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Coke — Determination of coke reactivity index (CRI) and coke strength after reaction (CSR)

Coke — Détermination de l'indice de réactivité du coke (CRI) et de la résistance post-réactionelle du coke (CSR)

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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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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 3, *Coke*.

This second edition cancels and replaces the first edition (ISO 18894:2006), which has been technically revised.

Coke — Determination of coke reactivity index (CRI) and coke strength after reaction (CSR)

1 Scope

This document specifies the equipment and techniques used for determining lump-coke (nominal top size >20 mm) reactivity in carbon dioxide gas at elevated temperatures and its strength after reaction in carbon dioxide gas by tumbling in a cylindrical chamber.

Main application is the testing of coke to be used in iron making blast furnaces (CRI~ < 33, CSR~ > 55). This standard can also be applied to other coke types (e.g. foundry coke), but limited precision is to be expected. Application to coke for non-iron making blast furnaces is beyond the scope of this document.

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 579, *Coke — Determination of total moisture*

ISO 3310 (all parts), *Test sieves — Technical requirements and testing*

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 13909-5, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

ISO 18283, *Hard coal and coke — Manual sampling*

IEC 60584-1, *Thermocouples — Part 1: EMF specifications and tolerances*

IEC 60584-3, *Thermocouples — Part 3: Extension and compensating cables — Tolerances and identification system*

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 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
abrasion value
lack of resistance to abrasion of the coke after reaction with carbon dioxide in the CRI test, measured as the percentage of a sample passing through a 0,5 mm sieve after tumbling under conditions specified in this document

Note 1 to entry: See Annex D.

3.2
coke reactivity index
CRI
percentage weight loss of coke after reaction with carbon dioxide to form carbon monoxide under conditions specified in this document

3.3
coke strength after reaction
CSR
strength of coke after reaction with carbon dioxide in the CRI test, measured as the percentage retained on either a 10,0 mm or a 9,5 mm sieve after tumbling under conditions specified in this document

