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Hard coal and coke — Determination of volatile matter

Houille et coke — Détermination des matières volatiles

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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

This fourth edition cancels and replaces the third edition (ISO 562:2010), which has been technically revised.

The main changes are as follows:

— title and references changed to be consistent with the new name of ISO/TC 27;

— editorial updates to be in line with ISO 80000-1.

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

In this document, volatile matter is determined as the loss in mass, less that due to moisture, when coal or coke is heated out of contact with air under standardized conditions. The test is empirical and, in order to ensure reproducible results, it is essential that the conditions specified in this document are strictly followed. The moisture of the sample is determined at the same time as the volatile matter so that the appropriate correction can be made.

Mineral matter associated with the sample can also lose mass under the conditions of the test specified in this document. The magnitude of the loss is dependent on both the nature and the quantity of the minerals present.

The apparatus and procedure are specified so that one or more determinations can be performed simultaneously in the furnace.

Hard coal and coke — Determination of volatile matter

1 Scope

This document specifies a method of determining the volatile matter of hard coal and of coke. It is not applicable to 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 687, *Coke — Determination of moisture in the general analysis test sample*

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

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 terminology 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

A portion of the sample is heated out of contact with air at 900 °C for 7 min. The mass fraction of volatile matter, expressed as a percentage, is calculated from the loss in mass of the test portion after deducting the loss in mass due to moisture.

5 Reagents and materials

5.1 Cyclohexane, of recognized analytical grade.

6 Apparatus

6.1 Furnace, heated by electricity, in which a zone of uniform temperature of 900 °C ± 5 °C can be maintained.

The furnace may be of the stop-ended type or fitted at the back with a flue approximately 25 mm in diameter and 150 mm long (see Figure 1).

For furnaces with flues, the furnace door shall seal well. The flue should not reach far out of the oven and should be fitted with a butterfly valve to restrict airflow through the furnace.

The heat capacity of the furnace shall be such that, with an initial temperature of 900 °C, the temperature is recovered within 4 min after insertion of a cold stand and its crucibles. The temperature is measured with a thermocouple (6.2).

Normally, the furnace is designed specifically either for multiple determinations using a number of crucibles in one stand or for receiving one crucible and its stand. In the first case, the (rectangular) zone of uniform temperature shall be at least 160 mm × 100 mm; in the latter case, a (circular) zone with a diameter of 40 mm is sufficient.

A position for the crucible stand shall be chosen within the zone of uniform temperature and this position shall be used for all determinations. The temperature of 900 °C shall be attained as closely as possible with a tolerance of ±5 °C in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

6.2 Thermocouple, unsheathed, of wire no thicker than 1 mm.

When inserted through the front or rear of the furnace, the thermocouple should be long enough to reach the centre of the underside of each crucible when placed in the zone of uniform temperature. The thermojunction shall be placed 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.

A sheathed thermocouple may be permanently installed in the furnace (see Figure 1) with its thermojunction as close as possible to the centre of the zone of uniform temperature. In this case, furnace temperature readings shall be correlated at frequent intervals with those of the unsheathed thermocouple, which is thus inserted only when necessary. The thermocouple readings should be checked at regular intervals, e.g. on a monthly basis.

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

6.3 Crucible, cylindrical, with a well-fitting lid, both of fused silica.

The crucible with lid shall have a mass between 10 g and 14 g and dimensions approximating those shown in Figure 2. The fit of the lid on the crucible is critical to the determination. A lid shall be selected to match the crucible so that the horizontal clearance between them is no greater than 0,5 mm. The selected crucible and lid shall be ground together to give smooth surfaces and then be given a common distinguishing mark.

When performing multiple determinations on highly swelling coals, taller crucibles may be used. These may be up to 45 mm in height without affecting the determined volatile matter, provided that the rate of temperature recovery specified in 6.1 be maintained.

6.4 Crucible stand, on which the crucible is placed in the furnace, such that the appropriate rate of heating can be achieved.

