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Proximate analysis of coal - Instrumental method

煤的工业分析方法 仪器法

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Proximate analysis of coal - Instrumental method

1 Scope

This standard specifies the method summary, reagents and materials, instruments and equipment, measurement procedures, calculation of results, precision for determining the moisture, ash content and volatiles of coal by automatic industrial analyzers, as well as calculation of fixed carbon.

This standard applies to lignite, bituminous coal, and anthracite.

2 Normative references

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

GB/T 212 Proximate analysis of coal

GB/T 483 General rules for analytical and testing methods of coal

3 Method summary

3.1 Moisture determination

A certain amount of general analysis test coal sample is taken and dried in a heating furnace in air or nitrogen flow at 105 °C to 110 °C until the mass is constant; the moisture mass fraction of the coal sample is calculated based on the mass loss of the coal sample.

3.2 Determination of ash content

A certain amount of general analysis test coal sample is taken and heated to (815 ± 10) °C in a heating furnace according to the specified procedure; during this process, it is ashed and burned in air or oxygen flow until the mass is constant. The ash content mass fraction of the coal sample is calculated according to the mass of the residue.

3.3 Determination of volatile matter

A certain amount of general analysis test coal sample is taken and heated in a heating furnace for 7 minutes at (900 ± 10) °C without air; the moisture mass fraction of the coal

sample is subtracted from the mass fraction of the decrease mass in the mass of the coal sample, the remaining is taken as the volatile mass fraction of the coal sample.

4 Reagents and materials

4.1 Nitrogen: with a purity of 99.9%.

4.2 Oxygen: 99.9%, no hydrogen.

4.3 Anhydrous calcium chloride: chemically pure, granular.

4.4 Color-changing silica gel: industrial supplies.

4.5 Crucible: It is used for measuring moisture and ash content, made of porcelain or quartz, with sufficient bottom area to ensure that the mass of the coal sample per square centimeter does not exceed 0.15 g after the coal sample is spread out.

4.6 Volatile matter crucible: It is made of porcelain or quartz, and equipped with a tight cover, the shape and size comply with the requirements of GB/T 212.

4.7 Certified coal reference materials: national first-class coal physical property and chemical composition analysis reference materials.

5 Instruments and Equipment

5.1 Automatic industrial analyzer

5.1.1 General

The automatic industrial analyzer shall include a high-temperature furnace, a built-in balance, a sample receiving and conveying device, a temperature measurement and control and display system, a furnace atmosphere control system, a result display and printing device, etc. Appendix A gives the structural schematic diagrams of several automatic industrial analyzers. The automatic industrial analyzer shall record and provide detailed information such as empty crucible mass, coal sample mass, hot crucible mass, and buoyancy effect correction value in each test by printing or other means.

5.1.2 High-temperature furnace

5.1.2.1 Moisture determination furnace: It can control the temperature between 105 °C and 110 °C, has a sufficient constant temperature zone, a small free space in the furnace, and ventilation and exhaust ports. The constant temperature zone is measured at least once a year.

6 Measurement procedures

6.1 General

6.1.1 Moisture and ash content can be measured continuously with the same sample in the same heating furnace, or two samples can be measured separately; volatile matter shall be weighed and measured separately.

6.1.2 Each measurement shall use one or more empty crucibles at the same time to correct the buoyancy effect of the thermal mass of the crucible.

NOTE: See Appendix B for buoyancy effect correction calculation.

6.1.3 All weighings during the test shall be accurate to 0.0002 g.

6.1.4 The instrument shall be operated according to the instrument instruction manual.

6.1.5 The instrument shall be inspected from time to time by using certified coal reference materials based on actual usage conditions.

6.2 Moisture determination

6.2.1 In the measuring instrument, accurately weigh the crucible that has been pre-dried to a constant mass, add (1 ± 0.1) g general analysis test coal sample to the crucible, flatten it, and weigh it accurately.

6.2.2 According to the provisions of 5.1.4, air or nitrogen must be introduced into the heating furnace in advance (nitrogen must be introduced for lignite samples). The furnace temperature slowly rises to 105 °C~110 °C and remains constant. The instrument automatically weighs according to the set time interval until the mass is constant (the mass decrease does not exceed 0.0005 g at a 10-minute interval) or the mass increases. In the latter case, the mass before the mass increase is used as the basis for calculation.

6.3 Ash content determination

6.3.1 Individual determination by a slow ashing method

6.3.1.1 Accurately weigh the crucible that has been pre-dried to a constant mass in an environment close to room temperature, add (1 ± 0.1) g of general analysis test coal sample to the crucible, flatten it, and weigh it accurately.

6.3.1.2 According to the provisions of 5.1.4, introduce air or oxygen into the furnace, slowly increase the furnace temperature to 500 °C in no less than 30 minutes, and maintain it at this temperature for 30 minutes. Continue to raise the temperature to (815 ± 10) °C, and burn the sample until the mass is constant at this temperature. The

calculation is based on the mass of the residue after the last ignition. During the burning process, the instrument automatically weighs according to the set time interval until the mass change does not exceed 0.0005 g at 10-minute intervals.

6.3.2 Continuous determination by a slow ashing method

After the moisture measurement is completed (see 6.2), measure the ash content according to steps 6.3.1.2.

6.3.3 Rapid determination

Rapidly heat the furnace to $(815 \pm 10)^\circ\text{C}$ at a speed that will prevent deflagration of the coal sample (heating time shall not be less than 30 minutes). Otherwise, the rest is the same as 6.3.1.1 and 6.3.1.2. Airflow shall be increased if necessary.

6.4 Determination of volatile matter

6.4.1 In the weighing chamber of the measuring instrument, accurately weigh the crucible that has been pre-dried to a constant mass, add (1 ± 0.01) g general analysis test coal sample into the crucible, flatten it, and weigh it accurately. Lignite and long-flame coal samples shall be pressed into cakes first, and then cut into small pieces with a particle size of about 3 mm.

6.4.2 Preheat the high-temperature furnace to $900^\circ\text{C} \sim 920^\circ\text{C}$ (if necessary, nitrogen can be introduced to maintain an inert atmosphere in the furnace according to 5.1.4). The sample receiving and conveying device will send the covered crucible into the constant temperature zone of the high-temperature furnace. Heat it accurately for 7 minutes before returning it to the weighing room, and weigh it again after cooling to the set temperature.

6.4.3 After the volatile matter crucible is placed in the high-temperature furnace, the furnace temperature shall return to $(900 \pm 10)^\circ\text{C}$ within 3 minutes, and then remain at $(900 \pm 10)^\circ\text{C}$; otherwise, the test will be invalid. Heating time includes temperature recovery time.

7 Result calculation

7.1 Moisture calculation

Calculate the moisture content of the general analysis test coal sample according to formula (1).

$$M_{\text{ad}} = \frac{m_0 + m_1 - m_2}{m_1} \times 100 \quad \dots\dots\dots (1)$$

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