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

*Coke — Mesures de l'indice de réactivité du coke (CRI) et de la  
résistance post-réactionnelle du coke (CSR)*

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Reference number  
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Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
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Published in Switzerland

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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 18894 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 3, *Coke*.

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

## 1 Scope

This International Standard specifies the equipment and techniques used for determining lump-coke (> 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.

## 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 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 2309, *Coke — Sampling*

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*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 and the following apply.

### 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 International Standard

NOTE 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 International Standard

## 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 International Standard

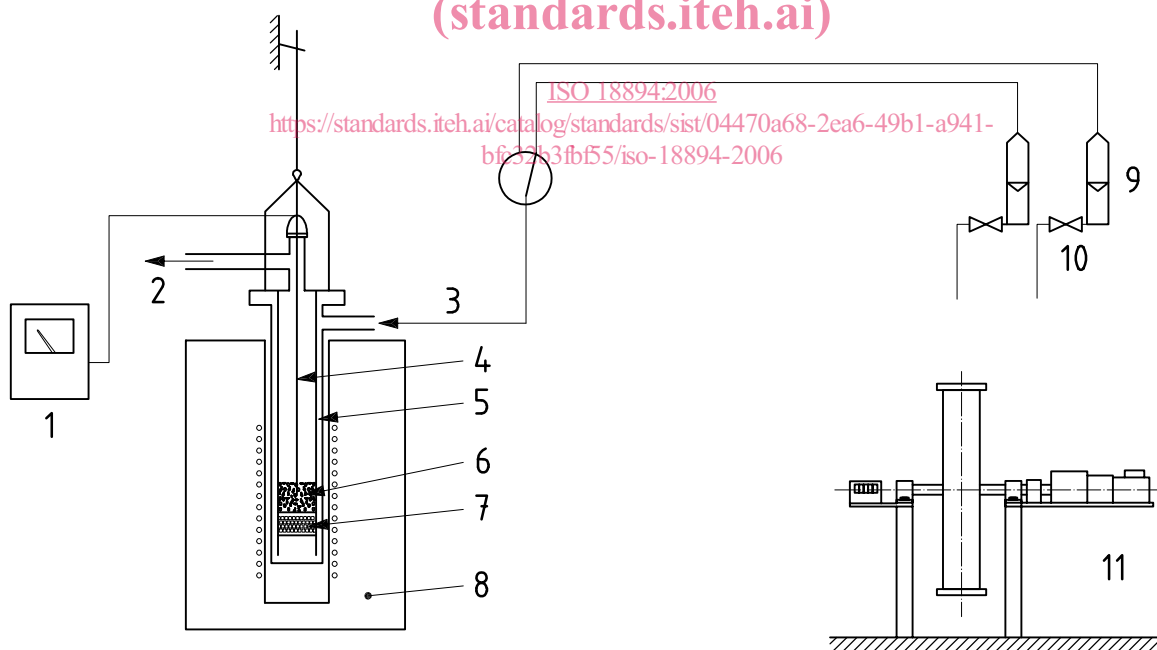
## 4 Principle

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 International Standard, 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 International Standard.

**Key**

- |   |   |    |                             |
|---|---|----|-----------------------------|
| 1 | plotter for recording temperature                                   | 7  | layer of ceramic balls      |
| 2 | gas outlet to stack   | 8  | electrically heated furnace |
| 3 | gas inlet   | 9  | gas-flow meters             |
| 4 | thermocouple  | 10 | control valves              |
| 5 | single or double wall retort with perforated plate as sample holder | 11 | tumbler                     |
| 6 | test portion  |    |                             |

**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 ( $\text{CO}_2 + \text{O}_2$ ) 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  $(1\,100 \pm 3)^\circ\text{C}$  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  $\text{Al}_2\text{O}_3$  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  $\pm 5$  % 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**, conforming to the requirements of IEC 60584-1 and IEC 60584-2, 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 conforming to the requirements of 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

Sample the coke in accordance with ISO 2309 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. 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 must be of sufficient size to ensure that it is representative. Therefore, smaller sample amounts may be used only when it is guaranteed that they are representative. 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  $200 \text{ g} \pm 2 \text{ g}$  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.

