INTERNATIONAL STANDARD

ISO 5071-1

Third edition
2021-12

Brown coals and lignites — Determination of the volatile matter in the analysis sample —

Part 1: Two-furnace method

Charbons bruns et lignites — Détermination des matières volatiles dans l'échantillon pour analyse —

Partie 1: Méthode avec utilisation de deux fours

Reference number
ISO 5071-1:2021(E)

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Published in Switzerland

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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, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 5071-1:2013), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— referenced documents have been updated;

— terms and definitions have been added;

— sample has been added;

— calculation and expression of results have been amended;

— precision has been amended;

— test report has been amended.

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.

Introduction

The volatile matter is determined as the loss in mass, corrected for moisture, when an analysis sample of brown coal or lignite is heated out of contact with air under specified conditions. The test is empirical and, in order to obtain reproducible results, it is essential that the rate of heating, the final temperature and the overall duration of the test be carefully controlled. Due to the nature of brown coals and lignites, initial heating of the sample at 400 °C is necessary to minimize the possibility of ejection of sample from the test crucible.

Mineral matter associated with the sample may also lose mass under the conditions of the test, the magnitude of the loss being dependent on both the nature and the quantity of the minerals present.

Brown coals and lignites — Determination of the volatile matter in the analysis sample —

Part 1: Two-furnace method

1 Scope

This document specifies a method of determining the volatile matter of brown coals and lignites.

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

ISO 18283, *Coal and coke — Manual sampling*

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 https://www.electropedia.org/

4 Principle

The coal is heated out of contact with air for 7 min at 400 °C, then immediately transferred to another furnace and heated at 900 °C for a further 7 min. The volatile matter mass fraction is calculated from the loss in mass of the oven-dried sample or from the loss in mass of the analysis sample corrected for moisture.

5 Reagents

5.1 Desiccants, fresh or freshly regenerated and preferably self-indicating. Suitable desiccants are magnesium perchlorate, silica gel, activated alumina and anhydrous calcium sulfate.

WARNING — Magnesium perchlorate is a strong oxidizing agent. Do not attempt to regenerate the absorbent. Do not permit contact with organic materials or reducing agent.

5.2 Nitrogen, dry, with a maximum oxygen content of 30 µl/l.

6 Apparatus

6.1 Furnace (Figure 1), heated by electricity. Two such furnaces are required. One furnace shall have a zone of 160 mm × 100 mm maintained at a uniform temperature of 400 °C ± 10 °C. The second shall have a zone of 160 mm × 100 mm maintained at a uniform temperature of 900 °C ± 5 °C. The furnaces may be the stop-ended type or fitted at the back with a flue approximately 25 mm in diameter by 150 mm long.

The heat capacity of the 900 °C and/or 400 °C furnace shall be such that, with an initial temperature of 900 °C and/or 400 °C, a temperature of 900 °C ± 10 °C and/or 400 °C ± 10 °C is regained within 4 min after insertion of a cold stand and its crucibles. The temperature shall be measured with a thermocouple as described in 6.3. The furnace can be designed specifically either for multiple determinations using a number of crucibles in one stand or for receiving one crucible and its stand. A position for the crucible stand shall be chosen within the zone of uniform temperature and this position used for all determinations.

6.2 Oven, capable of being controlled at a temperature within the range 105 °C to 110 °C and with provision for passing a current of dry, oxygen-free nitrogen through it at a rate sufficient to change the atmosphere 15 times per hour. The size of the chamber is suitable for containing the crucible (6.4).

6.3 Thermocouple, unsheathed, of wire not thicker than 1 mm, used to check the temperature characteristics of the furnace. The thermo-junction shall be inserted midway between the base of the crucible in its stand and the floor of the furnace. If the stand holds more than one crucible, the temperature under each crucible shall be checked in the same manner. If desired, a sheathed thermocouple may be permanently installed in the furnace with its thermo-junction as close as possible to the centre of the zone of uniform temperature; in this case, its temperature readings shall be correlated at frequent intervals with those of the unsheathed thermocouple, which is then inserted only when necessary.

NOTE The temperature/electromotive force relationship of a thermo-junction maintained at elevated temperatures gradually changes with time.

6.4 Crucible and lid, a cylindrical crucible with a well fitting lid, both made of fused silica. The crucible and lid shall weigh between 10 g and 14 g and have dimensions approximating those shown in Figure 2. The fit of the lid on the crucible is critical to the determination and a lid shall be selected to match the crucible so that the horizontal clearance between them is not greater than 0,5 mm. After selection, the crucible and the lid shall be ground together to give smooth surfaces and then be given a common distinguishing mark. Crucibles of other refractory materials, or of platinum, can be used, provided that they give results which agree with the recommended silica crucible, within the stated precision of the method (see Clause 10).

