



SLOVENSKI STANDARD
SIST ISO 11724:2005

01-november-2005

Trda goriva -- Določitev skupne vsebnosti fluora v oglju, koksu in pepelu

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

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

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Solid fuels

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INTERNATIONAL
STANDARD

ISO
11724

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2004-11-01

**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

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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 11724 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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

1 Scope

This International Standard 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 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 589:2003, *Hard coal — Determination of total moisture*

ISO 687:1974, *Coke — Determination of moisture in the analysis sample*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*

ISO 11722:1999, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

ISO 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-6:2001, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

3 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.

4 Reagents

CAUTION — Care must 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.

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

CAUTION — Fine silica is dangerous to health if inhaled.

4.2 Solutions for ISE measurement

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

a) For direct-comparison method

Dissolve 0,221 0 ± 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 ± 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 (4.2.3). Dilute to 500 g ± 0,5 g net with water, and mix.

4.2.2 Absorption solution (0,025 mol/l NaOH).

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

4.2.3 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.

4.2.4 Solution for conditioning fluoride ISE. [SIST ISO 11724:2005](https://standards.iteh.ai/catalog/standards/sist/fc0c0166-fca4-484b-b5c6-7360411742-331-iso-11724-2005)

Weigh 20 g of water, 20 g of absorption solution (4.2.2) and 10 g of buffer (4.2.3) into a polystyrene vial (5.2). Add approximately 200 mg of standard fluorine solution [of 4.2.1 a) or 4.2.1 b)], and mix.

4.3 Solutions for IC measurement

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

Dissolve 0,221 0 ± 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.

4.3.2 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 litres.

4.4 Oxygen, compressed.

5 Apparatus

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

5.2 Polypropylene bottles or polystyrene vials, tared, of capacity 125 ml, wide necked with linerless leakproof screw caps.

5.3 Balances: a balance capable of weighing to the nearest 0,000 1 g; a balance capable of weighing approximately 200 g to the nearest 0,001 g; a balance capable of weighing 600 g to the nearest 0,01 g; and a balance capable of weighing 3 000 g to the nearest 0,1 g.

5.4 Polyethylene dispensing bottles, for the standard fluorine solution (4.2.1), absorption solution (4.2.2) and buffer (4.2.3).

5.5 Micropipette, of variable volume, ranging to at least 1 ml.

This is a satisfactory alternative to the 250 ml polyethylene dispensing bottle (5.4) for delivery of small weighed volumes of standard fluorine solution (4.2.1).

5.6 Apparatus for tube-furnace pyrohydrolysis (see Figure 1).

5.6.1 Steam generator, consisting of the following:

- a) round-bottom flask, 2 litre capacity;
- b) heating mantle, 2 litre capacity;
- c) Y-piece, glass, 10 mm diameter;
- d) oxygen distribution tube, porosity 0;
- e) stopcocks, one three-way and one two-way.

5.6.2 Silica-tube furnace and accessories

The apparatus is similar to that used in ISO 351.

a) **Silica tube**, made of translucent pure silica (25 mm outer diameter, 20 mm inner diameter), of length (typically 700 mm) appropriate to the particular furnace used (generally of an approximate length of 400 mm). Preferably, the gas outlet end should be narrowed to a tubule of approximately 7 mm diameter.

NOTE Combustion tubes of alternative refractory compositions do not have adequate thermal stress characteristics for operation with this method.

b) **Silicone stoppers**, 20 mm diameter, positioned at inlet end (and outlet, if applicable) of the silica tube described in a).

c) **Combustion boats**, made of high-alumina unglazed porcelain (approximately 97 mm × 16 mm × 12 mm), preignited at 1 000 °C for 1 h.

d) **Silica pusher and T-tube**, comprising a silica push rod (5 mm diameter and 500 mm long) fused at one end to provide a flat disc surface of 10 mm to 12 mm diameter, and having a piece of magnetic steel affixed to the other end by epoxy resin. The T-tube (500 mm long) is composed of borosilicate glass and protrudes 10 mm into the silica tube a) through a stopper b). The stem of the T-tube is connected to the steam generator by way of the three-way stopcock [5.6.1 e)]. A magnet is used to move the pusher inside the T-tube.

e) **Combustion furnace**, capable of reaching a temperature of at least 1 200 °C.

5.6.3 Absorption vessel: Dreschel gas washing bottle or equivalent, of capacity 125 ml, with gas-distribution tube fitted with a sintered glass disc of 15 µm to 40 µm pore size [5.6.1 d)] bent at 90°, approximately 60 mm from the top.

5.6.4 Water jacket, suitable for containing the absorption vessel.