NOTE Recording the number of pieces in each test portion can be helpful for comparing the test runs.

## 8 Procedure

### 8.1 Number of tests

A minimum of two tests shall be carried out.

### 8.2 Determination of CRI

**CAUTION —** The waste gas leaving the reaction vessel during the CO<sub>2</sub> 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  $(1\,100 \pm 3)^\circ\text{C}$  within 30 min to 40 min. Before the reaction vessel is placed in the electric furnace, place the weighed sample in the reaction vessel in a manner so as to 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. Purge the reaction vessel for 5 min with nitrogen at  $10 \text{ l/min} \pm 0,5 \text{ l/min}$  before loading the vessel into the furnace.

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  $(1\,100 \pm 3)^\circ\text{C}$  within 30 min to 40 min in the nitrogen atmosphere.



Adjust the temperature to  $1\,100\text{ }^{\circ}\text{C}$ ; the allowed deviation of  $\pm 3\text{ }^{\circ}\text{C}$  is used only for temperature regulation during the test.

Once the sample temperature of  $(1\,100 \pm 3)\text{ }^{\circ}\text{C}$  is reached, soak the reaction vessel for a further 10 min in nitrogen before switching over to carbon dioxide with a flow rate of  $5\text{ l/min} \pm 0,25\text{ l/min}$ . Keep the sample at  $(1\,100 \pm 3)\text{ }^{\circ}\text{C}$  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  $(1\,100 \pm 3)\text{ }^{\circ}\text{C}$ , 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  $10\text{ l/min} \pm 0,5\text{ l/min}$  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  $50\text{ }^{\circ}\text{C}$  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.

### 8.3 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  $(20 \pm 0,1)\text{ min}^{-1}$ .

Remove all coke from the drum. 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. Calculate the CSR in accordance with 9.2.

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### 9 Expression of results (standards.iteh.ai)

#### 9.1 Coke reactivity index (CRI)

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The CRI expressed as a percentage by mass is given by Equation (1).  
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$$\text{CRI} = 100 \times \frac{m_0 - m_1}{m_0} \quad (1)$$

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.

#### 9.2 Coke strength after reaction (CSR)

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

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

where

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

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

## 10 Precision

### 10.1 Verification

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.

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It is recommended that certified calibration equipment be used for checking and that internal reference material be prepared and used periodically to verify repeatability and reproducibility.

See also Annex E.

## 10.2 Repeatability limit

### 10.2.1 General

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 shown in Tables 1 and 2.

### 10.2.2 Coke reactivity index

For a paired result, the value of the range  $|X_1 - X_2|$  shall govern whether additional tests are required as specified in Table 1 and 10.2.2 a) to 10.2.2 c).

**Table 1 — Criteria for multiple determinations (CRI)**

CRI	Range $ X_1 - X_2 $		
	A	B	C
$\leq 10$	—	—	—
$> 10 \leq 20$	2,0	2,5	2,7
$> 20 \leq 30$	2,5	3,2	3,5
$> 30 \leq 40$	3,0	4,0	4,5
$> 40 \leq 60$	3,5	5,0	5,5
$> 60$	—	—	—

#### a) Given two results

- If range  $|X_1 - X_2| \leq A$ , average the two results.
- If range  $|X_1 - X_2| > A$  and range  $|X_1 - X_2| \leq B$ , perform a third test.
- If range  $|X_1 - X_2| > B$ , perform two more tests.

#### b) Given three results

- If  $X_{(\max)} - X_{(\min)} \leq B$ , average the three results.
- If  $X_{(\max)} - X_{(\min)} > B$ , perform a fourth test.

#### c) Given four results

- If  $X_{(\max)} - X_{(\min)} \leq C$ , average the four results.
- If  $X_{(\max)} - X_{(\min)} > C$ , discard  $X_{(\max)}$  and  $X_{(\min)}$  and average the remaining two results.

The mean index shall be rounded off to the first decimal place.