6.5 Stand, on which the crucible is placed in the furnace (6.1), so that the appropriate specified rate of heating can be achieved. For example, it may consist of the following:

a) for a single determination, a ring of heat-resistant steel wire as shown in Figure 3 a), 27 mm in diameter and 1,5 mm thick, resting on the inner projection of its legs,

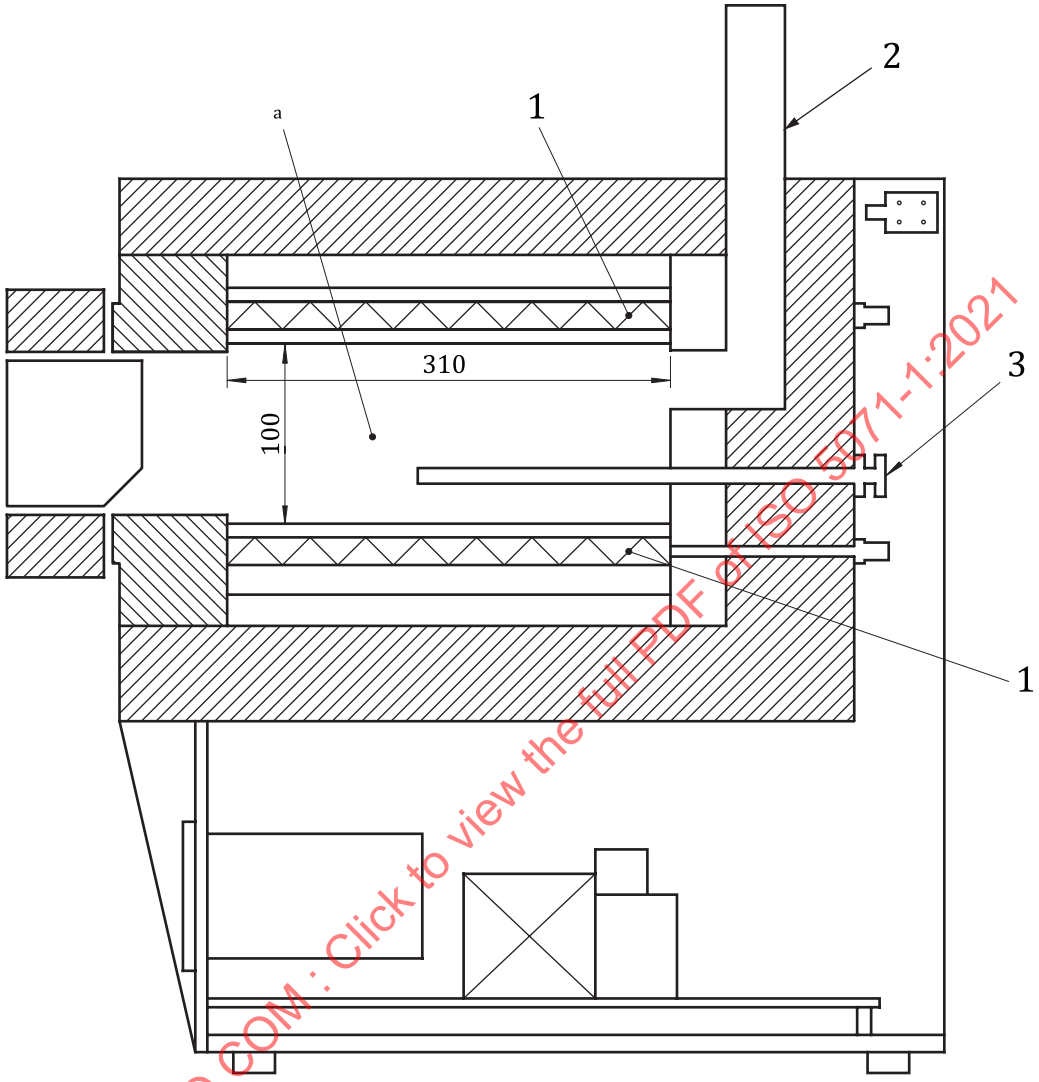
or

b) for multiple determinations, a tray of heat-resistant steel wire as shown in Figure 3 b), of appropriate size, with ceramic plates 2 mm thick supporting the crucibles.

6.6 Balance, with a resolution of 0,1 mg.

6.7 Desiccator.

Dimensions in millimetres



Key

1 heating system
2 flue
3 thermocouple
[a] Chamber width is 200 mm.

