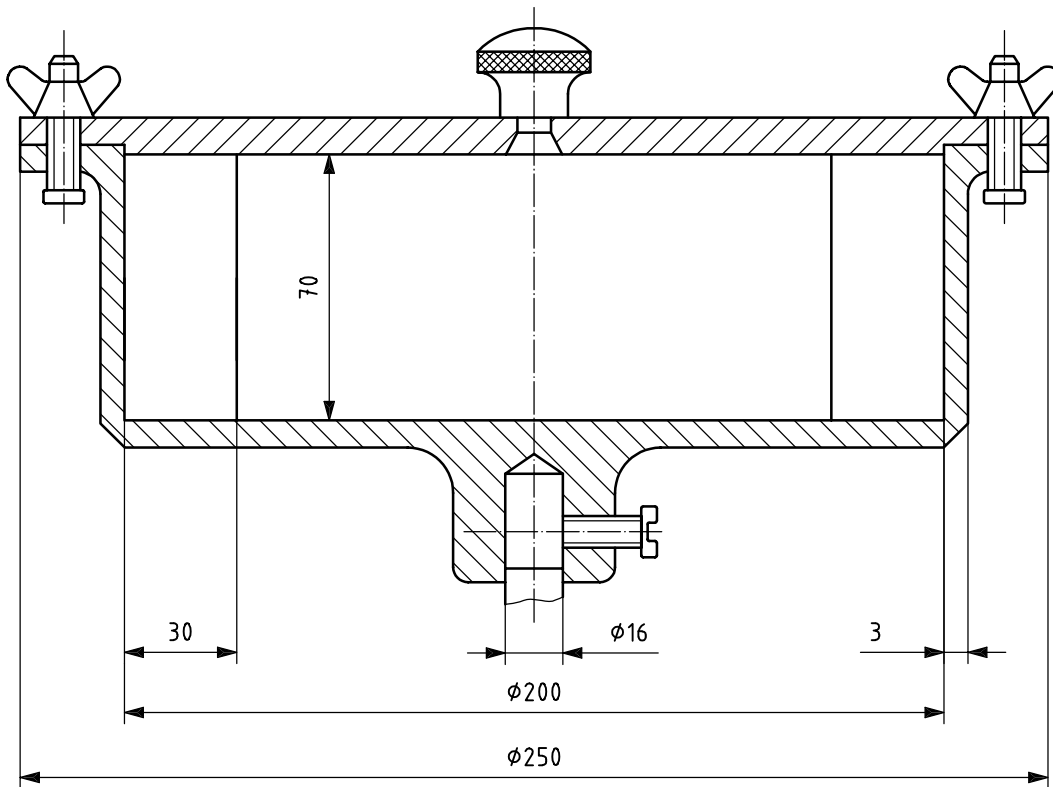


Figure 5 — Drum used for test

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 Coal 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 mass % to 40 mass % 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. The time between preparation of the sample and carrying out the test shall not exceed one week.

8 Procedure

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.

Weigh a clean, dry crucible, 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 steel weight on it. Press the whole for at least 30 s under the weight of a 6 kg mass. Remove the crucible from the press and cover it with the lid, leaving the steel weight in the crucible.

Raise the temperature of the furnace to $850\text{ °C} \pm 10\text{ °C}$ 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 $850\text{ °C} \pm 10\text{ °C}$ is regained within 6 min from the insertion of the crucible. After a total heating time of 15 min, remove the crucible from the furnace and allow it to cool on a heat resistant plate for 45 min.

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 weight and weigh the crucible plus contents to an accuracy of 0,01 g. 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 ± 2 rev/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 weighings shall be made to an accuracy of 0,01 g.

9 Calculation of result

The caking index, G , is given by Equation (1):

$$G = 10 + \frac{30 \cdot m_1 + 70 \cdot m_2}{m} \quad (1)$$

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.

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 Supplementary test

If the value of G obtained as above is less than 18, a further test should be carried out with the ratio of test sample to standard anthracite changed to 3:3, i.e. 3 g of test sample and 3 g of standard anthracite. The remaining procedure is the same as that in Clause 8. Calculation of the result is as given in Equation (2):

$$G = \frac{30 \cdot m_1 + 70 \cdot m_2}{5 \cdot m} \quad (2)$$

11 Precision

Table 1 — Precision

Caking index	Maximum acceptable difference between results	
	Same laboratory (Repeatability)	Different laboratory (Reproducibility)
≥ 18	3	4
< 18	1	2

11.1 Repeatability limit

The results of duplicate determinations, carried out at different times, in the same laboratory by the same operator with the same apparatus on the same analysis sample, should not differ by more than the above values in Table 1.

11.2 Reproducibility critical difference

The means of the results of duplicate determinations, carried out in each of two laboratories on representative portions taken from the same sample after last stage of sample preparation, should not differ by more than the above values in Table 1.

12 Test report

The test report shall include the following particulars:

- a) sample identification;
- b) reference to the method used;
- c) results, consisting of the arithmetic mean of duplicate determinations, expressed as a whole number.

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 589, ISO 1171 and ISO 562, respectively; the size analysis; 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.

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 three years.

B.1.1 Method of preparation

Four samples are extracted and prepared in accordance with Annex A . Each sample should have a mass of 4 kg and each sample should 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, 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

Each standard anthracite sample 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 averaged results of each of the eight bituminous coals are compared by statistical analysis (based on the S and the Grubbs tests) to the results of the tests on the standard anthracite; 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.

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, ash and volatile matter, should conform to the following:

$$M_{\text{ad}} = 1,5 \text{ mass \% to } 2,5 \text{ mass \%};$$

$$A_{\text{d}} = 1,5 \text{ mass \% to } 4,0 \text{ mass \%};$$

$$V_{\text{daf}} = 6,5 \text{ mass \% to } 8,0 \text{ mass \%}.$$

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

B.2.3 Scheme of test

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 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 to 3,0 S , where S is the collective standard deviation.

B.2.4 Preservation of the test samples

Keep in a dry, cool dark place, time of preservation is three years (counted from the date of issuing of verification certificate).

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