ICS 71.100.10
CCS Q 52

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 26930.8-2014

Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Part 8: Determination of coking value

原铝生产用炭素材料 煤沥青 第8部分：结焦值的测定

Issued on: July 24, 2014 **Implemented on: April 1, 2015**

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

Table of Contents

Foreword Foreword

GB/T 26930, Carbonaceous materials used in the production of aluminium - Pitch for electrodes, is divided into 13 parts: Part 1: Determination of the water content - Azeotropic distillation method; Part 2: Determination of the softening point - Ring-and-ball method; Part 3: Determination of the density - Pyknometer method; Part 4: Determination of the quinoline-insoluble content; Part 5: Determination of the toluene-insoluble content; Part 6: Determination of ash; Part 7: Determination of the softening point - Mettler method; Part 8: Determination of the coking value; Part 9: Determination of the sulfur content by oxygen bomb combustion; Part 10: Determination of the sulfur content by the instrumental method; Part 11: Determination of the dynamic viscosity; Part 12: Determination of the volatile matter content; Part 13: Determination of the C/H atomic ratio in the quinoline insolubles. This part is Part 8 of GB/T 26930.

This part is drafted according to the rules given in GB/T 1.1-2009. It adopts ISO 6998:1997, Carbonaceous materials for the production of aluminium - Pitch for electrodes - Determination of coking value, by the translation method and is identical with it. The Chinese document having a consistent correspondence with the international document referred to normatively in this part is GB/T 26297.5-2010, Sampling method for carbonaceous materials used for aluminium - Part 5: Coal tar pitch (ISO 6257:2002, MOD).

This part is under the jurisdiction of the National Technical Committee on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

1 Scope

This part of GB/T 26930 fixes the method for determining the coking value of the coal tar pitch used in the production of primary aluminium.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

ISO 565:1990 Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings; ISO 5725-1:1994 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions; ISO 6257 Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Sampling.

3 Principle of the method

The test portion is heated for 2.5 h in a furnace at 550 °C +/- 10 °C and the residue is weighed.

4 Filler

Calcined petroleum coke: the fraction of particle size 212 μm to 1 mm (see ISO 565:1990) is used.

5 Apparatus

5.1 Porcelain crucible: of 25 mL to 50 mL capacity, the ratio of the height to the outside diameter at the top being 0.7 to 0.9, fitted with an inset lid.

5.2 Nickel crucible: of about 130 mL capacity, both the height and the diameter being 60 mm, fitted with a lid.

5.3 Stainless steel support (see Figure 1): it sets the porcelain crucible correctly inside the nickel crucible so that the bottoms of the two crucibles are 10 mm +/- 1 mm apart.

5.4 Nickel crucible rack (see Figure 2): made of stainless steel plate 1 mm thick, with four holes. The edge of the rack is bent downwards and four legs are welded at the corners, so that when a nickel crucible is set in one of the holes the bottom of the crucible is kept at least 7 mm above the surface of the heating plate of the furnace.

5.5 Test sieves: of nominal sizes 1 mm, 300 μm and 212 μm respectively (see ISO 565:1990).

5.6 Muffle furnace: able to be held steadily at 550 °C +/- 10 °C.

Figure 1 is a schematic drawing of the nickel crucible and the porcelain crucible assembled together, with the key naming the stainless steel support (5.3), the calcined petroleum coke (Clause 4), the nickel crucible (5.2), the porcelain crucible (5.1) and the test portion (7.2), and carrying the dimension 10 mm +/- 1 mm. Figure 2 is a schematic drawing of the nickel crucible rack, carrying the dimension not less than 7.

6 Sampling and preparation of the test portion

6.1 Sampling. Sampling shall be carried out according to the procedure laid down in ISO 6257.

6.2 Preparation of the test portion. When the sample is rather hard it may be crushed in a small jaw crusher and ground in a mortar until it passes at least the 300 μm sieve, and if possible the 212 μm sieve. When the ambient temperature is too high the sample may be pre-cooled to make the operation easier. When the pitch is too soft to be crushed, the sample may be melted and a sufficient quantity of the melted sample taken for the determination; the melting temperature shall not be above 150 °C and the melting time shall not exceed 10 min. Soft pitch to be determined may also be transferred directly into the porcelain crucible without any pre-treatment.