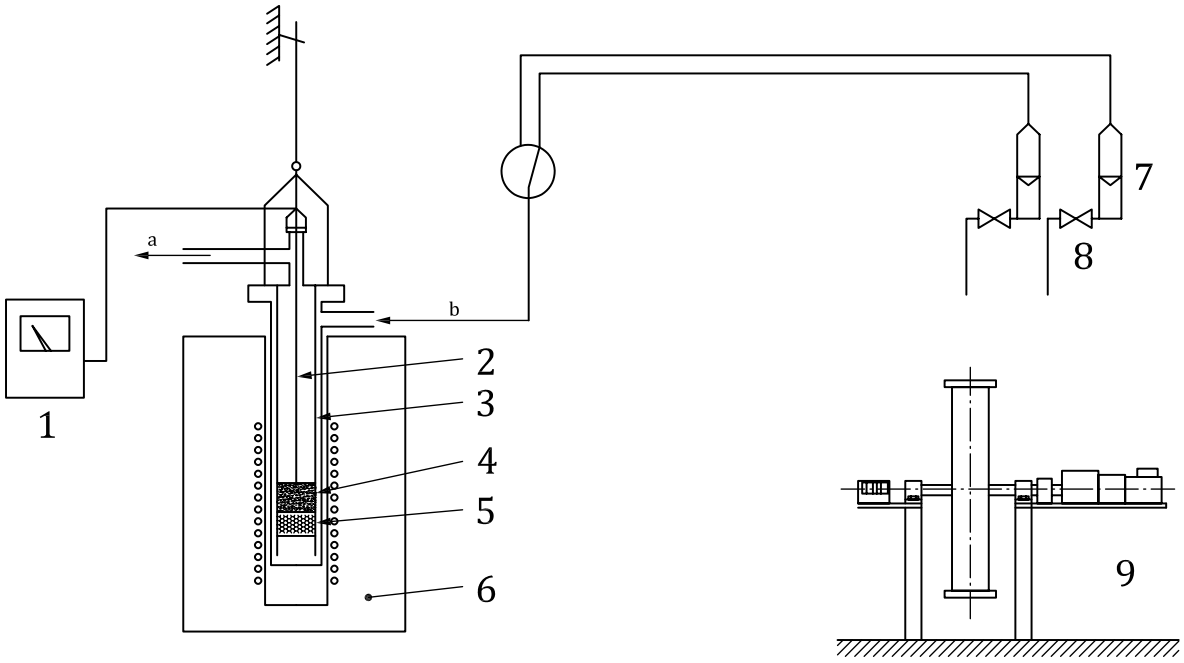
4 Principle

A test portion of the dried coke sample having a size range from 19,0 mm to 22,4 mm is heated in a reaction vessel to 1 100 °C in a nitrogen atmosphere. For the test, the atmosphere is changed to carbon dioxide for exactly 2 h. After the test, the reaction vessel is allowed to cool down to about 50 °C in a nitrogen atmosphere. The comparison of the sample weight before and after the reaction determines the coke reactivity index (CRI).

The reacted coke is treated in a specially designed tumbler for 600 revolutions for 30 min. The coke strength after reaction (CSR) value is determined by sieving and weighing the amount of coke passing through either a 10,0 mm or a 9,5 mm sieve.

An example of the arrangement of the test unit is shown in Figure 1.

NOTE During the development of this document, it was found that 10,0 mm and 9,5 mm sieves are both commonly used for these types of test. When reacted coke is tumbled, abrasion usually takes place. Particles of about 20 mm lose some edges, but they do not break into pieces. Therefore, it makes almost no difference if the sieving after tumbling is made with a 10,0 mm or a 9,5 mm sieve, as the size of the coke pieces is either about 20 mm or 0 mm to 5 mm, but not in the range of 10 mm. This has been verified by experiments over a long period of time. It has been shown that the difference in CSR using both sieve sizes is within the precision range of this document.



Key

1	device for temperature recording	6	electrically heated furnace
2	thermocouple	7	gas-flow meters
3	single or double wall retort with perforated plate as sample holder	8	control valves
4	test portion	9	tumbler
5	layer of ceramic balls		
a	Gas outlet to stack.		
b	Gas inlet.		

Figure 1 — Example of test unit arrangement

5 Reagents

5.1 Nitrogen, having a purity of >99,9 % by volume, dry and having a maximum oxygen and carbon dioxide (CO_2 + O_2) concentration of 100 mg/kg.

5.2 Carbon dioxide, having a purity of >99,5 % by volume, dry and having an oxygen concentration <100 mg/kg.

6 Apparatus

6.1 Electric furnace (see Annexes A and B), capable of housing the reaction-vessel assembly containing the test portion and providing a uniform temperature of (1 100 ± 3) °C in the centre of the test portion. The uniform temperature zone shall be at least three times longer than the sample height.

It is preferable that the furnace have independently controlled heating in three zones to achieve uniformity of heating in the reaction vessel.

6.2 Reaction vessel (see Annexes A and B), constructed from heat-resistant steel or nickel alloy to the dimensions required to fit inside the electric furnace selected for use.

The coke to be tested is placed on a perforated plate in the reaction vessel. Below this perforated plate, a gas preheater, such as a bed of ceramic Al_2O_3 balls on a second perforated plate, diffuses the nitrogen and carbon dioxide introduced into the vessel up through the coke bed during the course of the test.

Both perforated plates are fixed between two sets of lugs in the reaction vessel. The gas enters through inlets at the bottom and exits through outlets positioned at the top of the reaction vessel.

The reaction vessel is positioned such that the coke sample contained in the vessel is in the centre of the uniform temperature zone of the furnace.

6.3 Flowmeters, **variable area flowmeter** or, preferably, **mass flowmeters**, used to monitor the nitrogen and carbon dioxide flow during the test, having an accuracy of gas flow rates of ±5 % for both nitrogen and carbon dioxide.

NOTE Fluctuations in the gas flow can cause variability in the test results.

Gas pressures through the flowmeters shall be maintained at the manufacturer's calibration specification.

6.4 Thermocouple, in accordance with IEC 60584-1 and IEC 60584-3, used for measuring and controlling the sample temperature, which shall be designed according to the test conditions [e.g. platinum–rhodium/platinum (90 % Rh and 10 % Pt, percentage by mass)], enclosed in a heat-resistant steel or nickel alloy or ceramic protection tube. The protection tube shall be made of gas-tight casing to prevent faulty measurement caused by a poisoning of the thermocouple by gaseous products. The protection tube is fastened to the centre of the lid to ensure the positioning of the thermocouple tip in the centre of the coke bed.

