GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.040

D 21

GB/T 25214-2010

Determination of total sulfur in coal by IR spectrometry

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(ISO 19579:2006, Solid mineral fuels - Determination of sulfur by IR spectrometry, 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 uses ISO 19579:2006, Solid mineral fuels - Determination of sulfur by IR spectrometry.

This Standard is redrafted in accordance with ISO 19579: 2006. Appendix A lists a comparison list of chapter numbers of this Standard and chapter numbers of ISO 19579: 2006.

In consideration of China's national conditions, while adopting ISO 19579: 2006, this Standard makes some modifications. Relevant technical differences have been incorporated into the text and are marked with a single vertical line in the margins of the articles to which they relate. Appendix B provides a list of these technical differences and their causes for reference.

The main technical differences between this Standard and ISO 19579: 2006 are as follows:

-- Use 1300 °C instead of 1350 °C as the sample combustion temperature;

-- Add the schematic diagram of instruments and apparatus and the description of the composition system;

-- Use about 0.3 g instead of (0.2 ~ 0.5) g as the weighing sample;

-- Combine the calibration content and calibration check in the step into a separate clause; add detailed calibration step requirements;

-- Determine the method precision according to domestic collaborative experiments in China; give it in stages according to the range of total sulfur content.

For convenience of use, this Standard also makes the following editorial changes:

-- Change "this International Standard" into "this Standard";

-- Use the decimal point “.” to replace the comma ",";

-- Delete the foreword and introduction of ISO 19579: 2006.

Appendix A and Appendix B of this Standard are informative appendixes.

This Standard was proposed by China National Coal Association.

This Standard shall be under the jurisdiction of National Technical Committee 42 on Coal of Standardization Administration of China (SAC/TC 42).

Determination of total sulfur in coal by IR spectrometry

1 Scope

This Standard specifies the method summary, reagents and materials, instruments and apparatus, determination, calibration, and method precision for the determination of total sulfur in coal by high-temperature combustion IR spectroscopy.

This Standard applies to lignite, soft coal, hard coal, and cokes.

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 483, General rules for analytical and testing methods of coal (GB/T 483-2007, ISO 1213:1992, Solid mineral fuels - Vocabulary - Part 2: Terms relating to sampling, testing and analysis, NEQ)

3 Method summary

The coal sample is combusted and decomposed at 1 300 °C in a stream of oxygen. Particles and water vapor are removed from the gas stream by traps of glass wool and magnesium perchlorate. The gas stream then passes through a cell in which the sulfur dioxide is measured by an infrared absorption detector. Use standard reference materials to calibrate the instrument. The percentage of sulfur in the sample is calculated from this prior calibration by the microprocessor.

7.2.1 Measure the moisture content of air-dried basis of certified coal reference material according to GB/T 212; convert the sulfur standard value to air-dried basis total sulfur St, ad.

7.2.2 According to the determination procedure, use the calibrated instrument to determine the total sulfur content of certified coal reference materials. Repeatedly measure each reference material four times; take the average value of the four measured values as the total sulfur measurement value of the certified coal reference material.

7.2.3 Input the total sulfur measurement value and standard value (converted air-dried basis value) of certified coal reference material into the sulfur measuring instrument (or the instrument automatically reads it), to generate the working curve or correction coefficient.

Note: for some instruments, the correction coefficient needs to be calculated manually; enter the coefficient into the sulfur meter after calculation.

7.3 Calibration validity check

Additionally select (1 ~ 2) certified coal reference material(s) or other control samples; use the sulfur measuring instrument that has been calibrated to determine its total sulfur content; if the difference between the measured value and the standard value (or control value) is within the range of the combined uncertainty of the standard value (or control value) and the measured value, the calibration is valid; otherwise, find out the cause and recalibrate.

7.4 Calibration check

For the calibration check, during the sample measurement, use a coal sample of known total sulfur content or a certified coal reference material to perform the measurement. When the measurement value is not within the repeatability limit of the known coal sample measurement value or within the uncertainty range of the standard value, find the cause, and solve the problem; recalibrate the instrument according to step of 7.2 if necessary; re-test the test result that is completed before the check. The calibration check is recommended to be performed at the beginning and end of each batch of samples. When the sample size is large, insert (1 ~ 2) time(s) in the middle of the test.

8 Result expression

The total sulfur content of the tested samples (in terms of mass fraction, %) is reported, to 0.01% according to GB/T 483, as the average value of the repeated measurement results.

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