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GB/T 26930.12-2014

Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Part 12: Determination of volatile matter content

原铝生产用炭素材料 煤沥青 第12部分：挥发物含量的测定

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1 Scope

This part of GB/T 26930 lays down an empirical method for determining, by gas chromatography, the volatile substances of the toluene-soluble matter in coal tar pitch.

2 Normative references

The application of the following documents to this part is indispensable. For a dated reference only the edition bearing that date applies; for an undated reference the latest edition, including all amendments, applies.

ISO 565, Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings.

ISO 1042, Laboratory glass ware - One-mark volumetric flasks.

ISO 6257, Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Sampling.

3 Terms, definitions and symbols

The following terms and definitions apply to this part.

3.1.1 volatile matter content: under the specified determination conditions, the mass fraction of all the substances measured by gas chromatography within the retention time running from the boiling point at atmospheric pressure up to 360 °C.

3.2.1 Symbols. The legend printed in the document lists the area of a chromatographic peak, the slope of the regression line, the equivalent boiling point at atmospheric pressure, the intercept of the regression line, the conversion factor by which the percentage content of the original pitch sample is calculated from the total peak area, the sulfur-to-phenanthrene area ratio, the retention time ratio, the mass of the calibration mixture, the reproducibility, the quantitation factor of the calibration mixture defined in 5.2, the repeatability, the retention time of a chromatographic peak, and the mass fraction of sulfur in the test sample. Two of the letters printed in the legend could not be told apart with certainty on the scan and the individual letter assignments are therefore not reproduced here.

3.2.2 Superscripts and subscripts. The subscript for sulfur; the subscript for the i-th peak; the subscript for phenanthrene; the subscript for pitch; the subscript for retention time; the subscript for the peak area of the calibration mixture in the chromatogram; the subscript that marks the original pitch sample; and the prime that marks a result obtained after the sulfur internal standard has been added. The letters A, B and C of the legend correspond to the chromatograms A, B and C of the solutions of the same names.

4 Principle

Under defined conditions the mixture is separated by a gas chromatograph on a column having a non-polar stationary phase. The analytical result shows the retention time of each component of the mixture and its relative characteristic coefficient, that coefficient being the ratio between the average equivalent boiling point of the internal standard and the boiling point of the component. The analysis also gives the ratio between the total peak area of the volatile substances and the mass of the pure substance of the calibration mixture; because coal tar pitch consists mainly of coal tar oils, which have a good linear relationship with the boiling points of the substances chosen as internal standard, a solution containing an internal standard is necessary for the chromatographic determination.

The peak area of each substance is compared with the peak area of the internal standard in the two solutions in order to calculate the mass. These data, together with the graph

obtained beforehand, allow the relative characteristic coefficient of the quantity of substance the peak area represents to be established; the linear relationship between the retention time of each component of the calibration mixture and the boiling point at atmospheric pressure is set up, and the distillation curve or the table of values for the sample is derived. From the boiling point at atmospheric pressure up to 360 °C, the percentage content of each component of the calibration mixture and of the test sample chromatogram is calculated, and the result can be corrected.

5 Reagents

A warning states that the safety and health data supplied by the reagent supplier are to be consulted for the safe use of the reagents listed in 5.1 to 5.3.

5.1 Sulfur: purity at least 98 percent.

5.2 Calibration mixture: made up of naphthalene, 2-methylnaphthalene, acenaphthene, dibenzofuran, fluorene, phenanthrene, fluoranthene and pyrene. The purity of all the reagents is required to be at least 98 percent, and under the chromatographic analysis conditions none of them may contain any impurity that co-elutes with sulfur. A note adds that the boiling points of the components of the calibration mixture are given in Annex A.

5.3 Toluene: analytical grade.

6 Apparatus

6.1 Gas chromatograph: able to operate at 300 °C, with a heated injector, a flame ionisation detector with amplifier, and a capillary column oven whose heating rate can be set between 1 °C/min and 10 °C/min over the range 50 °C to 300 °C. A split injector may be used, but if other equipment is used it is to be stated in the test report. Data workstation: an instrument that can process the retention times and peak areas delivered by the gas chromatograph; the data workstation may also be a separate instrument matched to the chromatograph.

6.3 Capillary column: has a non-polar stationary phase that does not bleed under gas chromatographic analysis conditions. When the sample is injected into the column no obvious deviation of the baseline is to appear over the retention time. A typical capillary column is 12 m to 25 m long, made of fused silica of minimum internal diameter 0.53 mm, with a stationary phase such as polydimethylsiloxane; the volume injected is chosen according to the internal diameter of the column. Other stationary phases are required to have chemically stable low-bleed characteristics and a good linear relationship with the volume, so that the peaks of the calibration mixture appear as similar as possible to those of the system used.

6.4 Chromatographic injector: a typical injection volume lies between 1 microlitre and 25 microlitres.

6.5 Volumetric flasks: conforming to ISO 1042, of 10 mL to 100 mL capacity. 6.6 Ultrasonic bath: typically 100 W to 200 W in power, operating at 40 kHz. 6.7 Mortar: made of a hard, wear-resistant material such as agate or tungsten carbide. 6.8 Test sieve: nominal aperture 212 μm, conforming to ISO 565, with lid and receiving pan.

7 Sampling and sample preparation

Following ISO 6257, take 5 g to 10 g of representative pitch sample and grind it in the mortar until the whole of the sample passes the test sieve of 6.8.

8 Preparation of the test solutions

8.1 Test solution A, without internal standard. Weigh about 1 g of the ground sample from Clause 7 into a 10 mL volumetric flask as in 6.5, recording the mass to 0.1 mg. Add toluene to the flask but do not fill to the mark; then open the stopper. If the ultrasonic bath of 6.6 has a heating function, keep the temperature between 60 °C and 70 °C and treat for 20 min. If the ultrasonic bath has no heating function, treat ultrasonically for 5 min, then heat the flask for 5 min in a water bath at 60 °C to 70 °C, repeating the ultrasonic and heating cycle at least three times so that the total ultrasonic time reaches 20 min. Take the flask out, cool it to room temperature, fill to the mark with toluene, stopper it and shake it vigorously for 20 s, then let it stand for at least 1 h, preferably overnight. Keep the flask away from direct sunlight or strong light before the solution is used, and complete the determination within 48 h. A note adds that letting the solution stand overnight before use is preferable, because undissolved matter carried into the chromatograph injector can settle out and block the capillary, affecting the performance of the equipment; centrifuging or filtering through a membrane that neither absorbs nor dissolves the solution may be used to shorten the standing time.

8.2 Test solution B, with internal standard. Weigh about 0.01 g of sulfur into a 10 mL volumetric flask, recording the mass to 0.1 mg, and then follow the procedure described in 8.1, recording the mass of the sulfur and of the pitch sample taken.

9 Preparation of the calibration solution - solution C

Weigh 0.1 g +/- 0.001 g of the sulfur of 5.1 and about 0.001 g of each component of the calibration mixture of 5.2 directly into a 100 mL volumetric flask of 6.5, recording the masses to 0.1 mg. Add enough toluene to dissolve all the solid particles, treating the flask in the ultrasonic bath, then fill to the mark with toluene and shake the flask to mix the solution evenly. Keep the solution out of direct sunlight or strong light and store it between 15 °C and 30 °C; it is not to be used for more than one month, after which it is to be prepared again.