7 Determination procedure

7.1 Number of determinations. Two parallel determinations are carried out, and two crucibles are used for each determination.

7.2 Test portion. Put two lidded porcelain crucibles together into the muffle furnace (5.6) at 550 °C +/- 10 °C and heat them for 2 h. Take the porcelain crucibles out, put them into a desiccator and cool them to room temperature, then weigh each porcelain crucible to the nearest 1 mg (m1). Weigh separately two test portions of 1 g +/- 0.05 g (m0) and pour one into each porcelain crucible, to the nearest 1 mg.

7.3 Determination. Place the stainless steel support (5.3) inside the nickel crucible and spread a layer of calcined petroleum coke (Clause 4) 10 mm +/- 1 mm thick on the bottom of the crucible. Set the porcelain crucible containing the test portion on the stainless steel support, with the bottom of the porcelain crucible touching the calcined petroleum coke. Put the lid on the porcelain crucible, fill the gap between the two crucibles with calcined petroleum coke so that the porcelain crucible is completely embedded in it, and put the lid on the nickel crucible. Repeat the above operations with the other porcelain crucible and test portion. Set the prepared crucibles on the nickel crucible rack and move them quickly into the muffle furnace (5.6) at 550 °C +/- 10 °C, so as to avoid a large loss of heat from the furnace. The temperature of the space in the muffle furnace where the nickel crucible rack is placed shall be uniform and shall meet the condition laid down, that is it shall be held at 550 °C +/- 10 °C. The initial temperature of the muffle furnace shall be calibrated with a

potentiometer, and only the part of the muffle furnace that meets this temperature requirement may be used for placing the nickel crucibles and their support. There shall be a space of not less than 7 mm between the nickel crucibles and the bottom, the walls and the top of the furnace, and the space between any nickel crucible and the front wall of the muffle furnace (including the furnace door) or the rear wall of the furnace shall not be less than 50 mm. After 2.5 h, move the nickel crucibles out of the muffle furnace and cool them. Take the porcelain crucibles out of the nickel crucibles and carefully remove the calcined petroleum coke powder adhering to them, so as to avoid contaminating the sample. Transfer the porcelain crucibles with the test portion inside them into a desiccator, cool them to room temperature and weigh each crucible to the nearest 1 mg (m2). A note states that the porcelain crucibles, once cleaned, can be used again, and that they are to be ignited by heating to 700 °C to 1 000 °C in order to remove the calcined petroleum coke and any carbonaceous residue. Repeat the above operations for the second determination.

8 Expression of the result of the determination

8.1 Calculation of the result. The coking value is expressed as a mass fraction and is calculated by formula (1): the mass of the porcelain crucible with the residue, less the mass of the empty porcelain crucible, divided by the mass of the test portion, multiplied by 100 %. In the formula, m0 is the mass of the test portion, in grams (g); m1 is the mass of the empty porcelain crucible, in grams (g); m2 is the mass of the porcelain crucible with the residue, in grams (g). Each of the results obtained with the four porcelain crucibles is calculated. Results in doubt are discarded (see 8.2.1 and 8.2.2), and the determination shall be repeated according to 7.3 in order to obtain four valid results. The four valid results are averaged and reported as a mass fraction, to 0.1 %.

8.2 Precision.

8.2.1 Reproducibility of the results of the two porcelain crucibles within one determination. If the difference between the results of the two porcelain crucibles placed into the electric furnace at the same time within one determination is greater than the square root of 2 times the reproducibility of this method, the two results shall be regarded as in doubt.

8.2.2 Repeatability limit (r). When the difference between the results obtained by the same operator testing the same test portion with the same instrument, each result being the mean of the results of the two porcelain crucibles of one determination, exceeds 1.0 % (mass fraction), the results are regarded as in doubt, that is r = 1.0 % (mass fraction).

8.2.3 Reproducibility limit (R). When the difference between the results obtained in different laboratories testing the same test portion, each result being the mean of the results of the two porcelain crucibles of one determination, exceeds 2.0 % (mass fraction), the results are regarded as in doubt, that is R = 2.0 % (mass fraction).