GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.040

D 21

GB/T 480-2010

Replacing GB/T 480-2000

Test of low temperature distillation of coal by aluminum retort

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(ISO 647:1974, Brown coals and lignites - Determination of the yields of tar, water, gas and coke residue by low temperature distillation, MOD)

Issued on: September 26, 2010 **Implemented on: February 01, 2011**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration of the People's Republic of China.

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Foreword

This Standard modifies and adopts ISO 647:1974, Brown coals and lignites - Determination of the yields of tar, water, gas and coke residue by low temperature distillation (English version).

This Standard is redrafted in accordance with ISO 647:1974. Compared with ISO 647:1974, there are some adjustments in the structure. Appendix A makes a comparison list of changes in chapter numbers between this Standard and ISO 647:1974.

In consideration of China's national conditions, when this Standard is adopted, there are technical differences compared with ISO 647:1974; the terms which are involved in these differences have been marked by vertical single lines (|) on the outer page margins; Appendix B gives a list of corresponding technical differences and their causes.

The main technical differences between this Standard and ISO 647:1974 are as follows:

-- Remove the calculation method summary of each product yield;

-- Retain only electrical heating for the type of heating; remove the gas heating method;

-- Add technical requirements for moisture measuring tubes, condensers and balances;

-- Add equipment drawings, air tightness inspection methods, and pretreatment methods for coal of strong expansibility;

-- Add regulations for the export method of dry distillation products, the method for determining the water content in the condensate (dry distillation total moisture) and the calculation formula for the yield of each product;

-- Remove the calculation of coal gas production rate;

-- Add conversion and correction formulas for the dry ash-free basis coke residue yield result basis;

-- Modify the precision for the determination of dry distillation product yield;

This Standard replaces GB 480-2000, Test for carbonization of coal in aluminum retort; compared with GB/T 480-2000, the main technical changes, except editorial changes, are as follows:

Test of low temperature distillation of coal by aluminum retort

1 Scope

This Standard specifies the method summary, instruments and apparatuses, test procedure and result expression of the test of low temperature distillation by aluminum retort.

This Standard applies to brown coal and soft coal.

2 Normative references

The terms in the following documents become the terms of this Standard by reference to this Standard. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this standard. However, parties to agreements that are based on this Standard are encouraged to study whether the latest versions of these documents can be used. For undated references, the latest edition applies to this Standard.

GB/T 212, Proximate analysis of coal (GB/T 212-2008, 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)

GB/T 218, Determination of the carbonate carbon dioxide content in coal (GB/T 218-1996, eqv ISO 925:1997)

GB 474, Preparation of coal sample (GB 474-2008, ISO 18283:2006, Hard coal and coke - Manual sampling, MOD)

GB/T 19494.2, Mechanical sampling of coal - Part 2: Method for sample preparation (GB/T 19494.2-2004, ISO 13909-1:2001 Hard coal and coke - Mechanical sampling - Part 1: General introduction; ISO 13909-4:2001 Hard coal and coke - Mechanical sampling - Part 4: Coal - Preparation of test samples, NEQ)

3 Method summary

Hold the coal sample in the aluminum retort; use a certain temperature-rising program to heat to 510 °C, and maintain for a certain period of time; measure the yields of tar, pyrolysis water, coke residue and coal gas that are obtained after dry distillation.

4 Materials and reagents

4.1 Xylene or toluene: chemically pure.

4.2 Lubricating oil and selected flake graphite powder.

5 Instruments and apparatuses

5.1 Aluminum retort

5.1.1 Aluminum retort body (Figure 1), made of aluminum;

5.1.2 Aluminum retort cover (Figure 2), made of aluminum;

5.1.3 Delivery line (Figure 3), made of copper;

5.1.4 Connecting nut (Figure 4), made of steel.

5.2 Heating device: with temperature control device; specifications and performance shall meet the following requirements:

a) The temperature shall be increased to 260 °C within the first (15 ~ 20) min;

b) During (260 ~ 510) °C, the heating rate is 5 °C/min. The total temperature rise error is ±10 °C;

c) The final temperature can reach above 510 °C.

