Designation: D5341 – 99 (Reapproved 2010)

Standard Test Method for Measuring Coke Reactivity Index (CRI) and Coke Strength After Reaction (CSR)[1]

This standard is issued under the fixed designation D5341; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method, patterned after the Nippon Steel test procedure, describes the equipment and techniques used for determining lump coke reactivity in carbon dioxide (CO_2) gas at elevated temperatures and its strength after reaction in CO_2 gas by tumbling in a cylindrical chamber referred to as an I-tester.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *British Carbonization Research Association Report:*

Carbonization Research Report 91, "The Evaluation of the Nippon Steel Corporation Reactivity and Post-Reaction-Strength Test for Coke."[3]

3. Summary of Test Method

3.1 A sample of dried coke of designated origin and size is reacted with CO_2 gas in a retort at a specified elevated temperature for a specified length of time. Two indices, coke reactivity index (CRI) and coke strength after reaction (CSR), are determined using the reacted coke residue. The weight loss after reaction determines the CRI. The weight retained after sieving the tumbled reacted coke in a designated number of revolutions over a designated turning rate determines the CSR.

4. Significance and Use

4.1 When coke lumps descend in the blast furnace, they are subjected to reaction with countercurrent CO_2 and to abrasion as they rub together and against the walls of the furnace. These concurrent processes physically weaken and chemically react with the coke lumps, producing an excess of fines that can decrease burden permeability and result in increased coke rates and lost hot metal production. This test method is designed to measure indirectly this behavior of coke in the blast furnace.

5. Apparatus

5.1 *Electric Furnace* (Fig. 1), capable of housing the reaction vessel assembly containing the coke sample and providing a uniform temperature of 1100 ± 5°C in the assembly. Furnace dimensions do not impact the test results and may vary from 240 to 1035 mm in length and 76.2 to 88.9 mm in outside diameter. However, it is preferable that the furnace have independently controlled heating in three zones to achieve uniformity of heating in the retort and that this control be achieved with a programmable controller.

5.2 *Reaction Vessel* (Fig. 1), constructed of a heat-resistant steel or nickel alloy to the dimensions required to fit snugly inside the electric furnace selected for use (Note 1). The coke to be tested sits on a porous plate in the reaction vessel. Below this porous plate, a gas preheater, such as a bed of ceramic Al_2O_3 balls sitting on a second perforated plate, diffuse the nitrogen (N_2) and carbon dioxide introduced into the vessel up through the coke bed during the course of the test. The gas enters through inlets and exits through outlets varying from 6 to 15 mm in inside diameter and positioned at the top and bottom of the reaction vessel. During the test, it is important that no backpressure be detected when gas enters or exits through these inlets or outlets. The reaction vessel is positioned such that the coke sample contained in the vessel on top of the ceramic Al_2O_3 balls is in the center of the controlled temperature zone in the furnace.

[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.15 on Metallurgical Properties of Coal and Coke.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from British Carbonization Research Association, Chesterfield, Derbyshire, England.

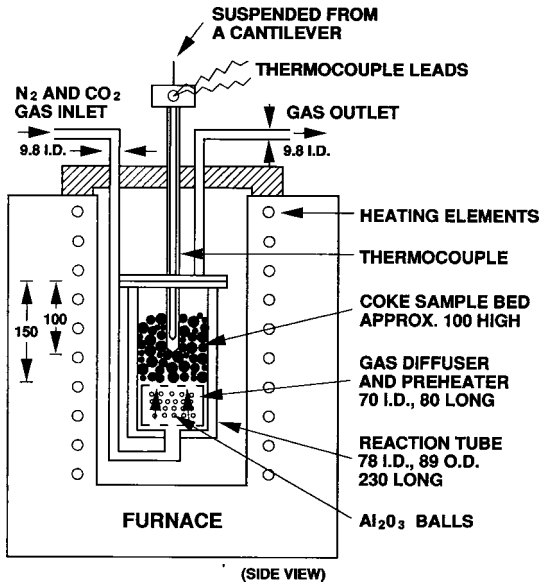

FIG. 1 Example of Reactivity Test Apparatus (Dimensions in mm)

NOTE 1—Inconel 601 is recommended over stainless steel. Inconel 601 does not leave scale, that, if not properly removed, can alter a coke sample weight after the test.