Figure 1 — Example of a suitable furnace

Dimensions in millimetres

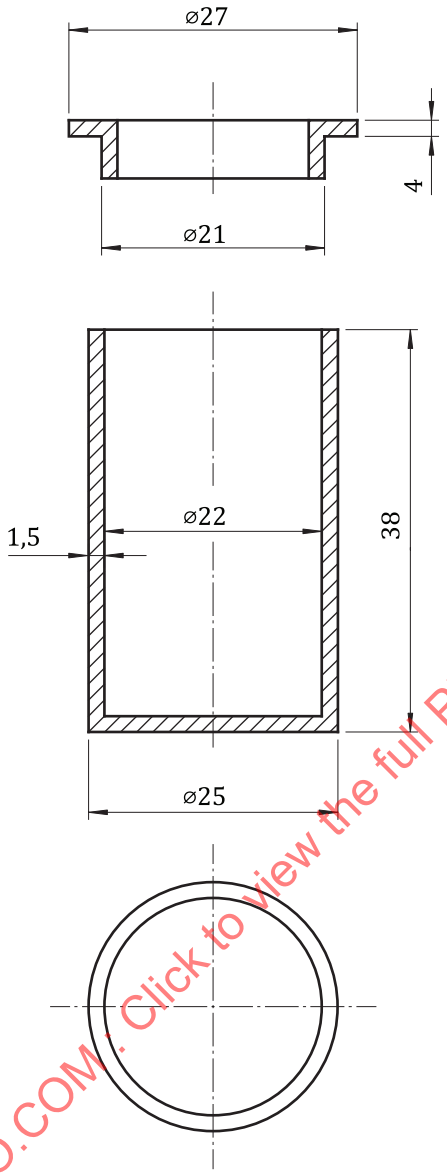
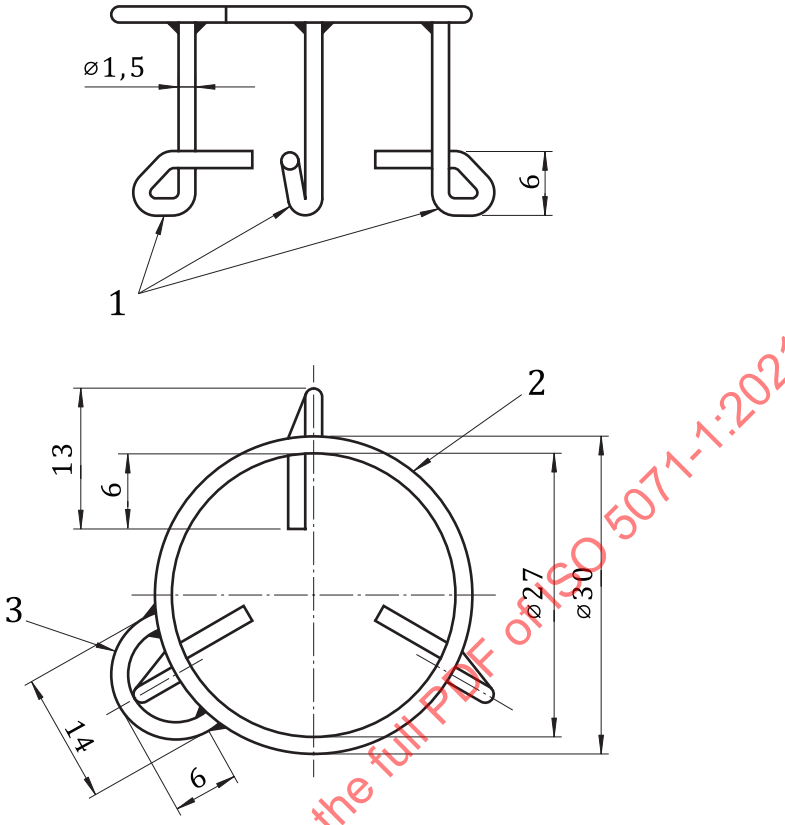
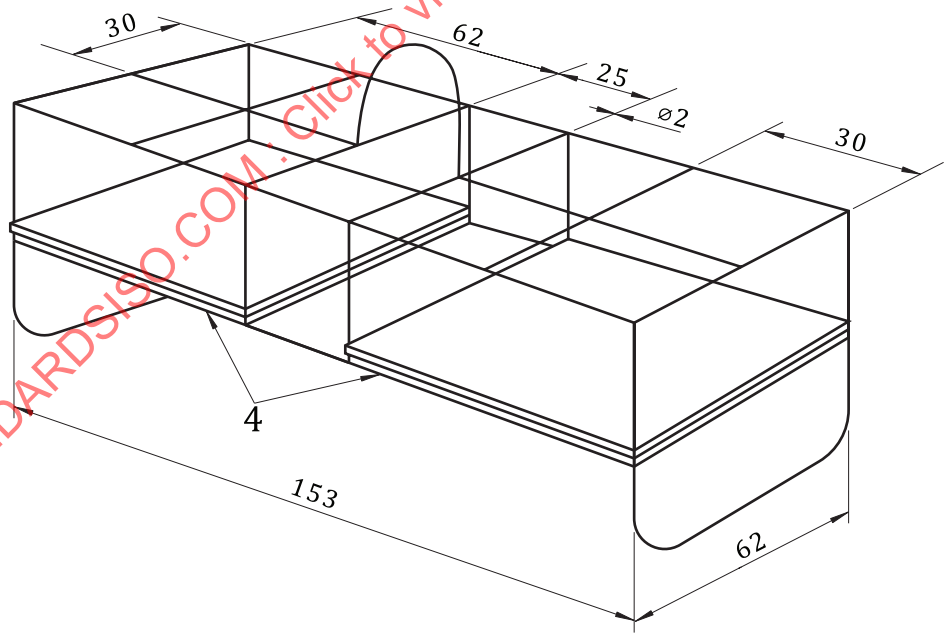


