TECHNICAL SPECIFICATION

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Solid mineral fuels — Determination of Chlorine content

Combustibles minéraux solides — Dosage de la teneur en chlore

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

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

Solid mineral fuels — Determination of Chlorine content

1 Scope

This Technical Specification specifies two methods (high temperature combustion and bomb combustion) for the determination of chlorine in solid mineral fuels. It is applicable to hard coals, brown coals, coke, and tailings.

The chlorine in the digestion solution can be determined using different methods, e.g. an ion-selective electrode, coulometric or potentiometric titration, spectrophotometry, or ion chromatography. In this Technical Specification, the ion chromatographic separation with conductivity detection is described.

The method is applicable to determine the chlorine content higher than 0,005 per cent mass fraction.

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 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

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

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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

EN ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 apply.

4 Principle

4.1 Method A — High temperature combustion in oxygen

The sample is combusted at high temperature in an oxygen atmosphere. The gaseous combustion products including the formed chloride are collected in a trap filled with water, in which they are dissolved.

4.2 Method B — Bomb combustion

The sample is combusted in a high pressure bomb in an oxygen atmosphere. The formed chloride is collected in an absorption solution inside the bomb.

5 Reagent

5.1 Method A

5.1.1 Oxygen, pure, with an assay of at least 99,5 % volume fraction.

5.1.2 Combustion aid (optional), spectroscopic carbon or iron phosphate ($FePO_4$).

5.2 Method B

5.2.1 Oxygen, pure, with an assay of at least 99,5 % volume fraction.

5.2.2 Combustion aid (optional), paraffin, benzoic acid, polyethylene combustion bags, acetobutyrate capsules, or other suitable materials.

5.2.3 Fuse, ignition wire (e.g. platinum) and cotton fuse (optional).

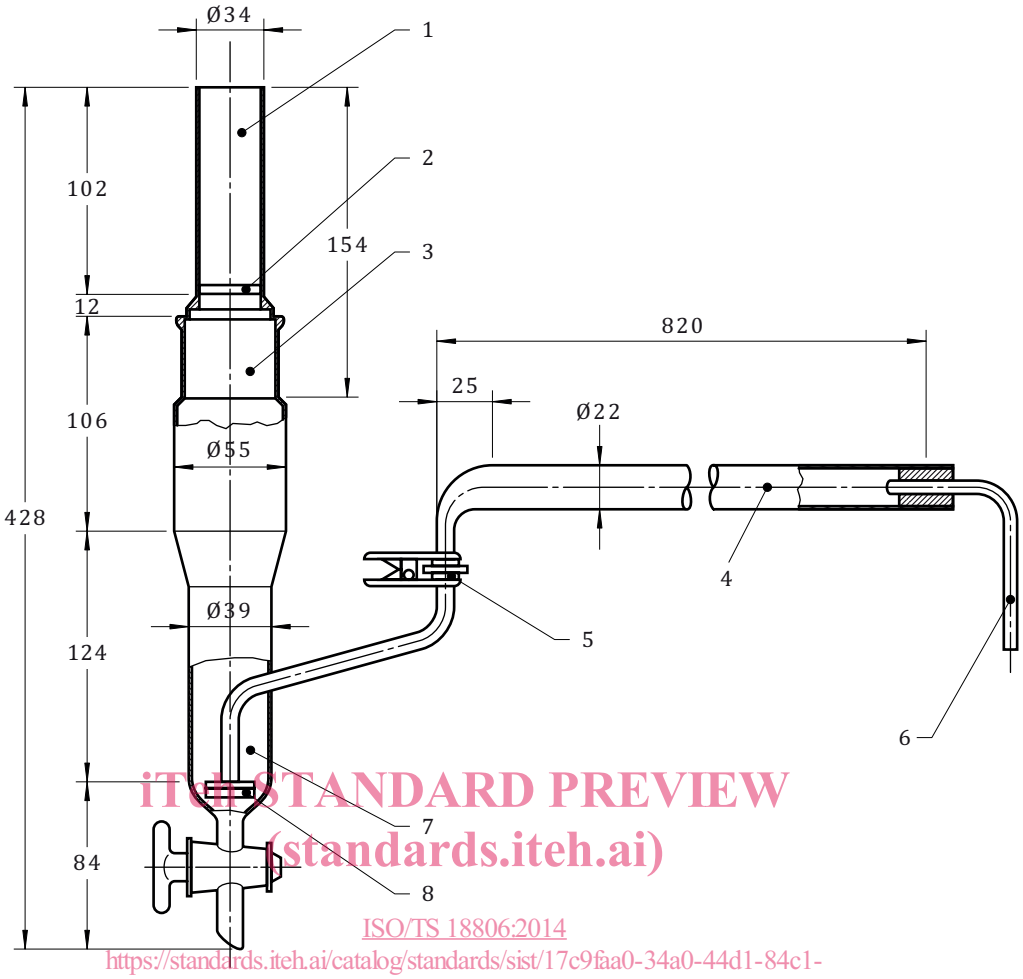
Note A possible contribution of the cotton fuse to the chlorine content should be considered.

5.2.4 Absorption solution, either eluent used for ion chromatographic determination, or alkaline solution (e.g. 0,2 mol/l KOH or 0,1 mol/l NaOH), or deionised water.

