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Gray-King Assay of Coal

煤的格金低温干馏试验方法

(ISO 502:1982, Coal - Determination of Caking Power -

Gray-King Coke Test, NEQ)

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Gray-King Assay of Coal

1 Scope

This Standard specifies the method and principle, instruments and equipment, test procedures, result expression and method precision of the Gray-King low-temperature dry distillation assay of coal.

This Standard is applicable to lignite and bituminous coal.

2 Normative References

Through the reference in this Standard, the clauses of the following documents become clauses of this Standard. In terms of references with a specified date, all the subsequent modification sheets (excluding the corrected content) or revised versions are not applicable to this Standard. However, the various parties that reach an agreement in accordance with this Standard are encouraged to explore the possibility of adopting the latest version of these documents. In terms of references without a specified date, the latest version is applicable to this Standard.

GB/T 212 *Proximate Analysis of Coal* (GB/T 212-2001, eqv ISO 11722:1999, eqv ISO 1171:1997, eqv ISO 562:1998)

GB 474 *Method for Preparation of Coal Sample*

GB/T 5448 *Determination of the Crucible Swelling Number of Bituminous Coal - Electrical Heating* (GB/T 5448-1997, eqv ISO 501:2003)

GB/T 19494.2 *Mechanical Sampling of Coal - Part 2: Method for Sample Preparation* (GB/T 19494.2-2004, ISO 13909-4:2001, NEQ)

3 Method Summary

Place coal sample in a dry distillation tube, then, place it in a Gray-King low-temperature dry distillation furnace. In accordance with the specified temperature raising program, heat it to the final temperature of 600 °C; preserve the heat for a certain period of time. Then, determine the yield of tar, pyrolysis water and semi-coke obtained. Meanwhile, compare the semi-coke with a set of standard coke types, so as to determine the model. In terms of strongly swelling coal, a certain amount of electrode charcoal needs to be mixed into the coal sample; its coke type is expressed in the minimum amount of electrode charcoal (integer grams) required to obtain the coke type consistent with the standard coke type (G).

4 Reagents and Materials

4.1 High-temperature graphitized electrode charcoal:

If moisture is less than 0.5%, determine in accordance with GB/T 212.

If ash content is less than 2%, determine in accordance with GB/T 212.

If volatile component is less than 1.5%, determine in accordance with GB/T 212.

If particle size is less than 0.2 mm, the mass fraction of particle size that is less than 0.1 mm shall be 60% ~ 90%.

When using it, in accordance with the procedures specified in Chapter 6 and Chapter 7, determine the moisture and semi-coke yield. The same batch of electrode charcoal shall be re-determined at least once every six months.

4.2 Xylene (HG/T 3-1011) or toluene (GB/T 684): chemically pure.

4.3 Acetone (GB/T 6026): industrial product.

4.4 Asbestos wool and asbestos board: asbestos wool needs to be pre-burned at 800 °C for 1 h. After it cools down, place it into a glass bottle for later usage. The thickness of asbestos board is around 2 mm.

5 Instruments and Equipment

5.1 Gray-King dry distillation furnace (Figure 1): double-hole or multiple-hole; constant-temperature area is not less than 200 mm; automatic program temperature control.

5.2 Dry distillation tube (Figure 2): made of heat-resistant glass or quartz glass.

5.3 Conical flask: with a capacity of 250 mL; equipped with a moisture determination tube; with a grinding mouth.

5.4 Moisture determination tube (Figure 3): scale range is (0 ~ 5) mL of (0 ~ 10) mL; division value is 0.05 mL; with a grinding mouth.

5.5 Condenser: straight-tube type; with a grinding mouth; length of condensate sleeve is not less than 300 mm.

5.6 Balance: division value is 0.01 g.

5.7 Push rod (Figure 4): made of metal.

5.8 Electric furnace: single-type; double or multiple; temperature adjustable.

the dry distillation tube is stained with coal, a soft brush may be used to brush it below the scale of the dry distillation tube.

6.3 Place the dry distillation tube branch, which is loaded with the coal sample, downwards and horizontally. Place the notch of a notched asbestos round gasket (the diameter is equivalent to the inner diameter of the dry distillation tube; cut a small piece from $1/6 \sim 1/4$ of the diameter) upwards, put it in from the nozzle; use the push rod (5.7) to slowly push it to the scale; pay attention not to leave the coal sample outside the asbestos gasket. Then, put in a ball of asbestos; use the push rod to push it close to the asbestos gasket; gently press it to (5 ~ 10) mm thick, but do not squeeze it too tightly.

