
**Solid mineral fuels — Determination
of total sulfur by Coulomb titration
method**

*Combustibles minéraux solides — Détermination de la teneur totale
en soufre par la méthode de titrage de Coulomb*

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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Introduction

The objective of this document is to provide an alternative method for determining the total sulfur content in solid mineral fuels with high-temperature combustion — Coulomb titration using automated instruments.

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Solid mineral fuels — Determination of total sulfur by Coulomb titration method

1 Scope

This document specifies an alternative method of determining the total sulfur content of coal and coke by high-temperature combustion — Coulomb titration.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

In the presence of the catalyst, a weighed sample is combusted at 1 150 °C in a stream of purified air. The sulfur oxides are formed and dissolved in the electrolytic solution. The sulfur dioxides are titrated by the iodine and bromine liberated from the electrolysis of the potassium iodide and potassium bromide solution. The quantity of the electricity consumed by electrolysis is integrated and used to calculate the content of sulfur dioxides. The instrument is calibrated with certified reference materials. The percentage of total sulfur in the sample is calculated according to the sulfur dioxides content and the prior calibration.

5 Reagent and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Potassium iodide.

5.2 Potassium bromide.

5.3 Glacial acetic acid.

5.4 Self-indicating silica gel, industrial grade.

5.5 Sodium hydroxide, chemical grade.

5.6 Electrolytic solution.

5 g of potassium iodide (5.1) and 5 g of potassium bromide (5.2) are dissolved in (250 to 300) ml of water, then add 10 ml of glacial acetic acid.

5.7 Glass wool.

5.8 Aluminosilicate wool.

5.9 Catalyst, tungsten trioxide.

5.10 Certified reference materials (CRMs), coal or coke.

6 Apparatus

6.1 Instrument.

For determining sulfur by Coulomb titration, commercially available, consisting of the following (see [Figure 1](#)).

6.2 Tube resistance furnace and combustion tube.

Capable of maintaining a temperature of $(1\ 150 \pm 10)$ °C in the combustion zone which is at least 70 mm long, with the Pt-Rh—Pt thermocouple and the temperature controller. Combustion tube is made of silicon or corundum, capable of withstanding temperatures greater than 1 300 °C.

6.3 Electrolytic cell and magnetic stirrer.

Electrolytic cell consists of a cylindrical glass cup of over 400 ml capacity, with (120 to 180) mm high, in which there are a pair of Pt electrolytic electrodes each of 150 mm² area and a pair of Pt indicating electrodes each of 15 mm² area. The response time of indicating electrodes should be less than 1 s. The magnetic stirrer with speed of 500 r/min and regulator is also provided.

6.4 Coulomb integrator.

The integration linearity error is less than 0,1 % at (0 to 350) mA of electrolyzing current, with a (4 to 6) digital display or printer to give the content of the sulfur in the sample.

6.5 Program controller.

The sample can be pushed into and withdrawn from the combustion tube according to the specified program.

6.6 Purified air stream supply system.

Consists of the magnetic pumps and purifying tubes. Supply an air stream at the rate of 1 500 ml/min and draw it at 1 000 ml/min, which is purified and dried by the sodium hydroxide and self-indicating silica gel packed in the purifying tubes respectively.

NOTE The sodium hydroxide is not necessary for the laboratories in which the air is not polluted by acid gas.

6.7 Combustion boat.

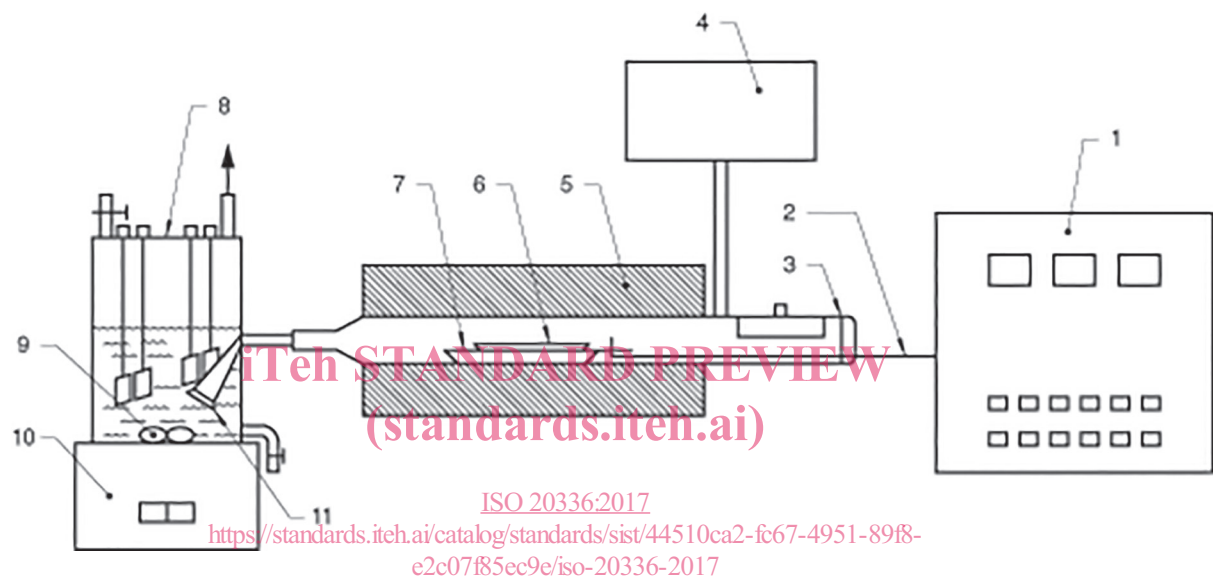
Made of unglazed porcelain or quartz, about 60 mm long at the part of packing the sample, capable of withstanding over 1 200 °C.

6.8 Quartz tray.

Capable of withstanding over 1 200 °C and supporting the combustion boat moving in and out the combustion tube.

6.9 Analytical balance.

Capable of weighing to the nearest 0,1 mg.



Key

- 1 program controller
- 2 pulling stem
- 3 combustion tube
- 4 purified device for air stream
- 5 resistance furnace
- 6 sample boat
- 7 quartz tray
- 8 electrolytic cell
- 9 stirrer stem
- 10 magnetic stirrer
- 11 gas filter

Figure 1 — Instrument for determining sulfur by Coulomb titration

7 Preparation of test sample

The coal or coke used for the determination of total sulfur content is the general analysis test sample ground to pass a sieve of 212 µm aperture, and for the coal or coke with higher than 5 % of the sulfur, the test sample ground to pass a sieve of 100 µm aperture.