6 Apparatus

6.1 Method A

6.1.1 Fused silica combustion tube, absorber, and headpiece, the sintered glass discs in the absorber and the headpiece shall have a pore size of 90 µm to 150 µm (see Figure 1).



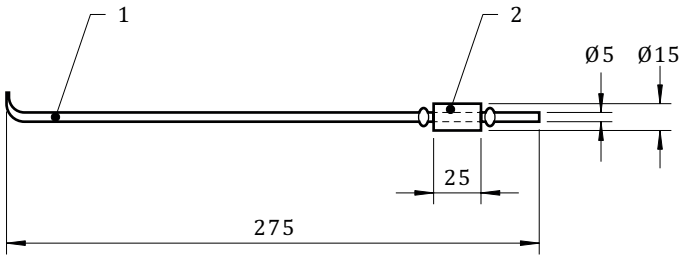
Key

1 headpiece
2 sintered glass disc
3 V45/50 conical ground joint (ISO 383)
4 combustion tube
5 VS 13 spherical joint (ISO 641)
6 oxygen inlet
7 absorber
8 sintered glass disc

Figure 1 — Apparatus for Method A

6.1.2 Porcelain combustion boat, with handle, e.g. 70 mm long, 10 mm wide, and 7 mm deep.

6.1.3 Silica pusher, with iron inlay (see Figure 2).



Key

1 silica pusher

2 iron inlay

Figure 2 — Silica pusher

6.1.4 Magnet.

6.1.5 Electrical tube furnace, about 300 mm long, capable of being heated to at least 1 300 °C and maintained at (1 250 ± 25) °C.

6.1.6 Flow meter.

6.1.7 Oxygen inlet, consisting of a pierced silicon stopper with a glass tube.

6.2 Method B

6.2.1 Digestion unit

6.2.1.1 Digestion bomb, with inner surface that is resistant to corrosion by acidic gases formed or emitted during combustion.

6.2.1.2 Pressure regulator, to control the filling of the bomb with oxygen and discharging afterwards.

6.2.1.3 Pressure gauge, with relieve valve operating at 3,5 MPa.

6.2.1.4 Ignition circuit.

If appropriate, the equipment for determination of calorific value according to ISO 1928 can be used simultaneously for chlorine determination. Attention is drawn to the fact that deionised water should be used as absorption solution and that 10 ml instead of 1 ml (as in ISO 1928) are used.

6.2.2 Crucible, of silica, nickel-chromium, platinum, or similar non-reactive material that is resistant to corrosion by acidic gases formed or emitted during combustion.

7 Preparation of sample

The coal or coke used for the determination of the chlorine content is the general analysis test sample ground to pass a sieve of 212 µm aperture. Expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere. Before commencing the determination, mix the air-dried sample.

After weighing the test portion (see 8.2.2 or 8.3.2), determine the moisture content using a further portion of the test sample by the method described in ISO 687, ISO 5068-2, or ISO 11722, as appropriate.

8 Procedure

8.1 Blank Value Determination

Blank values shall be measured by performing a complete analysis (method A or method B, whatever used for analysis of samples) without sample but including combustion aid, if used. Blank values should be measured on a daily basis or when series of analyses are started.

8.2 Method A

8.2.1 Preparation

Adjust the tube furnace to ensure a temperature of (1 250 ± 25) °C in the combustion zone. Place the combustion tube in the furnace so that its projecting vertical section with the spherical ground joint is as near as possible to the hottest zone to prevent any condensation of combustion products.

Adjust the oxygen flow to (1 ± 0,1) l/min.

Mix samples having more than 30 % mass fraction ash with 0,1 g to 0,2 g of combustion aid to achieve uniform combustion.

8.2.2 Combustion procedure

Weigh, to an accuracy of 0,1 mg, about 50 mg to 1 000 mg (depending on the chlorine concentration) of the sample prepared according to Clause 7 into a combustion boat. Push the boat into the combustion tube using the silica pusher until the tube can be closed with the oxygen inlet.

NOTE Carefully place the front end of the pusher into the handle of the combustion boat. Otherwise problems to remove the boat after test might occur.

Fill the absorber with 150 ml deionised water. After starting the oxygen flow, mount the headpiece on the absorber and fill it with 20 ml deionised water.

To prevent deflagration, initially use the magnet to move the pusher together with the combustion boat containing the sample into the combustion zone so far only, that the front section of the sample is ignited only. After the sample has reached red heat (approximately one minute after ignition), push it into the combustion zone to burn it completely. High temperature coke samples can be pushed into the combustion zone directly.

Combustion time is depending on the ash of the sample. It is about 20 mins for coals having 10 % to 20 % mass fraction ash. After combustion has been terminated, transfer the absorption liquid from headpiece and absorber into a 250 ml volumetric flask. Rinse headpiece and absorber with deionised water and add to the flask. Fill up to the mark with deionised water (V_D).

8.3 Method B — Bomb Combustion

8.3.1 Preparation

Bomb parts shall be inspected regularly for wear and corrosion; particular attention shall be paid to the condition of the threads of the main closure. Manufacturers' instructions and any local regulations regarding the safe handling and use of the bomb shall be observed. Before starting a test ensure that no gas leakage occurs.

NOTE Gas leakage can be checked easily by complete immersing the pressurized bomb in cold water.