
**Solid mineral fuels — Determination
of chlorine content**

Combustibles minéraux solides — Dosage de la teneur en chlore

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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).

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

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 first edition cancels and replaces ISO/TS 18806:2014, which has been technically revised.

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 chlorine content

1 Scope

This document specifies two methods (high temperature combustion and high pressure vessel combustion) for the determination of chlorine in solid mineral fuels. It is applicable to solid mineral fuels.

The chlorine in the absorption solution can be determined using different finishes, such as coulometric or potentiometric titration, spectrophotometry or ion chromatography.

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

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

3 Terms and definitions

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

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

4.1 Method A — High temperature combustion

The sample is combusted in an oxygen atmosphere at high temperature. The gaseous combustion products collected in a trap filled with water include dissolved chloride compounds.

4.2 Method B — High pressure vessel combustion

The sample is combusted in an oxygen atmosphere in a high pressure vessel containing an absorption solution. The combustion products collected in an absorption solution include dissolved chloride compounds.

5 Reagents

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)**, e.g. spectroscopic carbon, iron phosphate (FePO_4), tungsten or quartz.

5.1.3 **Deionised water**, with a specific conductivity not higher than 0,2 mS/m at 25 °C.

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 Check for a possible contribution of the cotton fuse to the chlorine content.

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 ([5.1.3](#)).

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6 Apparatus

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6.1 Method A

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An example for the equipment for high-temperature combustion is given below. This equipment is suitable for manual determinations and is well established.

Automated systems with autosamplers can also be used if their main features comply with the manual apparatus specified below, i.e. mainly temperature of the oven, the use of fused silica glass tubes, and the absorption unit.

6.1.1 **Fused silica combustion tube, absorber and headpiece** (see [Figure 1](#)).

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](#)).

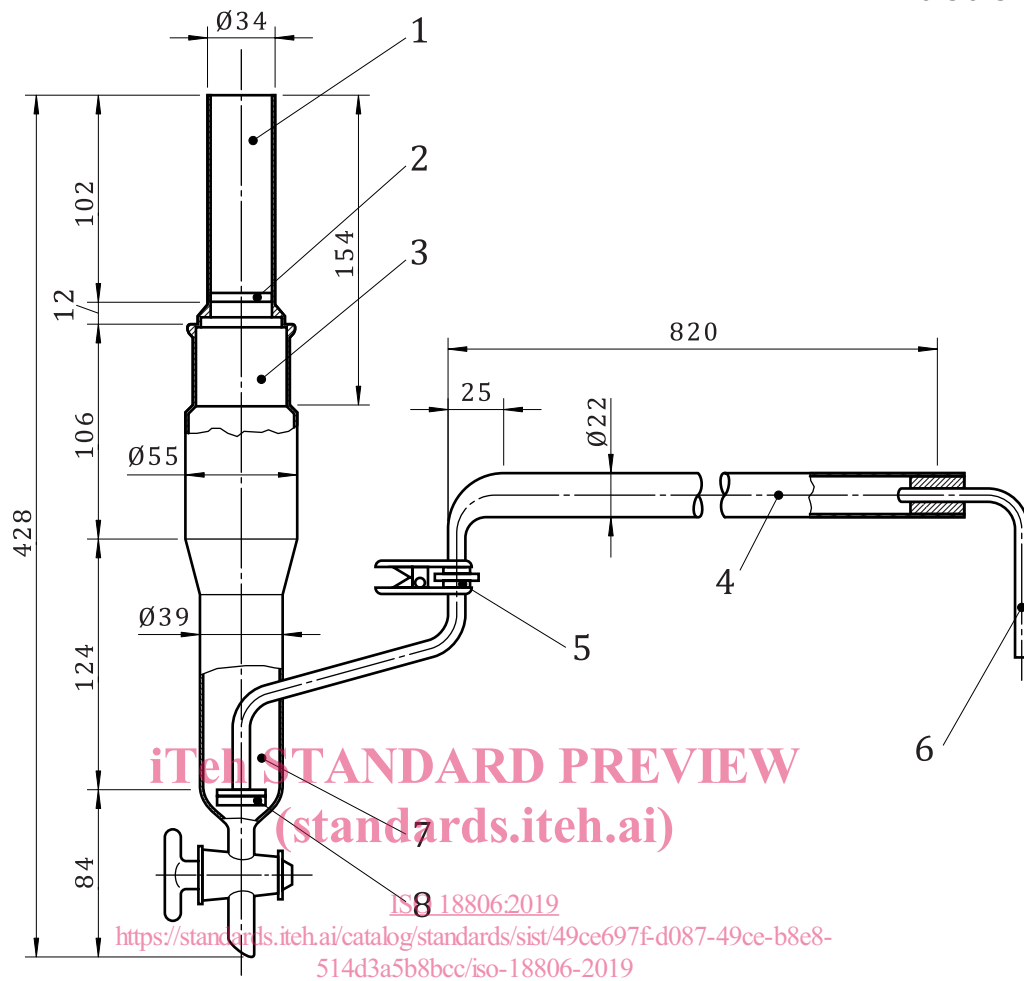
6.1.4 **Magnet**.

6.1.5 **Electrical tube furnace**, about 300 mm long, capable of being heated to preferably 1 300 °C and maintained at the combustion temperature of $(1\,250 \pm 25)$ °C.

