ICS 73.040
D 21

GB

National Standard

of the People's Republic of China

GB/T 4663-2014

Replacing GB/T 4663-1997

Determination of fluorine in coal

煤中氟的测定方法

Issued on: June 09, 2014 **Implemented on: October 01, 2015**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration Committee.

Table of Contents

Foreword

This Standard is drafted according to the rules of GB/T 1.1-2009.

This Standard replaces GB/T 4663-1997 Determination of fluorine in coal. Compared with GB/T 4663-1997, the major changes of this standard are as follows:

- ADD normative references (see Chapter 2);

- ADD result reports (see Chapter 10);

- MODIFY the calculation formula of fluorine content in coal;

This Standard was proposed by China National Coal Association.

This Standard shall be centralized by National Coal Standardization Technical Committee of China (SAC/TC 42).

Drafting organization of this Standard: China Coal Research Institution Testing Research Branch.

The main drafters of this standard: Yang Yuhua, Shi Mingzhi, and Li Ting.

The previous versions replaced by this Standard are:

- GB/T 4663-1984; GB/T 4663-1997.

Determination of Fluorine in Coal

1 Scope

This Standard specifies the method abstract, reagents and materials, apparatus and device, measurement steps, result calculation, method precision, and test reports of using high temperature combustion hydrolysis - fluoride ion selective electrode (abbreviated as: fluoride electrode) method to determine the fluorine content in coal.

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 editions apply to this document. For undated references, the latest editions (including all corrections) of the referenced documents apply to this document.

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

GB/T 6682 Water for analytical laboratory use - specification and test methods

3 Method abstract

Samples of coal are burned and hydrolyzed in the mixed gas stream of oxygen and vapor. All of the fluorine in coal are transferred into volatile fluoride (SiF_4 and HF) and dissolved in water quantitatively. Use fluorine electrode as the indication electrode; and saturated calomel electrode as the reference electrode. Use standard addition method to determine the fluoride ion concentration in the sample solution. Calculate the fluorine content in coal samples.

4 Reagents and materials

Unless otherwise indicated, it shall only use the analytic reagents and distilled water that are confirmed qualified, or deionized water, or water with equivalent purity.

4.1 Water: GB/T 6682, Grade 2.

4.2 Silica sand (SiO_2): Particle size is 0.5mm~1.0mm.

circuit, gas circuit and cooling waterway. Squeeze a few refractory wool in air outlet of combustion tube. Rise the temperature of high temperature furnace to 1100°C. Use another platinum rhodium - platinum thermocouple pyrometer to measure the 300°C, 600°C, and 900°C temperature zones' positions; and the position and length of 1100°C high-temperature zone in the combustion tube.

6.1.2 Add 300mL of water into the flat-bottomed flask. And heat to a boil. Open the condenser pipe to cool the water. Stuff up the silicon stopper of injection push rod. Access the oxygen into the flat-bottomed flask before the water is boiled. Adjust the oxygen flow to 400mL/min. The water evaporation capacity of water steam generator is about 2mL/min. After checking that the system is not leaked, Access the steam and oxygen into it. Emptily steam for 15min.

6.2 Combustion and hydrolysis

6.2.1 Weigh (0.50±0.01) g of general-analysis test coal (accurate to 0.1mg) and 0.5g of quartz. Place in porcelain boat uniformly and carefully. Then cover it with about 0.5g of quartz.

6.2.2 Place 100mL volumetric flask under the condenser pipe to receive condensate. Access in oxygen and water vapor. Take down the injection push rod. Place the porcelain boat into combustion tube. Insert the injection push rod. Stuff with silicon stopper. Push the front-end of the porcelain boat to the 300°C zone where has been measured in advance for 5 min. Push the porcelain boat into the 600°C zone for 5min. Then push it into the 900°C zone for 5min. Finally, push it into the 1100°C constant temperature zone. Return the injection push rod. The sample continues to burn and resolve for 15min in the constant temperature zone. During the whole operating process, adjust the evaporation capacity of the water steam generator. Within 15min of starting of hydrolysis, about 3mL of water is collected in the volumetric flask per min; for later 15min, collect about 2.5mL per min. The final total volume shall be controlled within 85mL.

6.2.3 Add 3 drops of bromocresol green indicator into volumetric flask which contains condensate. Then neutralize it with sodium hydroxide solution until it turns to blue. Add 10mL of total ionic strength adjustment buffer solution. Dilute it to the scale with water. Shake well. Stand for 30min.

7 Potential measurement

7.1 Preparation of titration

Add a certain amount of water into 100mL beaker. Connect the device of potential measurement according to Figure 4. Turn on the blender. Change the water in beaker for several times until the potential value showed in digital ion meter (or millivolt meter)

— abnormal phenomena occurred in the test;

— test date.

_____ END _____

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------