6.3 Coal sample

The coal sample is an air-dried coal sample, whose particle size is less than 0.2 mm, that is prepared according to GB 474 or GB/T 19494.2.

6.4 Instrument assembly

6.4.1 Cover the retort cover. Use a small wooden hammer to lightly tap the retort cover tightly. Use a tight airtight cork stopper to connect the aluminum retort delivery line to the conical flask. Extend the aluminum retort delivery line into the bottle; the length of the extension shall not be less than half the height of the bottle, but shall not be in contact with the bottom of the bottle. Insert a slightly curved glass air duct from another small hole of the cork stopper for gas discharge. All junctions shall be airtight. The conical flask shall be washed, dried and weighed, to 0.01 g, in advance before it is connected to the aluminum retort.

6.4.2 Put the aluminum retort into the electric furnace; at the same time, put the conical flask into the cooling bath. Close the furnace cover; insert the thermocouple or thermometer into the temperature measuring hole of the aluminum retort.

6.4.3 Put a certain amount of water and ice cubes in the cooling bath; the conical flask shall be immersed in the water as much as possible, but the bottle mouth shall be higher than the water surface. Circulating water below 15 °C can also be used; but the circulating water flow shall be able to make tar and water vapor condense.

6.4.4 The test device shall be placed in a fume hood; otherwise, a rubber tube shall be connected to the air duct on the conical flask, so as to lead the gas out of the room.

7 Test procedure

7.1 Fully stir the coal samples; take (20 ± 0.5) g of coal sample (weigh to 0.01 g) from 4 ~ 5 different parts of the coal sample container; put it in a watch glass; then, transfer all the coal samples to the aluminum retort.

Note: If the preliminary test which is conducted with reference to Appendix D shows that the coal sample is very expandable, an appropriate amount of sand can be added to prevent it from expanding. Coal sample and sand shall be mixed evenly, and put in the retort; make the surface smooth.

7.2 After everything is prepared, it can be heated by electricity. The sides and bottom of the aluminum retort shall be heated at the same time, to keep the temperature of each part of the bottle body uniform. Within the first (15 ~ 20)

min, the temperature shall be raised to 260 °C; after reaching 260 °C, the heating rate shall be strictly controlled to 5 °C/min; the actual temperature shall be checked every 10 min, to keep the difference from the to-be-reached temperature within 10 °C. When it reaches 510 °C, keep it warm for 20 min and stop heating.

7.3 During the test, the tar, water vapor and coal gas which are produced after the coal sample is heated enter the conical flask through the delivery line. The tar and water vapor shall be condensed in the conical flask. The coal gas is led outside by the rubber tube.

7.4 After the heating is stopped, use an alcohol lamp or other heat source to slightly heat the aluminum retort delivery line, so that the tar that is attached to the delivery line wall flows into the conical flask.

7.5 After the tar that is attached to the delivery line wall flows out, drain the cold water in the cooling bath or remove the cooling bath; remove the thermocouple or thermometer; remove the cork stopper that connects the conical flask to the retort body; remove the aluminum retort; put it in the shade place to cool. In order to prevent the coke residue from absorbing moisture in the air, the delivery line of the aluminum retort shall be sealed at this time.

7.6 Dry the outer wall of the conical flask that contains the coolant; weigh it after placing it for about 5 min (weigh to 0.01 g). The difference between the mass of the conical flask that contains the condensate and the mass of the conical flask before dry distillation is the mass of the dry distillation condensate.

7.7 Determine the water content in the condensate (dry distillation total moisture) as described in 8. The difference between the mass of condensate and the mass of water is the mass of tar.

7.8 After the aluminum retort cools, remove the retort cover; pour the coke residue into the weighing bottle and weigh it (weigh to 0.01 g). Be careful not to scrape the retort body when scraping the coke residue on the retort wall.

7.9 The total coal sample minus the sum of the condensate mass and the coke residue mass is the mass and loss of coal gas. The mass fraction of each product to the mass fraction of the coal sample is the air-dried basis yield.

7.10 The pyrolysis water yield is obtained by subtracting the air-dried basis moisture M_{ad} (measured according to GB/T 212) from the dry distillation total moisture (7.6).

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