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Solid mineral fuels — Determination of total fluorine in coal, coke and fly ash

Combustibles minéraux solides — Détermination de la teneur totale en fluor dans le charbon, le coke et les cendres

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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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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 11724:2016), of which it constitutes a minor revision. This document incorporates changes related to dated references and other minor items following its systematic review.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Solid mineral fuels — Determination of total fluorine in coal, coke and fly ash

1 Scope

This document specifies a method for the determination of total fluorine in coal, coke and fly ash.

From measurement of the total fluorine alone, it is not possible to estimate the amount of fluorine released to the environment by utilization of the coal and subsequent disposal of the ash residue.

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 589, *Hard coal — Determination of total moisture*

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*

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 defined in this document.

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

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

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

4 Principle

Mixing of the sample of coal, coke or fly ash with silica, and pyrohydrolysis in a tube furnace at approximately 1 200 °C in an atmosphere of oxygen and water vapour. Absorption of the volatilized fluorine compounds in a suitable solution and processing for determination by ion-selective electrode (ISE) or ion chromatographic (IC) techniques.

5 Reagents

CAUTION — Care shall be exercised when handling reagents, some of which are toxic and corrosive.

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

5.1 Silica of top size 75 µm, ignited at 1 000 °C for 1 h.

CAUTION — Fine silica is dangerous to health if inhaled.

5.2 Solutions for ISE measurement.

5.2.1 Standard fluorine solution (1 g contains 200 µg of F).

a) For direct-comparison method:

Dissolve 0,221 0 g ± 0,000 2 g of dry (110 °C for 1 h) sodium fluoride in approximately 400 ml of water contained in a tared plastic bottle, dilute to 500 g ± 0,5 g net with water, and mix.

b) For analyte-addition method:

Dissolve 0,221 0 g ± 0,000 2 g of dry (110 °C for 1 h) sodium fluoride in a tared plastic bottle containing 150 ml of water and 100 g of buffer (5.2.3). Dilute to 500 g ± 0,5 g net with water and mix.

5.2.2 Absorption solution (0,025 mol/kg NaOH).

Dissolve 2,0 g of sodium hydroxide in about 500 ml of water. Transfer to a tared 2,5 l plastic bottle, dilute to 2 000 g net with water, and mix.

5.2.3 Buffer (pH 6,5).

Dissolve 10,0 g of potassium nitrate, 5 g of 1,2-cyclohexylenedinitrilotetraacetic acid (CDTA) and 115 g of ammonium acetate in 350 ml of water. Adjust the pH to 6,5 with glacial acetic acid. Dilute to 500 g net with water and mix.

5.2.4 Solution for conditioning fluoride ISE.

Weigh 20 g of water, 20 g of absorption solution (5.2.2) and 10 g of buffer (5.2.3) into a polystyrene vial (6.2). Add approximately 200 mg of standard fluorine solution [of 5.2.1 a) or 5.2.1 b)] and mix.

5.3 Solutions for IC measurement

5.3.1 Absorption solution for IC measurement.

Dissolve 0,300 g of sodium hydrogen carbonate and 1,120 g of sodium carbonate in approximately 500 ml of water and dilute to 2 l.

5.4 Oxygen, compressed.

6 Apparatus

6.1 Vials, made of glass or polystyrene, of capacity 10 ml to 30 ml with tightly fitting snap-on plastic caps.

6.2 Polypropylene bottles or polystyrene vials, tared, of capacity 125 ml, wide necked with linerless leak-proof screw caps.

6.3 Weighing device: an analytical balance with a resolution of at least 0,1 % relative of the test portion mass.

6.4 Polyethylene dispensing bottles, for the standard fluorine solution (5.2.1), absorption solution (5.2.2) and buffer (5.2.3).