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Proximate analysis of coal

煤的工业分析方法

(ISO 11722:1999 Solid mineral fuels - Hard coal - Determination of moisture in the general analysis test sample by drying in nitrogen, ISO 1171:1997 Solid mineral fuels - Determination of ash, ISO 562:1998 Hard coal and coke - Determination of volatile matter, NEQ)

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Proximate analysis of coal

1 Scope

This standard specifies the methods for determining the moisture, ash, volatile content of coal and coal water mixture as well as the method for calculating the fixed carbon.

This standard applies to lignite, bituminous coal, anthracite, coal water mixture.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 218 Determination of carbonate carbon dioxide content in coal (GB/T 218-1996, eqv ISO 925:1980)

GB/T 7560 Determination of mineral matter in coal (GB/T 7560-2001, eqv ISO 602:1983)

GB/T 18510 Guideline for the validation of alternative methods of analysis for coal and coke

GB/T 18856.1 Test methods for quality of coal water mixture - Part 1: Sampling for coal water mixture

3 Determination of moisture

This chapter specifies three methods for determining the moisture content of coal. Among them, method A is applicable to all coal types; method B is only applicable to bituminous coal and anthracite; microwave drying method (see Appendix A) is suitable for rapid determination of moisture content of lignite and bituminous coal.

In the case of arbitration analysis, it is necessary to use the method A to determine the moisture content of the coal sample for general analysis test when using the moisture of coal sample for general analysis test for calibration

3.1.4 Test procedure

3.1.4.1 In a pre-dried and weighed weighing bottle, weigh (1 ± 0.1) g of coal sample for general analysis test which has a particle size of less than 0.2 mm, accurate to 0.0002 g; spread it flatly in the weighing bottle.

3.1.4.2 Open the lid of the weighing bottle and put it into a drying box (3.1.3.1) that has been pre-filled with dry nitrogen and heated to $(105 \sim 110)$ °C. Anthracite is dried for 1.5 hours, whilst lignite and anthracite are dried for 2 hours. At 10 min before putting the weighing bottle in the drying box, start leading in nitrogen, at a flow rate of 15 times per hour.

3.1.4.3 Take out the weighing bottle from the drying box; immediately cover it; put it in a desiccator and cool it to room temperature (about 20 min); weigh it.

3.1.4.4 Perform inspecting drying for 30 min each time, until the mass of the continuously dried coal sample is not reduced by more than 0.0010 g or the mass is increased. In the latter case, the previous mass is used as the basis for calculation. When the moisture is less than 2.00%, no inspection drying is necessary.

3.2 Method B (air drying method)

3.2.1 Method summary

Weigh a certain amount of coal sample for general analysis test; place them in blast drying oven at $(105 \sim 110)$ °C; dry them in an air stream to a constant mass. The mass fraction of water is calculated from the mass loss of the coal sample.

3.2.2 Instrumentation

3.2.2.1 Blast drying oven: It is equipped with an automatic temperature control device, which can keep the temperature in the range of $(105 \sim 110)$ °C.

3.2.2.2 Glass weighing bottle: Same as 3.1.3.2.

3.2.2.3 Dryer: Same as 3.1.3.3.

3.2.2.4 Analytical balance: Same as 3.1.3.6.

3.2.3 Test procedure

3.2.3.1 In a pre-dried and weighed weighing bottle, weigh (1 ± 0.1) g of the coal sample for general analysis test which has a particle size of less than 0.2 mm, accurate to 0.0002 g; flatly spread it in the weighing bottle.

3.2.3.2 Open the lid of the weighing bottle and put it in a drying box (3.2.2.1)

0.2 mm, accurate to 0.002 g; evenly spread it in an ash dish, so that the mass per square centimeter does not exceed 0.08 g.

4.2.1.3.4 Put the ash dish containing coal samples on the conveyor of the fast ash tester, then the ash dish is automatically sent into the furnace.

4.2.1.3.5 When the ash dish is sent out of the furnace, remove it; place it on a heat-resistant porcelain plate or asbestos plate; cool it in air for about 5 min. Transfer it in a desiccator to cool it to room temperature (about 20 min). Weigh it.

4.2.2 Method B

4.2.2.1 Method summary

The ash dish containing the coal sample is gradually sent from the outside of the furnace into a muffle furnace which was preheated to $(815 \pm 10) ^\circ\text{C}$ and ashed and burned to a constant mass. The mass fraction of the residue to the mass of the coal sample is taken as the ash content of the coal sample.

4.2.2.2 Instruments and equipment: Same as 4.1.2.

4.2.2.3 Test procedure

4.2.2.3.1 In an ash dish pre-burned to a constant mass, weigh (1 ± 0.1) g of coal sample for general analysis test with a particle size of less than 0.2 mm, accurate to 0.0002 g. Uniformly and flatly spread it in an ash dish so that the mass per square centimeter does not exceed 0.15 g. Dispense the ash dishes containing coal samples on heat-resistant porcelain plates or asbestos plates in advance.

4.2.2.3.2 Heat the muffle furnace to $850 ^\circ\text{C}$; open the furnace door; slowly push the heat-resistant porcelain plate or asbestos plate with the ash dish into the muffle furnace; first place the coal sample in the first row of gray dish to ash. When the coal sample no longer emits smoke after $(5 \sim 10)$ minutes, push the remaining rows of ash dishes sequentially into the hot part of the furnace at a speed of not more than 2 cm per minute (if the coal sample catches fire, the test shall be abolished).

4.2.2.3.3 Close the furnace door and leave a gap of about 15 mm in the furnace door; burn it at $(815 \pm 15) ^\circ\text{C}$ for 40 minutes.

