ICS 75.160.10
CCS H 32

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 44037-2024

Test method for coke solution loss percentage and strength after loss

焦炭溶损率及溶损后强度试验方法

Issued on: May 28, 2024 **Implemented on: December 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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0 Warning

The document opens with a warning that its use may involve hazardous materials, operations and equipment, that it does not offer advice on every safety problem connected with them, and that the user is responsible for adopting suitable safety and protective measures beforehand and for complying with the conditions laid down in national regulations.

3 Terms and definitions

3 adopts the terms of GB/T 9977 and adds three of its own.

3.1 coke solution loss percentage, abbreviated CSP: the percentage of the total mass that the coke loses in the solution loss reaction.

3.2 coke strength after solution loss reaction, abbreviated CSS: the mechanical strength of the coke after the solution loss reaction, that is its ability to resist breakage and abrasion.

3.3 coke solution loss rate, abbreviated CLR: the speed at which the coke loses mass through its reaction with carbon dioxide gas at high temperature. In this copy the English term printed beside the Chinese entry runs together as one unspaced string and cannot be resolved with certainty into words; it is therefore not reproduced here, and only the abbreviation CLR is given.

4 Test instruments, equipment and materials

4.1 the electric furnace provides a uniform temperature field for the sample, with a heating zone of not less than 600 mm and a working temperature of not less than 1350 °C.

4.2 the temperature control device controls and displays the temperature at the centre of the coke bed under test conditions and raises the temperature at the rate laid down; from 800 °C to 1300 °C the control accuracy is +/-3 °C.

4.3 the reactor is shown in Figure 1, whose legend names the reactor lid, the coke sample, the supporting sieve plate, the reactor, the sieve plate frame, the thermocouple, the gas inlet and the gas outlet, and marks the height of the coke sample, the thermocouple measuring point being at the centre of the sample. The reactor is made of silicon carbide or another refractory with a working temperature of not less than 1350 °C. The tube wall is 5 mm +/-2 mm thick, the internal diameter is 80 mm +/-1 mm and the length not less than 200 mm. The supporting sieve plate is 78 mm +/-1 mm in diameter and carries 273 evenly distributed through holes of 2 mm +/-0.5 mm diameter at a spacing of 4 mm, with a working temperature of not less than 1350 °C.

4.4 the type I drum is shown in Figure 2, whose legend names the drum, the coupling, the geared motor, the frame, the counter and the drum lid. It runs at a constant 20 r/min. The body is made from seamless steel tube of 130 mm internal diameter and 5 mm to 6 mm wall thickness, the clear length inside is 700 mm +/-1 mm and the lid is not less than 6 mm thick. The number of revolutions and the wear of the drum are checked periodically, and the drum is repaired or replaced if 600 revolutions take more than 30 min +/-1 min or if the body no longer meets 4.4.3.

4.5 the carbon dioxide supply system uses carbon dioxide of not less than 99.9 % purity, dried before the test, with an oxygen concentration below 0.01 %; where the purity falls short, the purification and drying apparatus of Annex B of GB/T 4000-2017 may be used. The mass flow controller has a range of 10 L/min and an accuracy of not less than +/-1.5 % of full scale, and a heated pressure-reducing valve for carbon dioxide is fitted at the inlet.

4.6 the nitrogen supply system uses nitrogen of not less than 99.99 % purity, likewise dried, with the same purification option, a mass flow controller of 10 L/min and +/-1.5 % of full scale, and a nitrogen pressure-reducing valve at the inlet. A note states that the gas volumes and flow rates used in the document are those at the standard state, 0 °C and 0.101325 MPa.

4.7 the round-hole screens serve to size the coke for the solution loss test and to screen the coke after the drum test; the holes are round and Table 1 fixes, in millimetres, five combinations of hole diameter and screen surface size: hole 10 with a screen face 200 in diameter, hole 23 with a face 300 in diameter, hole 25 with a face 300 in diameter, hole 23 with a face 400 by 500, and hole 25 with a face 400 by 500.

4.8 the drying oven is a forced-air oven of not less than 0.07 cubic metre, controlling from room temperature to 250 °C with an accuracy of +/-1 °C. 4.9 the weighing balance has a scale division of 0.1 g and the thermobalance one of 0.01 g. 4.10 the rhodium-platinum thermocouple is of type S and of not lower than industrial grade II.