6.4 Use both hands to hold both ends of the dry distillation tube, which is loaded with asbestos gasket and asbestos wool. Place the branch vertically downward. Along the horizontal direction, gently shake it to evenly spread the coal sample. Then, tap on the wooden table for several times to make the surface of the coal sample smooth. Pay attention not to allow the coal sample to get on top of the dry distillation tube. If the asbestos fleece layer is damaged after being tapped on, the push rod shall be used to re-press it into a layer.

6.5 Use a heat-resistant rubber plug to stuff up the mouth of the dry distillation tube. On the dry distillation tube branch, install a heat-resistant rubber plug with a glass airway tube (the airway tube is protruded from the rubber plug by around 5 mm); weigh it (accurate to 0.01 g).

6.6 Place an appropriate amount of water (temperature is below 15 °C) into the water tank of the Gray-King dry distillation furnace. Move the water tank upward, till $2/3$ of the height of the conical flask is immersed in the cooling water; maintain a constant water level.

7 Test Procedures

7.1 Through electrical heating, heat the Gray-King dry distillation furnace to 300 °C; maintain this temperature. Insert the dry distillation tube (6.5) into the furnace; put the dry distillation tube branch close to the furnace port. Starting from 300 °C, at 5 °C/min, raise the temperature, so as to continue heating the dry distillation furnace to 600 °C. In addition, maintain for 15 min at this temperature (during the whole process of heating, the difference between the actually measured temperature and the expected temperature shall not exceed 10 °C), then, stop heating.

During the test, tar, water vapor and coal gas generated by the decomposition of the coal sample enter the conical flask through the dry distillation tube branch. The tar and water vapor are condensed in the conical flask, while the coal gas is discharged from the airway tube (ignited and burned, or, discharged outside).

7.2 Move the water tank downward to expose the conical flask out of the water surface.

7.5 Determination of Total Moisture in Dry Distillation

7.5.1 Add 50 mL of xylene or toluene (4.2) to the conical flask containing condensate. Then, connect the moisture determination tube; connect it to the condenser. Use cotton or other objects to plug the upper end of the condenser.

NOTE: the measuring tube of the moisture determination tube shall be pre-calibrated.

7.5.2 Place the assembled conical flask on the sand bath; inlet cooling water into the condenser. Through electrical heating, heat the sand bath and control the distillation rate, so that the number of drops dripping from the end of the condenser is about (2 ~ 4) drops/s. Distillation shall be conducted in a fume hood.

7.5.3 When the amount of water in the moisture determination tube does not increase within 10 min, stop the distillation. The distillation generally takes around (1 ~ 1.5) h.

7.5.4 Wait till the conical flask cools down, turn off the cooling water; remove the conical flask and the moisture determination tube. If some of the water beads are attached to the wall of the moisture determination tube, or, if some of the solvent sinks to the lower part of the water layer, a thin metal (or glass) rod may be used to stir and remove it. Let it stand for layering, then, read the volume (mL) of water from the moisture determination tube (accurate to 0.01 mL).

Take the density of water at room temperature as 1 g/cm³, calculate the mass of the total moisture of dry distillation (m_b).

7.6 After all tests are completed, use a brush dipped in decontamination powder (if necessary, use acetone) to scrub the dry distillation tube, the conical flask and the moisture determination tube, then, dry them in a drying oven for later usage.

7.7 Mass Calculation of Various Products

7.7.1 Total mass of semi-coke (m_c): the mass of the dry distillation tube after degreasing (including the mass of asbestos wool, asbestos gasket, semi-coke and rubber stopper) minus the mass of the dry distillation tube before the test (including the mass of asbestos wool, asbestos gasket and rubber stopper). Under the circumstances with electrode charcoal, the mass of electrode charcoal semi-coke shall also be subtracted.

7.7.2 Total mass of tar: the mass of condensate (m_a) minus the mass of water (m_b), then, add the mass of tar attached inside the dry distillation tube and the branch (m_d).

7.7.3 Coal gas and dry distillation loss: the mass of coal sample (m) minus the sum of the total mass of tar, the total moisture of dry distillation and the mass of semi-coke.

Under the circumstances with electrode charcoal: the sum of the mass of coal sample (m) and the mass of electrode charcoal (m') minus the sum of the total mass of tar, the

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