INTERNATIONAL STANDARD

ISO 334

Third edition
2013-10-15

Solid mineral fuels — Determination of total sulfur — Eschka method

Combustibles minéraux solides — Dosage du soufre total — Méthode Eschka

Reference number
ISO 334:2013(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. www.iso.org/directives

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 334:1992), of which it constitutes a minor revision.

Introduction

An alternative reference method to that specified in this International Standard is given in ISO 351:1996.

Instrumental methods for a more rapid determination of total sulfur are now available. If such a method is to be used, it is important to demonstrate that the method is free from bias, when compared to this reference method, and will give levels of repeatability and reproducibility which are the same as, or better than, those quoted for the reference method (see Clause 9).

Solid mineral fuels — Determination of total sulfur — Eschka method

1 Scope

This International Standard specifies a reference method for determining the total sulfur content of hard coal, brown coals and lignites, and coke by the Eschka method.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 1170, *Coal and coke — Calculation of analyses to different bases*

ISO 5068-1, *Brown coals and lignites — Determination of moisture content — Part 1: Indirect gravimetric method for total moisture*

ISO 5069-2, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*

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

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

3 Principle

A test portion is ignited in intimate contact with the Eschka mixture in an oxidizing atmosphere at 800 °C to remove combustible matter and to convert the sulfur to sulfate. This is then extracted with hydrochloric acid solution and determined gravimetrically by precipitation with barium chloride.

4 Reagents

WARNING — Care should be exercised when handling the reagents, many of which are toxic and corrosive.

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

4.1 Eschka mixture

Mix two parts by mass of light calcined magnesium oxide with one part by mass of anhydrous sodium (or potassium) carbonate. The mixture shall entirely pass a test sieve of 212 µm nominal size of openings.

4.2 Hydrochloric acid

Concentrated ρ approximately 1,18 g/ml, approximately 36 % (m/m).

4.3 Potassium sulfate solution

Weigh, to the nearest 0,1 mg, about 2 g of potassium sulfate, previously dried at a temperature of 105 °C to 110 °C. Dissolve in water and dilute to 1 l.

4.4 Barium chloride, approximately 85 g/l solution

Dissolve 100 g of barium chloride dihydrate in water and dilute to 1 l. Filter before use through a close-textured, doubly acid-washed filter paper or filter-paper pad.

4.5 Methyl red indicator solution

Dissolve 1 g of 2-(4-dimethylaminophenylazo) benzoic acid sodium salt (methyl red) in 1 l of water.

4.6 Ammonia

Concentrated solution, not less than 25 % (*m*/*m*).

4.7 Silver nitrate, 17 g/l solution

Dissolve 17 g of silver nitrate in water and dilute to 1 l. Store in a dark, glass bottle.

5 Apparatus

5.1 Analytical balance

Capable of weighing to the nearest 0,1 mg.

5.2 Graduated glassware

Conforming to the requirements for class A in the International Standards prepared by ISO/TC 48.

5.3 Electrically heated muffle furnace

Capable of being maintained at a temperature of 800 °C ± 25 °C. The ventilation through the muffle furnace shall be such as to give about five air changes per minute.

5.4 Crucible

Of platinum, silica, or glazed porcelain, of capacity approximately 25 ml.

5.5 Flat plate

6 mm thick, of silica (or other suitable refractory material) which fits easily into the muffle furnace (5.3).

5.6 Gooch crucible

Of glazed porcelain or sintered glass.

5.7 Air oven

Capable of being maintained at a temperature of 130 °C ± 10 °C.

6 Preparation of test sample

The test sample is the general analysis test sample prepared in accordance with ISO 13909-4 or ISO 5069-2, as appropriate. Expose the sample, in a thin layer, for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere (this is particularly important for brown coals and lignites).

Before commencing the determination, thoroughly mix the test sample for at least 1 min, preferably by mechanical means.

If the results are to be calculated other than on an "air-dried" basis (see Clause 8), then, after weighing the test portion (see 7.1), determine the moisture content using a further portion of the test sample by the method described in ISO 11722, ISO 687, or ISO 5068-1, as appropriate.

7 Procedure

7.1 Test portion

7.1.1 For coal

Take a test portion of the mass given in Table 1 (for the expected total sulfur content), weighing to the nearest 0,1 mg.

Table 1 — Test portion for coal

Expected total sulphur content % (m/m)	Mass of test portion g
<5	1,0
5 to 10	0,5
>10	0,25

7.1.2 For coke

Take a test portion of 1 g, weighing to the nearest 0,1 mg.

7.2 Charging the crucible

Cover the bottom of the crucible (5.4) uniformly with 0,5 g of the Eschka mixture (4.1), weighed to the nearest 0,1 mg. Mix the test portion intimately with 2,5 g of the Eschka mixture, weighed to the nearest 0,1 mg, in a suitable vessel. Transfer the mixture to the 25 ml crucible. Level the contents by tapping the crucible gently on the bench and cover the contents uniformly with 1,0 g of the Eschka mixture, weighed to the nearest 0,1 mg.

The entire 4 g of the Eschka mixture should be weighed out and the 0,5 g and 1 g portions, required for the bottom and top layers, should be extracted from this. For this purpose, it is convenient to calibrate a small glass tube for each bath of the Eschka mixture to deliver 0,5 g and 1 g without weighing. The bottom layer of the Eschka mixture below the test portion mixture reduces attack on the porcelain surface so that the extraction of sulfate with hot water is complete even when the surface deteriorates.