4.11 requires the stability of the equipment to be checked periodically with a reference sample by the test method of GB/T 4000, and immediately after a major overhaul or the replacement of an important part such as the furnace chamber, a heating element, the reactor or the centre thermocouple.

4.12 shows the coke solution loss measuring system in Figure 3, whose legend names the carbon dioxide cylinder, the nitrogen cylinder, the pressure-reducing valve, the flowmeter, the thermobalance, the gas inlet, the centre thermocouple, the furnace chamber, the heating element, the sieve plate frame, the coke sample, the reactor, the electrical control system, the side-wall thermocouple and the computer.

5 Sampling and preparation of the test sample

5.1 takes 60 kg of representative coke larger than 25 mm by the sampling method of GB/T 1997, discarding the honeycombed pieces with large pores and the oven-mouth coke that is black or incompletely grey.

5.2 crushes the coke as laid down in YB/T 4494 and screens it on a 25 mm sieve; coke larger than 25 mm is crushed again and again until the proportion above 25 mm is less than 3 %, and the coke above 25 mm and below 23 mm is then removed. To keep the sample representative, the coke between 23 mm and 25 mm is to make up more than 10 % of the total mass sampled; where it does not, the fact and the reason are to be noted in the report.

5.3 divides out 1000 g of the prepared coke and dries it at 170 °C to 180 °C for 2 h, or rapidly according to Annex A, until the moisture is not more than 1 %. The dried sample is cooled to room temperature in a desiccator, screened again on the 23 mm and 25 mm sieves to remove the coke dust clinging to the lumps, and divided by quartering into four portions of not less than 220 g each, which are kept in sealed containers.

6 Test procedure

6.1 requires at least two tests on each coke sample.

6.2 weighs out 200 g +/-2 g of sample to 0.1 g and records it as the initial mass, the final adjustment being made by replacing a lighter or heavier lump. The number of lumps used in each test is recorded, and the number is not to differ by more than one lump between the tests used for repeatability or reproducibility; where it does, the fact and the reason are noted in the report.

6.3 loads the sample into the reactor and levels it, making sure that the coke bed sits in the middle of the temperature zone of the furnace, inserts the thermocouple into the reactor at the centre of the bed, fixes the reactor, connects the inlet to the gas supply and checks the gas tightness of the system.

6.4 controls the reaction temperature. The furnace is heated; when the centre of the bed reaches 400 °C nitrogen is admitted at 5 L/min to keep the coke from burning; when it reaches 800 °C the thermobalance reading is recorded, the temperature is raised at 4 °C/min, the nitrogen is cut off and carbon dioxide is admitted at 5 L/min. The thermobalance weighs at least once every 30 s; the point at which the coke has lost 1 % of its mass is taken as the start of the reaction, and the time from that point to the end of the reaction at 1300 °C is the solution loss reaction time. When the centre of the bed reaches 1300 °C the heating is stopped, the carbon dioxide is cut off and nitrogen is admitted at 5 L/min. Between 800 °C and 1300 °C the temperature is recorded every 10 min, the difference between the actual and the set temperature is not to exceed +/-5 °C, and the reaction time is 125 min.

6.5 cools the bed under nitrogen to below 50 °C, after which the nitrogen is stopped and the reactor taken out. 6.6 tips out all the reacted coke, weighs it to 0.1 g and records the mass and the number of lumps.

6.7 loads all the reacted coke into the type I drum and turns it at 20 r/min for 30 min, that is 600 revolutions, then screens it on the 10 mm round-hole screen, weighs the oversize to 0.1 g and records the mass and the number of lumps. 6.8 refers the recording of the raw data to the form of Annex B.

7 Calculation of results

7.1 gives formula (1) for the coke solution loss percentage, expressed as a percentage. The printed equation is broken by the extraction and is not reproduced here; its legend reads: the mass of the sample before the solution loss reaction, in grams, and the mass after it, in grams.

7.2 gives formula (2) for the strength after solution loss, also as a percentage, its legend adding the mass of coke above 10 mm after the drum test, in grams. The results of both indices are taken as the arithmetic mean of the parallel tests and kept to one decimal place.

7.3 gives formula (3) for the coke solution loss rate, whose legend adds the time from the initial reaction temperature to the end of the reaction, in minutes. The value is expressed with a factor of ten to the power three.

8 Precision

8 requires the repeatability of the two indices not to exceed the values of Table 2, which has two columns and two rows: a repeatability of not more than 2.4 % for the coke solution loss