6.5 Sieves, square hole in accordance with ISO 3310, with actual openings of 9,5 mm or 10,0 mm, 19,0 mm and 22,4 mm. A 0,5 mm sieve is also required if the abrasion test (see Annex D) is carried out.

6.6 Balance, capable of weighing to the nearest 0,1 g.

6.7 Tumbler (see Annex C), with a revolution counter and a time-relay device.

7 Preparation of test sample

The coke shall be sampled in accordance with ISO 18283 or ISO 13909-5.

Crush approximately 50 kg of the gross sample with a representative size distribution in a jaw crusher or rolling crusher. The opening of the crusher shall be set such that the gross sample yields between 10 % and 30 % of the fraction 19,0 mm to 22,4 mm.

NOTE 1 The specification for the crusher in the initial step is to avoid "over-crushing" samples. The first crushing step is without any sieving, so the opening of the crusher is set by experience. "Over-careful" crushing would reduce the size too little; therefore division of sample could easily be non-representative. "Heavy" crushing might lead to too much fines.

Divide the crushed sample to obtain a mass of approximately 25 kg in accordance with ISO 13909-6.

The mass of sample required for the test depends on the following.

a) The minimum mass required for the test is governed by the minimum mass of the 19,0 mm to 22,4 mm fraction, i.e. 1 000 g.

b) A sample of large coke shall be of sufficient size to ensure that it is representative. Therefore, smaller sample amounts (e.g. from pilot oven programs) may be used only when representativeness is guaranteed. This shall be indicated in the test report.

Sieve the crushed sample using a 22,4 mm sieve placed on top of a 19,0 mm sieve. Recycle the >22,4 mm fraction to the crusher until the oversize is less than 3 % of the crushed sample. Discard the <19,0 mm and >22,4 mm fractions.

Dry the 19,0 mm to 22,4 mm fraction in accordance with ISO 579 to less than 1 % moisture. Sieve the crushed and dried sample again using 22,4 mm and 19,0 mm sieves to remove adhering breeze. Divide the crushed and sieved sample to obtain a test sample of approximately 1 000 g.

Alternatively, the sample (fraction 19,0 mm to 22,4 mm) may be divided to approximately 1 000 g before drying and sieving.

Divide the test sample to get test portions of approximately 200 g each. For each test, prepare a test portion of 200 g ± 2 g and weigh accurately to the nearest 0,1 g. The final mass adjustment can be made by exchanging a single piece of coke for one slightly lighter or heavier as appropriate.

Record the number of pieces in each test portion.

NOTE 2 This can be helpful for comparing the test runs.

If necessary, use a magnet to remove any magnetic material from the test portions.

8 Procedure

8.1 Number of tests

A minimum of two tests shall be carried out.

8.2 Assembly of the reaction vessel

Carefully place the weighed test portion (coke pieces) on top of the perforated plate inside the reaction vessel (see Annex A for single-wall and Annex B for double-wall equipment).

Placing may be aided by the insertion of a temporary guide tube vertically into the centre of the reaction vessel and placing the test portion evenly around this temporary guide tube.

Ensure that the thermocouple sits vertically in the centre of the coke bed with its tip in the centre (at half the height of the test portion above the perforated plate) of the coke bed. With an average height of the test portion of about 100 mm, the tip of the thermocouple sits 50 mm above the perforated plate.

Place the seal and cover on top of the lower part of the reaction vessel (see Annex A for single-wall and Annex B for double-wall equipment).

8.3 Determination of CRI

CAUTION — The waste gas leaving the reaction vessel during the CO_2 gas charging is CO-rich and therefore hazardous. It should be burnt or led to a ventilated stack. Care should be taken regarding the hot surface (1 100 °C) of the reaction vessel.

Preheat the furnace to a temperature that will allow the reaction vessel and sample, when placed in the furnace, to reach (1 100 ± 3) °C within 30 min to 40 min. Connect the nitrogen gas line to the reaction vessel inlet and purge the reaction vessel for 5 min with nitrogen at 10 l/min ± 0,5 l/min before loading the vessel into the furnace. Check for the presence of leaks in the reaction vessel during this purge interval.