5.3 *Flowmeters*—Rotometers or, preferably, mass flowmeters shall be used to monitor the amount of N_2 and CO_2 gases used in the test. The accuracy of measuring gas flowrates should be ±1 % of full scale since varying gas flow can cause variability in the test results. Gas pressures through the flowmeters should be maintained at the manufacturer's calibration specification.

5.4 *Thermocouple* (Fig. 1), of the K, S, or R type normalized at 20 to 21°C and enclosed in a heat-resistant steel or nickel alloy or ceramic protection tube placed in the center of the coke sample in the reaction vessel. A centering pipe or tube also made of heat-resistant material is used to guide the thermocouple into its proper location in the coke bed.

5.5 *Sieves*, used for sieving the coke during its preparation for reactivity testing and after tumbling for strength after reaction testing. Square mesh sieves having 22.4-, 19.0-, and 9.5-mm actual openings between the wires are to be used. Standard test sieves that conform to Specification E11 should always be used.

5.6 *Balance*, capable of weighing up to 25 kg and sensitive to 0.1 g (0.000 22 lbs).

5.7 *Coke Strength After Reaction Tumbler* (Fig. 2), consisting of a cylindrical chamber with an internal length of 700 ± 10 mm and an internal diameter 130 ± 5 mm, with end caps of 10-mm thickness or more (Note 2). This cylindrical chamber is attached to its longitudinal center to an electric motor fitted with a direct drive fixed gearbox, a drive belt, or, preferably, a hydraulic drive set for a revolving rate of 20 ± 1 r/min (Note 3). A revolution counter is fitted so that the power is cut off when the cylinder has revolved 600 times in 30 min.

NOTE 2—Mild carbon steel should be selected to fabricate this tumbler apparatus.

NOTE 3—Most Japanese publications refer to this as an $I_{600/10}$ test.

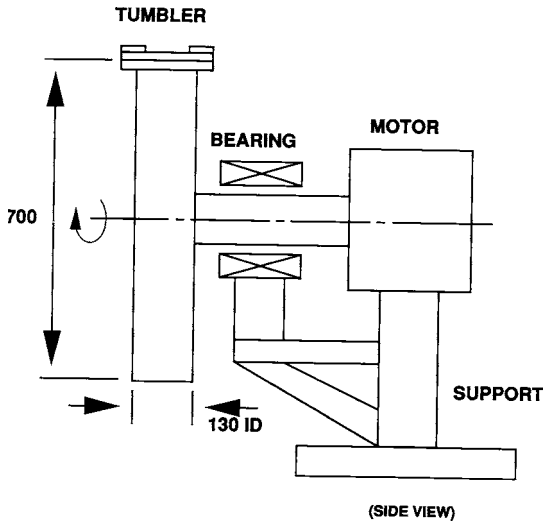

FIG. 2 Example of I-Type Coke Tumbler (Dimensions in mm)

6. Sampling

6.1 The gross sample of coke shall be collected in accordance with Test Method D346.

6.2 For the standard procedure, the quantity must be not less than 57 kg (125 lbs).

7. Preparation of Sample

7.1 Sieve the gross sample at 25.0 mm (1 in.) and discard the undersize.

7.2 With suitable crushing equipment, preferably a jaw or roll crusher, reduce the size of all of the remaining plus 25.0 mm (1 in.) to pass a 22.4-mm (⅞-in.) sieve opening.

7.3 Sieve the crushed sample using a 22.4-mm (⅞-in.) sieve placed on top of a 19.0-mm (¾-in.) sieve. Discard the minus 19.0-mm (¾-in.) coke, and retain the 22.4- by 19.0-mm (⅞- by ¾-in.) fraction for testing.

7.4 The size reduction of the plus 25.0 mm (1 in.) should be accomplished in stages by recrushing any plus 22.4-mm (⅞-in.) coke remaining after each subsequent double sieving step until there is no oversize retained on the 22.4-mm (⅞-in.) sieve. The opening to the crusher should be set such that the gross sample yields at least 10 % of 19.0- by 22.4-mm (¾- by ⅞-in.) test coke (Note 4).

