GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.040

D 21

GB/T 476-2008

Replacing GB/T 476-2001, GB/T 15460-2003, GB/T 18856.11-2002

Determination of carbon and hydrogen in coal

煤中碳和氢的测定方法

(ISO 625:1996, Solid mineral fuels - Determination of carbon and hydrogen - Liebig method, MOD)

Issued on: July 29, 2008 **Implemented on: May 01, 2009**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of PRC;

Standardization Administration of PRC.

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Determination of carbon and hydrogen in coal

1 Scope

This standard specifies the method principles, reagents and materials, devices, test procedures, result calculation, precision, etc., of the three-stage furnace method, two-stage furnace method, electricity consumption method, for the determination of the hydrogen in the coal and coal-water slurry dried coal sample, AND the weight method for the determination of carbon, in the carbon and hydrogen analysis of coal and coal-water slurry.

This standard applies to lignite, bituminous coal, anthracite, coal-water slurry.

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 212 Proximate analysis of coal (GB/T 212-2008, ISO 11722:1999, ISO 1171:1997, ISO 562:1998, NEQ)

GB/T 218 Determination of carbon dioxide content in the mineral carbonates associated with coal (GB/T 218-1996, eqv ISO 925:1980)

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

3 Three-stage furnace method and two-stage furnace method

3.1 Method principle

A certain amount of coal sample OR coal-water slurry dried coal sample is burned in an oxygen flow; the generated water and carbon dioxide are absorbed by a water absorbent and a carbon dioxide absorbent, respectively; the mass fraction of carbon and hydrogen, in the coal, is calculated from the increase of the absorbent. The interference of sulfur and chlorine, in coal

samples, on carbon determination, is eliminated by lead chromate and silver wire coils, in the three-stage furnace; it is eliminated by the pyrolysis product of silver permanganate, in the two-stage furnace. The interference of nitrogen on carbon determination, is eliminated by granular manganese dioxide.

3.2 Reagents and materials

3.2.1 Anhydrous magnesium perchlorate: Analytically pure, which has a particle size of 1 mm ~ 3 mm. OR anhydrous calcium chloride: Analytically pure, which has a particle size of 2 mm ~ 5 mm.

3.2.2 Granular manganese dioxide: Chemically pure, which is commercially available or prepared from manganese sulfate and potassium permanganate.

Preparation method: Weigh 25 g of manganese sulfate. Dissolve it in 500 mL of distilled water. Weigh another 16.4 g of potassium permanganate. Dissolve in 300 mL of distilled water. The two solutions are heated to 50 °C ~ 60 °C, respectively. Slowly pour the potassium permanganate solution, into the manganese sulfate solution, with constant stirring. Vigorously stir it. Then add 10 mL of (1 + 1) sulfuric acid. Heat the solution to 70 °C ~ 80 °C. Continue to stir for another 5 min. Stop heating. Let it stand for 2 h ~ 3 h. Use hot distilled water, to pour-wash it to neutrality. Move the precipitate into a funnel for filtration, to remove moisture. Then place it in a drying oven, for drying, at about 150 °C, for 2 h ~ 3 h, to obtain brown, loose manganese dioxide. Carefully crush and sieve it, to obtain a particle size of 0.5 mm ~ 2 mm, for later use.

3.2.3 Copper wire coil: The wire diameter is about 0.5 mm. Copper wire mesh: 0.15 mm (100 mesh).

3.2.4 Copper oxide: Chemically pure, linear (about 5 mm in length).

3.2.5 Lead chromate: Analytically pure, which is prepared to a particle size of 1 mm ~ 4 mm.

Preparation method: Use distilled water, to mix commercially available lead chromate into a paste. Extrude to shape it. Put it in a muffle furnace. Burn it at 850 °C, for 2 h. Take it out. Cool it, for later use.

3.2.6 Silver wire coil: The wire diameter is about 0.25 mm.

3.2.7 Oxygen: 99.9%, without hydrogen. Oxygen cylinders need to be equipped with a pressure gauge, which has a pressure reducing valve with adjustable flow (medical oxygen inhalers can be used).

3.2.8 Tungsten trioxide: Analytically pure.

3.2.9 Soda asbestos: Chemically pure, which has a particle size of 1 mm ~ 2

Figure 1 -- Schematic diagram of the carbon and hydrogen analyzers by three-segment furnaces and two-stage furnaces

3.3.1.1 Purification system, including the following components:

a) Gas drying tower: Capacity 500 mL, 2 units, the upper part (about 2/3) of one unit (A) is filled with anhydrous calcium chloride (or anhydrous magnesium perchlorate), the lower part (about 1/3) is filled with alkali asbestos (or soda lime); another one unit (B) is filled with anhydrous calcium chloride (or anhydrous magnesium perchlorate);

b) Flowmeter: Measuring range (0 ~ 150) mL/min.

3.3.1.2 Combustion device: It consists of a three-stage (or two-stage) pipe furnace and its temperature control system, mainly including the following components:

a) Electric furnace: three-stage furnace or two-stage furnace (double-pipe furnace or single-pipe furnace); the diameter of the furnace is about 35 mm.

Three-stage furnace: The first stage is about 230 mm long, which can be heated to (850 ± 10) °C, AND can move in the horizontal direction; the second stage is 330 mm ~ 350 mm long, which can be heated to (800 ± 10) °C; the third stage is 130 mm ~ 150 mm long, which can be heated to (600 ± 10) °C.

Two-stage furnace: The first stage is about 230 mm long, which can be heated to (850 ± 10) °C, AND can move in the horizontal direction; the second stage is 130 mm ~ 150 mm long, which can be heated to (500 ± 10) °C.

