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Solid mineral fuels — Determination of arsenic and selenium — Eschka's mixture and hydride generation method

Combustibles minéraux solides — Dosage de l'arsenic et du sélénium — Mélange d'Eschka et méthode par production d'hydrure

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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 (see 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 (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11723:2004), which has been technically revised. This document incorporates changes related to dated references and other minor items following its systematic review.

Solid mineral fuels — Determination of arsenic and selenium — Eschka's mixture and hydride generation method

1 Scope

This document specifies a method using Eschka's mixture during ashing, extraction of the ash residue with acid, and hydride generation atomic absorption spectrometry or hydride generation atomic fluorescence spectrometry, for the determination of arsenic and selenium in solid mineral fuels.

NOTE The method is also applicable to the determination of the analytes by hydride generation inductively coupled plasma atomic emission spectrometry.

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 1170, *Coal and coke — Calculation of analyses to different bases*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

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*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

A known mass of the sample is ignited in intimate contact with Eschka's mixture in an oxidizing atmosphere at 800 °C to remove the organic matter. The residue is then extracted with hydrochloric acid and the analytes determined by hydride generation atomic absorption spectrometry or hydride generation atomic fluorescence spectrometry.

5 Reagents

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

During the analysis, unless otherwise stated, use only reagents of recognized analytical reagent grade and water conforming to Grade 1 of ISO 3696.

5.1 Eschka's mixture, composed of two parts by mass of light magnesium oxide and one part by mass of anhydrous sodium carbonate.

5.2 Hydrochloric acid (ρ_{20} 1,19 g/ml).

5.3 Nitric acid (ρ_{20} 1,42 g/ml).

5.4 Potassium iodide solution, 500 g/l. Dissolve 50 g of analytical reagent (AR) grade KI in distilled water and adjust the volume to 100 ml (for determination of As).

5.5 Sodium borohydride solution. Weigh 1,50 g of sodium borohydride ($NaBH_4$) and 0,4 g of sodium hydroxide (NaOH) into a plastic bottle of 125 ml capacity and add 100 ml of distilled water. Prepare freshly on the day of use. Alternatively, commercially available pressed pellets of sodium borohydride may be used.

5.6 Arsenic stock solution (100 mg/l). Dissolve 0,132 0 g ± 0,000 5 g of arsenic trioxide (As_2O_3 of 99,9 % purity, dried at 110 °C for 1 h) in 25 ml of 20 % KOH solution. Add approximately 50 ml of water and 20 ml of hydrochloric acid (5.2). Adjust the volume with water to 1 litre in a volumetric flask. Alternatively, dissolve 0,416 5 g ± 0,000 5 g of sodium arsenate ($Na_2HAsO_4.7H_2O$) in approximately 100 ml of water. Add 10 ml of hydrochloric acid (5.2) and adjust the volume with water to 1 l in a volumetric flask.

NOTE A commercially available certified stock solution may be used as an alternative.

5.7 Dilute arsenic stock solution (1 mg/l). Transfer 5 ml of arsenic stock solution to a 500 ml volumetric flask. Add approximately 100 ml of water and 5 ml of hydrochloric acid (5.2). Adjust to volume with water.

5.8 Arsenic working standard (50 µg/l). Transfer 5 ml of dilute arsenic stock solution (5.7) to a 100 ml volumetric flask. Add approximately 20 ml of water and of 1 ml hydrochloric acid (5.2). Adjust to volume with water. Prepare freshly on the day of measurement.

5.9 Selenium stock solution (100 mg/l). Dissolve 0,100 0 ± 0,000 5 g of elemental selenium (of 99,9 % purity) in a minimum of nitric acid in a beaker. Evaporate to dryness. Add 2 ml of water and evaporate to dryness; repeat this procedure twice. Add 10 ml of water to the residue and 10 ml of hydrochloric acid (5.2). Dissolve the residue by heating. Allow the solution to cool and adjust the volume with water to 1 l in a volumetric flask. Alternatively, dissolve 0,467 4 g ± 0,000 5 g of sodium selenate ($Na_2SeO_4.10H_2O$) in approximately 100 ml of water. Add 10 ml of hydrochloric acid (5.2) and adjust the volume with water to 1 l in a volumetric flask.

NOTE A commercially available certified stock solution may be used as an alternative.

5.10 Dilute selenium stock solution (1 mg/l). Transfer 5 ml of selenium stock solution (5.9) to a 500 ml volumetric flask. Add approximately 100 ml of water and 5 ml of hydrochloric acid (5.2). Adjust to volume with water.