Place the reaction vessel in the furnace such that the centre of the coke charge is positioned in the centre of the heating zone and heat the sample to (1 100 ± 3) °C within 30 min to 40 min in the nitrogen atmosphere. Adjust the temperature to 1 100 °C; the allowed deviation of ±3 °C is used only for temperature control during the test.

Once the sample temperature of (1 100 ± 3) °C is reached, soak the reaction vessel for a further 10 min in nitrogen before switching over to carbon dioxide with a flow rate of 5 l/min ± 0,25 l/min. Keep the sample at (1 100 ± 3) °C in an atmosphere of carbon dioxide. After the switch to carbon dioxide, the temperature will drop (endothermic reaction). The heat capacity of the furnace shall be such that, with an initial temperature of (1 100 ± 3) °C, the temperature drop is minimized and the test temperature is regained within 10 min.

NOTE The temperature drop can be minimized by turning up the furnace temperature just prior to introducing the carbon dioxide. For unknown samples, this increase can be determined by experiment.

After exactly 120 min exposure to carbon dioxide, switch back to nitrogen at 10 l/min ± 0,5 l/min for 5 min to purge the reactor vessel of carbon dioxide. Remove the reaction vessel from the furnace and allow it to cool down to less than 50 °C under nitrogen flow. After cooling, remove the test portion from the reaction vessel, weigh the reacted coke to the nearest 0,1 g and calculate the CRI in accordance with 9.1.

8.4 Determination of CSR

Transfer the reacted coke completely to the tumbler, close and check for complete tightness. Tumble the residue for exactly 600 revolutions for 30 min at (20 ± 0,1) min^{-1}.

Remove all coke from the tumbler. Sieve using either a 10,0 mm or a 9,5 mm sieve and weigh the coke remaining on the sieve to the nearest 0,1 g.

To check if a loss of sample mass occurs during tumbling, also weigh the coke that passed the 10,0 mm or 9,5 mm sieve (m_3). Losses should not occur, i.e. the mass of the sample after reaction, m_1, should be equal to the sum of masses of the sized products after tumbling (m_2 + m_3). If a loss is not more than 1 % of m_1, it shall be added to the mass of the sample passing the sieve size selected, and not used in calculation of the CSR. If the loss is greater than 1 % of m_1, do not proceed to calculation of the CSR, reject the test, and correct the cause of material loss in the tumbler.

Calculate the CSR in accordance with 9.2.

9 Expression of results

9.1 Coke reactivity index (CRI)

The CRI expressed as a percentage by mass is given by Formula (1):

$$CRI = 100 \times \frac{m_0 - m_1}{m_0} \quad (1)$$

where

m_0 is the mass, in grams, of the sample before reaction;

m_1 is the mass, in grams, of the sample after reaction.

Round off the result to the first decimal place.

9.2 Coke strength after reaction (CSR)

The CSR, expressed as a percentage by mass, is given by Formula (2):

$$CSR = 100 \times \frac{m_2}{m_1} \quad (2)$$

where

m_2 is the mass, in grams, of the fraction of the sample >10,0 mm or >9,5 mm after tumbling;

m_1 is the mass, in grams, of the sample after reaction.

Round off the result to the first decimal place.

10 Precision

10.1 Quality control

Regular checking of apparatus and procedures is essential to verify the test results. The following items shall be checked at regular intervals.

a) Test sample preparation:

1) sieves;

2) balance.

b) For the reactivity test:

1) condition of the reaction vessel;

2) gas flow rate;

3) thermocouple;

4) timer.

c) For the strength test:

1) tumbler condition;

2) rotation speed;

3) revolution counter;

4) sieves;

5) balance.

It is recommended that certified calibration equipment be used for checking.

NOTE The frequency of checking the equipment lies in the responsibility of the laboratory. To give some advice the quality control plan could be as follows:

— Sieves – quarterly;

— Balances – once per shift or before each use;

— Reaction vessel – before every use;

— Gas flow rate – quarterly;

— Thermocouple – weekly;

— Time – quarterly;

— Tumbler condition – weekly;