NOTE 4—The size of the sample required for most coke tests depends on collecting sufficient received material to have sufficient natural sample for testing, that is, stability 75.0 by 50.0 mm (3 by 2 in.). For the CSR tests, most companies crush as-received coke to yield a 19.0- by 22.0-mm product. Therefore, 57 kg (125 lbs) of gross sample is not necessarily required. In fact, Nippon Steel Corporation, the originator of the test, believes 10-kg (22-lbs) sublots of the gross sample is representative of the gross sample collected and is sufficient, with suitable crushing and sieving equipment, to yield enough 19- by 22-mm coke to provide repeatable results.[4]

7.5 Using a riffle splitter, subdivide the 19.0- by 22.4-mm (¾- by ⅞-in.) coke into three test samples, each weighing approximately 250 g (0.55 lbs).

[4] Nishi, T., et al, *Journal of the Fuel Society of Japan* , Vol 61, No 668, 1982, pp 1066-1073.

7.6 Dry the test coke samples to less than 1 % moisture at 150°C for 2 h.

8. Procedure

8.1 From each test coke sample prepared in accordance with Section 7, randomly hand pick and accurately weigh to the nearest 0.1 g (0.0002 lb), a 200- ± 2.0-g sample for testing. Record the number of pieces selected from each test sample.

8.2 Before the reaction vessel is put into the electric furnace, place the weighed sample in the reaction vessel in a manner as to ensure that the thermocouple sits vertically in the center of the coke bed with its tip 50 mm from the bottom of the coke bed. A centering guide normally fastened to the center of the lid is used to ensure this positioning of the thermocouple tip.

NOTE 5—Variations in coke density may result in different total coke bed heights in the reaction vessel. Therefore, in situations where 50 mm is not the center of the coke bed, adjust the thermocouple tip accordingly.

8.3 Purge the reaction vessel for 5 min at 5 to 10 L/min of N_2 before loading the vessel into the furnace. Check for leaks in the assembly during this purge time.

8.4 Preheat the furnace to a temperature that will allow the sample, when the sample is loaded into the furnace, to reach 1100 ± 5°C in 30 min.

8.5 Place the reaction vessel into the furnace and heat the sample to 1100°C in the atmosphere of N_2 gas. Once the sample temperature of 1100 ± 5°C is reached, soak the sample for 10 min in N_2 gas for a total heat up time of 40 min. Then heat the samples for 120 min in an atmosphere of CO_2 gas, with a flow rate of 5.0 L/min ± 1.0 % (Note 6). Maintain the coke bed temperatures at 1100°C ± 5°C during the test. (Fig. 3)

8.6 After exactly 120-min exposure to CO_2 gas, switch back to the N_2 purge gas at 5 to 10 L/min for 5 min to purge the reactor vessel of CO_2. Subsequently, remove the reaction vessel from the furnace, and allow the sample temperature to cool to 100°C.

8.7 After cooling, remove the coke sample from the reaction vessel and weigh the coke to the nearest 0.1 g (0.0002 lbs).

8.8 Transfer the reacted coke to the strength after reaction tester and tumble for 600 revolutions in 30 min at 20 ± 1 r/min.

