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Solid mineral fuels — Determination of sulfur by IR spectrometry

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British Standards

National foreword

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Foreword

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 19579 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

Introduction

Sulfur is normally present in coal in three forms; inorganic sulfides such as pyrite (FeS_2), inorganic sulfates associated with the mineral matter and organic sulfur in the carbonaceous substance.

This International Standard describes a high-temperature combustion/infrared absorption method of analysis, which is used to determine the total sulfur content of coal.

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Solid mineral fuels — Determination of sulfur by IR spectrometry

1 Scope

This International Standard specifies an alternative method of determining the total sulfur content of hard coal, brown coal, and lignite by high-temperature combustion and infrared (IR) absorption using commercially available instruments.

This method has been shown to be applicable to coal samples having an ash yield of less than 40 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 334, *Solid mineral fuels — Determination of total sulfur — Eschka method*

ISO 351, *Solid mineral fuels — Determination of total sulfur — High temperature combustion method*

ISO 687, *Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample*

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 11722, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

3 Principle

The coal sample is combusted at 1 350 °C in a stream of oxygen. Particulates and water vapour 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, connected to a microprocessor. The instrument is calibrated with standard reference materials. The percentage of sulfur in the sample is calculated from this prior calibration by the microprocessor.

4 Apparatus

4.1 Instrument, commercially available, consisting of the following:

a) resistance furnace, capable of maintaining a temperature of approximately 1 350 °C in the combustion zone;

b) combustion tube, of ceramic material, to contain the sample and combustion gases;

c) combustion boats, of ceramic material, into which the sample is weighed and then combusted;

d) combustion gas-purification train, through which the combustion gases pass from the combustion tube to the detector (the train consists of a glass wool filter and a trap containing anhydrous magnesium perchlorate);

e) detector/microprocessor, a non-dispersion infrared measurement system.

4.2 **Balance**, capable of weighing to 1 mg.

5 Reagents

5.1 **Magnesium perchlorate**, anhydrous granular.

5.2 **Oxygen**, compressed industrial grade, 99,5 % by volume.

5.3 **Glass wool**.

5.4 **Reference coals or cokes**.

Certified reference materials (CRMs) are coal or coke samples of known sulfur concentration analysed in accordance with ISO 334 or ISO 351. The reference materials shall be traceable to an internationally recognized certifying organization.

NOTE 1 Calibration with coals or cokes that are similar in composition to the analysis samples is necessary. It has been noted that some coals containing high levels of carbonates and some low-rank coals containing high levels of carboxylic salts of calcium can require longer combustion/integration times, as the release of sulfur dioxide can be delayed during combustion.

If coal or coke samples (other than CRMs) are used for calibrations, then it is necessary to verify the calibration. This is done by analysing an appropriate CRM after calibration. If the results of the analysis of the CRM do not agree with the certified value of the sulfur content, the instrument should be recalibrated.

NOTE 2 Reference materials including CRMs can contain moisture and are usually certified on a dry basis. It is necessary to determine the moisture content in accordance with ISO 11722, ISO 687 and ISO 5068-2 as appropriate. This moisture is used to calculate the dry-basis sulfur value to an “as analysed” value, $w_{\mathrm{S,aa}}$, expressed in percent, as given in Equation (1):

$$w_{\mathrm{S,aa}} = w_{\mathrm{S,d,ac}} \times (100 - w_{\mathrm{H_2O}})/100 \qquad (1)$$

where

$w_{\mathrm{S,d,ac}}$ is the dry-basis sulfur content, expressed in mass percent;

$w_{\mathrm{H_2O}}$ is the moisture content, expressed in mass percent.

6 Preparation of sample

The coal or coke used for the determination of total sulfur content is the analysis sample ground to pass a sieve of 212 µm aperture.

Expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere. Before commencing the determination, mix the air-dried sample.

After weighing the test portion (see Clause 7), determine the moisture content using a further portion of the test sample by the method described in ISO 11722, ISO 687 or ISO 5068-2, as appropriate.

7 Procedure

Prepare the instrument according to the manufacturer's instructions. It is advisable to process two samples to condition the instrument prior to calibration. Calibrate the instrument using a reference coal or coke sample or reference coal or coke samples if a multi-point calibration is possible. The calibration should cover the range of sulfur concentrations below and above that present in the samples.

Use the calibration procedure recommended by the instrument manufacturer. Accurately weigh, to 0,001 g, 0,2 g to 0,5 g of the reference coal or coke into the combustion boat and enter the weight into the processor (computer). Push the boat plus sample into the hot zone of the furnace. The combustion products are pumped through the traps to the IR detector, where the absorption signal due to sulfur dioxide is integrated. At completion of the burn (indicated by the instrument), remove the boat containing the combustion residue.

NOTE Some instruments are designed for automatic operation and manual sample introduction and removal is not necessary.

Repeat the process with a further two replicates of the reference coal or coke. Check the accuracy of the calibration by analysing the calibrating reference coal or coke and another reference coal or coke of lower sulfur concentration as unknown samples. If the results obtained for the reference coals or cokes are not within the repeatability of the method (see 10.1), repeat the calibration procedure.

Determine the sulfur concentration in the sample by the same procedure as used for the reference coals or cokes.

8 Calibration check

A calibration check shall be completed periodically (it is recommended that this be after every five determinations). A standard of known sulfur content, not necessarily a CRM, shall be analysed.

If the result does not agree with the concentration of sulfur in the standard within the repeatability of the method, the preceding results shall be discarded and the calibration repeated (Clause 7) and the samples reanalysed.

9 Expression of results

The sulfur content of the sample as analysed is calculated by the microprocessor and expressed as a mass percent. The result, the mean of duplicate determinations, shall be reported to the nearest 0,01 mass %.

10 Precision

10.1 Repeatability

The results of duplicate determinations carried out at different times in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same analysis sample, shall not differ by more than the value of the repeatability, r, as given by Equation (2):

$$r = 0{,}02 + 0{,}03\,\overline{x} \tag{2}$$

where $\overline{x}$ is the mean of the intra-laboratory results.

10.2 Reproducibility

The means of the results of duplicate determinations, carried out in each of two laboratories, on representative portions taken from the same sample after the last stage of sample preparation, shall not differ by more than the value of the reproducibility, R, as given by Equation (3):

$$R = 0{,}02 + 0{,}09\,\bar{x} \tag{3}$$

where $\bar{x}$ is the mean of the inter-laboratory results.

11 Test report

The test report shall include the following particulars:

a) identification of the sample tested;

b) reference to the method used;

c) results and the method of expression used;

d) moisture content, if result is reported on an air-dried (as-received) basis.

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