6.1.6 **Flow meter**.

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

Dimensions in millimetres



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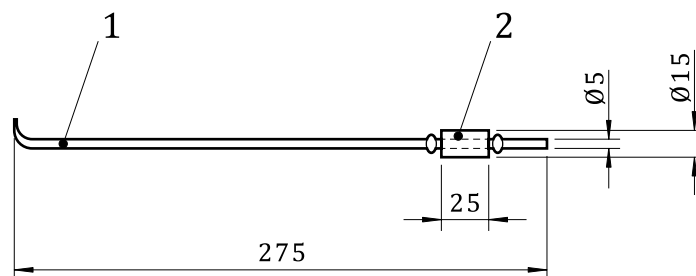
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Key

- | | |
|--|--|
| 1 headpiece | 5 VS 13 spherical joint (see ISO 641) |
| 2 sintered glass disc (pore size of 90 µm to 150 µm) | 6 oxygen inlet |
| 3 V45/50 conical ground joint (see ISO 383) | 7 absorber |
| 4 combustion tube | 8 sintered glass disc (pore size of 90 µm to 150 µm) |

Figure 1 — Example of an apparatus for Method A

Dimensions in millimetres



Key

- | |
|-----------------|
| 1 silica pusher |
| 2 iron inlay |

Figure 2 — Example of a silica pusher

6.2 Method B

6.2.1 Digestion unit.

6.2.1.1 Digestion vessel, capable of withstanding safely the pressures developed during combustion and with an 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 vessel with oxygen and discharging afterwards.

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

6.2.1.4 Ignition circuit.

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.

6.2.3 Alternate digestion equipment.

The apparatus employed for determination of calorific value in accordance with ISO 1928 can be used simultaneously for chlorine determination provided it meets the specifications of [6.2.1](#). Attention is drawn to the fact that deionised water should be used as absorption solution.

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7 Test sample

The sample 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 separate portion of the equilibrated test sample by the method described in ISO 687, ISO 5068-2 or ISO 11722, as appropriate.

8 Procedure

8.1 Blank value determination

Measure blank values using the same method A or B employed for the analysis sample. Perform a complete analysis without the analysis sample. Include combustion aid, if used. Measure blank values regularly, e.g. on a daily basis or when series of analyses are started.

8.2 Method A — High temperature combustion

8.2.1 General

The following subclauses specify the manual procedure.

When using automated systems, the temperature may be set lower than specified below. This is permitted only if the use of this lower temperature will give identical results compared to $(1\,250 \pm 25)^\circ\text{C}$, which shall be verified using certified reference materials. If appropriate, moistening of the oxygen flow is allowed.

8.2.2 Preparation

Adjust the tube furnace to establish a temperature of $(1\ 250 \pm 25)$ °C in the combustion zone. To prevent any condensation of combustion products, 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.

Adjust the oxygen flow to $(1 \pm 0,1)$ 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.

8.2.3 Combustion procedure

Depending on the chlorine content, weigh, to an accuracy of 0,1 mg, about 50 mg to 1 000 mg of the sample prepared in accordance with [Clause 7](#) into a combustion boat. Push the boat into the combustion tube using the silica pusher ([6.1.3](#)) until the tube can be closed with the oxygen inlet ([6.1.7](#)).

Carefully place the front end of the pusher into the handle of the combustion boat. Otherwise problems with removing the boat after the test can 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.

Use the magnet ([6.1.4](#)) to initially move the pusher and the combustion boat containing the sample into the combustion zone so that only the front portion of the sample is ignited. Once the sample has reached red heat (approximately one minute after ignition), push the sample into the combustion zone to burn it completely. High temperature coke samples can be pushed into the combustion zone directly.

Combustion time depends on the mineral content of the sample. It can be about 20 min for coals having 10 % to 20 % mass fraction ash. After combustion has been terminated, transfer the absorption liquid from the headpiece and absorber into a 250-ml volumetric flask. Rinse the headpiece and absorber with deionised water and add the rinsings to the flask. Fill up to the mark with deionised water, V_D .

8.3 Method B — High pressure vessel combustion

8.3.1 Preparation

Regularly inspect vessel parts for wear and corrosion. Pay particular attention to the condition of the threads of the main closure. Observe the manufacturers' instructions and any local regulations regarding the safe handling and use of the vessel. Before starting a test, ensure that no gas leakage occurs.

NOTE Gas leakage can be checked easily by completely immersing the pressurized vessel in cold water.

8.3.2 Combustion procedure

Depending on the chlorine content, weigh, to an accuracy of 0,1 mg, about 50 mg to 1 000 mg of the sample prepared in accordance with [Clause 7](#) into a crucible.

If appropriate, chlorine can be determined simultaneously with the calorific value in accordance with ISO 1928 (for details, see [6.2.3](#)).

Mix samples that are not easily combustible (e.g. having more than 30 % mass fraction ash) with 0,1 g to 0,2 g of combustion aid ([5.2.2](#)) to achieve complete combustion. Transfer 5 ml to 10 ml of the absorption solution ([5.2.4](#)) into the vessel. Rotate the vessel to wet the inner surface.

When the chlorine determination is performed simultaneously with the calorific value determination in accordance with ISO 1928, the volume of the absorption solution may be reduced to 1 ml. Complete absorption of the chlorine present in the coal sample shall be verified using certified reference materials.