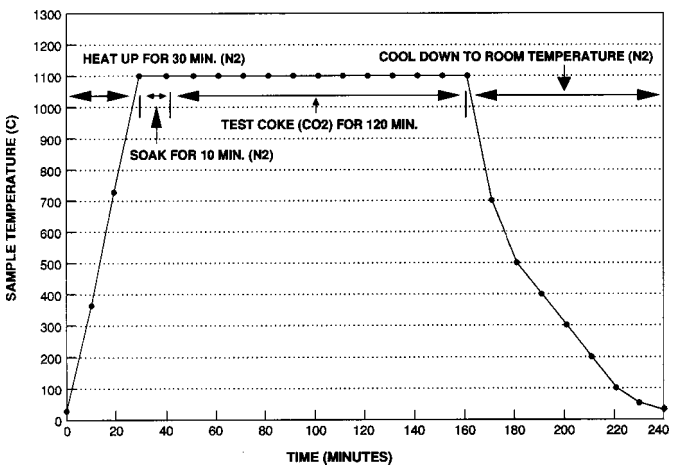

FIG. 3 CSR Test Procedure

8.9 After the 600 revolutions, remove all coke from the drum. Sieve the coke using a 9.5-mm (⅜-in.) sieve. Weigh the coke remaining on the 9.5-mm (⅜-in.) sieve for calculation of CSR. Weigh the coke passing the 9.5-mm (⅜-in.) sieve for checking material losses during tumbling.

NOTE 6—Since carbon monoxide (CO) is generated during the coke reactivity test, the test apparatus should be placed in an area with proper ventilation. In addition, a CO monitoring device should be used to detect any unsafe buildup of CO gas.

9. Number of Tests

9.1 Reactivity and strength after reaction tests should be determined in duplicate for each coke sample.

9.2 If the difference between the weights of coke remaining after reaction with CO_2 gas or after tumbling and sieving on the 9.5-mm (⅜-in.) sieve exceed 10 g (0.022 lbs), conduct one additional test and report the mean value of all three tests.

10. Calculation

10.1 For each test, calculate, to the nearest 0.1 %, the percentage of coke remaining after reaction in CO_2 gas and that remaining after tumbling on the 9.5-mm (⅜-in.) sieve. Report the average of these values for the two or more tests as the CRI and CSR values, respectively.

10.1.1 Calculations are made as follows:

$$CRI = \frac{A - B}{A} \times 100 \quad (1)$$

$$CSR = \frac{C}{B} \times 100 \quad (2)$$

where:

A = original test sample weight before reaction,
B = sample weight after reaction in CO_2, and
C = sample weight of +9.5-mm (⅜-in.) material after tumbling.

11. Precision and Bias

11.1 *Precision*—The relative precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials to be:

CRI

Material	Average	S_r	S_R	r	R
Sample 2	20.47	0.54	2.23	1.51	6.26
Sample 5	21.17	1.53	2.07	4.30	5.78
Sample 6	29.53	0.90	2.00	2.53	5.59
Sample 3	29.84	1.28	2.29	3.58	6.41
Sample 1	32.98	0.52	2.84	1.45	7.95
Sample 4	53.00	0.84	3.46	2.36	9.70

CSR

Material	Average	S_r	S_R	r	R
Sample 4	26.94	1.21	4.46	3.36	12.49
Sample 3	30.73	3.19	3.34	8.94	9.34
Sample 1	52.68	0.79	2.23	2.22	6.25
Sample 6	55.89	1.82	2.55	5.08	7.13
Sample 2	63.84	2.36	3.17	6.62	8.87
Sample 5	65.96	1.15	1.75	3.22	4.90

The relative precision of this test method for the determination of CRI and CSR of coke covers the range of 20 to 50 for CRI and 27 to 66 for CSR.

11.1.1 *Repeatability*—The difference in absolute value between two results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus, may be expected to occur with a probability of approximately 0.95 (95 % confidence level).

The repeatability limit for this method is (see Note 7):

CRI	2.8
CSR	5.4

11.1.2 *Reproducibility*—The difference in absolute value of replicate determinations, carried out in different laboratories using samples taken at random from a single sample prepared from the same bulk sample after the last stage of reduction, may be expected to occur with a probability of approximately 0.95 (95 %) confidence level).

The reproducibility limit for this method is (Note 7):

CRI	$R = 3.21 + 0.12\ \bar{x}$
CSR	$R = 14.4 - 0.13\ \bar{x}$

where $\bar{x}$ is the average of two single between-laboratory results.

NOTE 7—An interlaboratory study, designed consistent with Practice E691, was conducted in 1997. Seven laboratories participated. The details of the study and supporting data are given in ASTM Research Report D05–1022 filed at ASTM Headquarters.

11.2 *Bias*—Certified standard reference materials are not available for the determination of bias by this test method.

12. Keywords

12.1 coke; coke reactivity index; coke strength after reaction