Figure 2 — Silica crucible and lid

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determinations

Key

1	three legs spaced 120° apart	3	handle
2	ring	4	ceramic plates

Figure 3 — Crucible stands

7 Sample

The sample shall be the general analysis test sample, prepared to a nominal top size of 212 µm by the preparation procedures specified in ISO 13909-4 or ISO 18283.

The sample should be brought in moisture equilibrium with the laboratory atmosphere by exposure in a thin layer on a tray. Exposure time shall be kept to a minimum.

The sample shall be thoroughly mixed immediately before analysis, preferably by mechanical means.

Duplicate determinations of moisture from the same test sample shall be conducted concurrently with the determination of volatile matter by the method specified in ISO 5068-2.

8 Procedure

8.1 Preliminary procedure

Adjust the temperature of the zone in the furnaces (6.1), loaded with a stand and empty crucibles where appropriate, to 400 °C ± 10 °C and 900 °C ± 5 °C respectively, as indicated by the correctly unsheathed thermocouple (6.3). Remove the stand and crucibles and close the doors of the furnace. Heat, in a furnace (6.1) at 900 °C for 7 min, either one crucible and lid (6.4) or the requisite number of crucibles and lids to fill the multiple stand. Remove the crucible(s) from the furnace and cool them, first on a metal slab and finally in a desiccator (6.7) located next to the balance. As soon as it is cool to ambient temperature, determine the mass of each empty crucible and lid. Record the mass(es) of the preconditioned crucibles and lids to the nearest 0,1 mg (8.4, Note 1).

8.2 Predetermination procedure

Accurately transfer a 1,00 g to 1,01 g mass of sample (see Clause 7) to the nearest 0,1 mg into each preconditioned crucible. Replace the lid and tap the crucible on a clean hard surface until the sample forms a layer of even thickness on the bottom of the crucible. Proceed in accordance with either 8.3 or 8.4 depending on whether or not the coal has been oven-dried or air-dried.

8.3 Method using oven-dried coal

Place the charged crucible(s), with lid(s) removed, in the oven (6.2) at 105 °C to 110 °C. Dry the sample(s) to constant mass in accordance with the procedure for moisture determination specified in ISO 5068-2. Record the mass(es) of the oven-dried crucible(s), lid(s) and sample(s) to the nearest 0,1 mg.

Place the crucible(s) fitted with lid(s) containing the oven-dried sample(s) in a suitable stand (8.4, NOTE 2) and transfer to the furnace set at 400 °C for 7 min. Immediately transfer to the furnace set at 900 °C for a further 7 min. A temperature of 400 °C ± 10 °C or 900 °C ± 10 °C is regained within 4 min after insertion of a cold stand and its crucibles.

Remove the crucible(s) and cool and determine their mass in the same manner as for empty crucible(s) (see 8.1). If ash deposits are visible on the stand or crucible(s) lid(s) then discard the test and repeat (8.4, NOTE 3).

8.4 Method using air-dried coal

Place the charged crucible(s) with the lid(s) in a suitable cool stand (see NOTE 2) and transfer to the furnace (6.1) set at 400 °C for 7 min. Immediately transfer to the furnace (6.1) set at 900 °C for a further 7 min. A temperature of 400 °C ± 10 °C or 900 °C ± 10 °C is regained within 4 min after insertion of