4.2.2.3.4 Take out the ash dish from the furnace; cool it in the air for about 5 minutes; move it into a desiccator to cool to room temperature (about 20 minutes); weigh it.

4.2.2.3.5 Perform inspective burning at $(815 \pm 10) ^\circ\text{C}$ for 20 min each time, until the mass of the continuously dried coal sample is not changed by more than

required that the furnace temperature be restored to $(900 \pm 10) ^\circ\text{C}$ within 3 min, then maintained at $(900 \pm 10) ^\circ\text{C}$, otherwise the test is invalidated. The heating time includes the temperature recovery time.

Note: The pre-heating temperature of the muffle furnace can be adjusted according to the specific conditions of the muffle furnace, to ensure that the furnace temperature is restored to $(900 \pm 10) ^\circ\text{C}$ within 3 minutes after placing in the crucible and crucible holder.

5.3.3 Take out the crucible from the furnace; cool it in the air for about 5 minutes; move it into a desiccator and cool to room temperature (about 20 minutes); weigh it.

5.4 Classification of coke slag characteristics

The characteristics of coke slag obtained from the determination of volatiles are distinguished according to the following requirements:

- a) Powder (type 1): All powder, no particles sticking to each other;
- b) Adhesion (type 2): It becomes powder or basically power by gentle touching it with fingers, wherein the larger clumps become powder by gentle touching;
- c) Weak adhesion (type 3): It becomes small block once gently pressed by fingers;
- d) Non-melt bonding (type 4): It is cracked into small pieces by forced pressing with fingers; the upper surface of the coke slag is dull, whilst the lower surface is slightly silvery white;
- e) Non-swelling fusion bonding (type 5): Coke slag forms flat blocks; the boundaries of coal particles are not easy to distinguish; the upper surface of the coke slag has a clear silver-white metallic luster, whilst the lower surface has a more obvious silver-white luster;
- f) Micro-swelling fusion bonding (type 6): It is not broken by finger pressing; the upper and lower surfaces of the coke slag have a silver-white metallic luster, but the surface of the coke slag has relatively small swelling bubbles (or small blisters);
- g) Swelling, melting and sticking (type 7): The upper and lower surfaces of the coke slag have a silver-white metallic luster, which is obviously swelled, but the height does not exceed 15 mm;
- h) Strong-swelling, melting, bonding (type 8): The upper and lower surfaces of the coke slag have a silver-white metallic luster; the coke slag's height is greater than 15 mm.

Where:

V_{dmmf} - Mass fraction of dry mineral-free base volatile matter, %;

MM_{ad} - Mass fraction of air-dried base coal sample minerals (determined in accordance with GB/T 7560), %.

8 Industrial analysis of coal water mixture

8.1 Preparation of analysis samples

8.1.1 Preparation of coal water mixture specimen

Before the test, stir the coal water mixture specimen to make it free from hard and soft sediments and in a uniform state.

8.1.2 Preparation of dry specimen of coal water mixture

Prepare a dry specimen of coal water mixture in accordance with the method specified in GB/T 18856.1.

8.2 Determination of moisture in coal water mixture

8.2.1 Method summary

Weigh a certain amount of evenly stirred coal water mixture specimen; put it in a drying box at (105 ~ 110) °C; dry it in air flow to constant mass. Then calculate the mass fraction of moisture in the coal water mixture based on the mass loss of the coal water mixture.

8.2.2 Instrumentation

Same as 3.2.2.

8.2.3 Test procedure

8.2.3.1 Weigh (1.2 ~ 1.5) g of evenly stirred coal water mixture (accurate to 0.0004 g) in a pre-dried weighing bottle of known mass; quickly add the lid and weigh it. After weighing, spread flatly the coal water mixture on the bottom of the weighing bottle.

8.2.3.2 Open the lid of the weighing bottle; put the weighing bottle with coal water mixture into a drying box that has been blasted and heated to (105 ~ 110) °C; dry it under blast conditions for 1 h.

8.2.3.3 Take out the weighing bottle from the drying box; immediately put the lid on the dryer; cool it to room temperature (about 20 min) and weigh it.

Appendix A

(Normative)

Determination of moisture in coal - Microwave drying method

A.1 Scope

This appendix specifies a method for the fast determination of moisture in the coal sample for general analysis test by microwave drying.

This method is suitable for the fast determination of moisture in lignite and bituminous coal.

A.2 Method summary

Weigh a certain amount of coal sample for general analysis test and place them in a microwave moisture tester. The magnetron in the furnace emits non-ionizing microwaves, which causes water molecules to vibrate at very high speeds, generate frictional heat, quickly evaporate moisture in the coal. Calculate moisture based on the mass loss of coal sample.

A.3 Instrumentation

A.3.1 Microwave moisture tester (hereinafter referred to as moisture meter): It is equipped with a program controller; the input power is about 1000 W. The instrument is equipped with a glass-ceramic glass turntable with an asbestos pad with a marking ring and a thickness of about 2 mm.

A.3.2 Glass weighing bottle: Same as 3.1.3.2.

A.3.3 Dryer: Same as 3.1.3.3.

A.3.4 Analytical balance: Same as 3.1.3.6.

A.3.5 Beaker: Capacity is about 250 mL.

A.4 Test procedure

A.4.1 In a pre-dried and weighed weighing bottle, weigh (1 ± 0.1) g of coal sample for general analysis test which has a particle size of less than 0.2 mm, accurate to 0.0002 g; spread it evenly in the weighing bottle.

A.4.2 Place a beaker containing approximately 80 mL of distilled water which has a capacity of approximately 250 mL on a turntable in the water meter; after heating it for 10 minutes with a preheating program, remove the beaker. If several measurements are to be carried out continuously, it is only necessary to preheat before the first measurement.

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