Each stage of the furnace is equipped with thermocouples, temperature measurement, temperature control devices.

b) Combustion pipe: It is made of bisque, quartz, corundum or stainless steel, which is about 1100 mm ~ 1200 mm in length (about 800 mm in length when using a two-stage furnace), has an inner diameter of 20 mm ~ 22 mm, AND a wall thickness of about 2 mm.

c) Burning boat: It is made of bisque or quartz, at about 80 mm long.

d) Rubber stopper or rubber cap (preferably with heat-resistant silicone rubber) or copper joint.

e) Nickel-chromium wire hook: It is about 2 mm in diameter AND about 700 mm in length; one end is bent into a hook.

Connect the containers of the absorption system, which is prepared in accordance with the requirements of 4.3.5, in the order as shown in Figure 6. Use polyvinyl chloride hoses or polytetrafluoroethylene pipes, to connect the oxygen purification system and the combustion pipe. Use silicon rubber pipe, to connect the electrolytic cell and the U-shaped pipe, AND to connect the U-shaped pipe and U-shaped pipe.

When the following phenomena occur, the reagents in the U-shaped pipe shall be replaced, OR the electrolytic cell shall be cleaned.

a) After a certain test, when the mass of the second U-shaped pipe for absorbing carbon dioxide increases by more than 50 mg, the first U-shaped pipe shall be replaced;

b) Manganese dioxide, anhydrous magnesium perchlorate or anhydrous calcium chloride shall generally be replaced, after about 100 times of use; meanwhile it shall be replaced, when the calcium chloride in the water-absorbing U-shaped pipe begins to melt and hinders the smooth flow of gas;

c) When the electrolytic cell is used about 100 times OR when the electrolytic cell is found to have tailings, etc., the electrolytic cell shall be cleaned and re-coated.

4.4.5 Air tightness inspection of the measuring system

Adjust the oxygen flow to about 80 mL/min; the others are the same as 3.4.5.

4.4.6 Reliability inspection of test device

Carry out according to 3.4.6. BUT weigh 0.070 g ~ 0.075 g of standard coal sample, accurate to 0.0002 g.

4.5 Test procedure

4.5.1 Select the polarity of the electrolysis power supply (it shall be interchanged once a day). Lead in oxygen and adjust the flow to about 80 mL/min. Connect the cooling water. Turn on the power to heat up.

4.5.2 At the same time as the temperature rises, connect the U-shaped pipe for carbon dioxide absorption (the ground plug of the U-shaped pipe shall be opened first) and the bubble meter, to keep the oxygen flow rate at about 80 mL/min. Press the electrolysis key (or pretreatment key) to the end. Then, press the electrolysis key (or pretreatment key) every 2 min ~ 3 min. After 10 min, remove the U-shaped pipe for carbon dioxide absorption. Close all the ground plugs of the U-shaped pipe. Place it next to the balance, for about 10 min. Weigh it. Then connect to the system and repeat the above test, until the mass change

of the two U-shaped pipes for carbon dioxide absorption does not exceed 0.0005 g.

4.5.3 Control the temperature of the combustion furnace, purification furnace, catalytic furnace, at the specified temperature. Mix the coal samples evenly. Weigh 0.070 g ~ 0.075 g of coal samples for general analysis, which have a particle size of less than 0.2 mm, in a pre-burned burning boat, accurate to 0.0002 g. Spread them evenly. Cover with a layer of tungsten trioxide. If the measurement is not performed immediately, the burning boat can be temporarily stored in an airtight container without desiccant.

4.5.4 Connect a U-shaped pipe for carbon dioxide absorption with constant mass. Keep the oxygen flow at about 80 mL/min. Start electrolysis, to the end of electrolysis. Zero the hydrogen integral value and time counter. Open the rubber stopper with the nickel-chromium wire push rod. Quickly put the burning boat into the inlet end of the combustion pipe. Plug the rubber stopper with the push rod. Use the push rod to push the burning boat, so that half of it enters the burner opening. After the coal sample is burned (generally 30 s), press the electrolysis key (or the determination key). When the coal sample burns stably, push the whole boat into the furnace opening. Stay for about 2 minutes. Then push the burning boat into the high temperature zone AND immediately pull back the push rod (do not let the red-hot part of the push rod pull close to the rubber stopper, so as not to cause the rubber stopper to be overheated and decomposed).

4.5.5 After about 10 minutes (the electrolysis reaches the end point; otherwise, the time needs to be extended appropriately), remove the U-shaped pipe for carbon dioxide absorption. Close the ground stopper. Place it next to the balance for about 10 minutes, before weighing. If the mass change of the second U-shaped pipe for carbon dioxide absorption is less than 0.0005 g, it is ignored in the calculation. Record the mass (mg) of hydrogen, which is displayed by the coulometric integrator. Open the rubber stopper with push rod. Use a nickel-chromium wire hook, to take out the burning boat. Plug the rubber stopper with push rod.

4.5.6 Determination of blank value

4.5.6.1 The determination of the hydrogen blank value can be carried out simultaneously, with the constant mass test of the U-shaped pipe for carbon dioxide absorption; OR it can be carried out after the determination of carbons and hydrogens.

4.5.6.2 After the combustion furnace, purification furnace, catalytic furnace reaches the specified temperature, keep the oxygen flow at about 80 mL/min. Start electrolysis to the end point. Add tungsten trioxide to a pre-burned burning boat (the amount is equivalent to that of coal sample analysis). Zero the

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