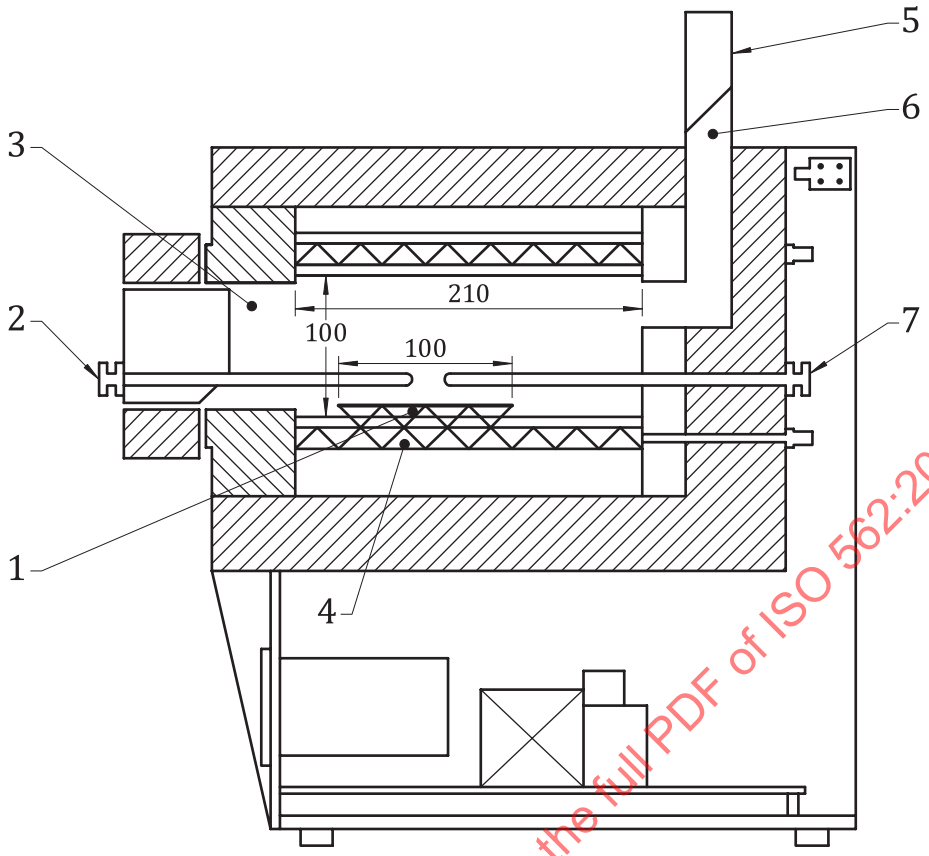
For example, it may consist of the following:

a) for single determinations, a ring of heat-resistant steel wire with ceramic discs, 25 mm in diameter and 2 mm thick, resting on the inner projection of its legs [see Figure 3 a)];

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

6.5 Analytical balance, capable of reading to the nearest 0,1 mg.

Dimensions in millimetres



Key

1 zone of uniform temperature
2 check thermocouple
3 chamber (width 200 mm)
4 heating system
5 flue
6 valve
7 thermocouple

Figure 1 — Example of a suitable furnace

Dimensions in millimetres

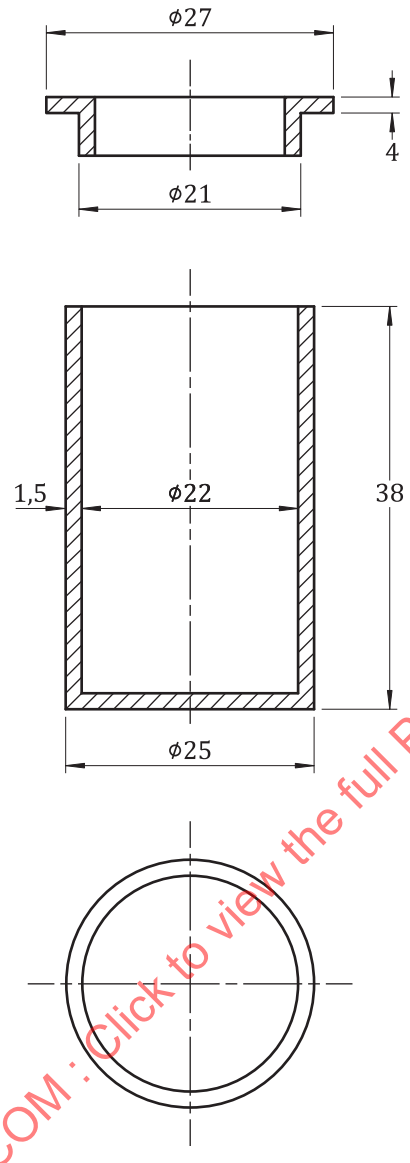
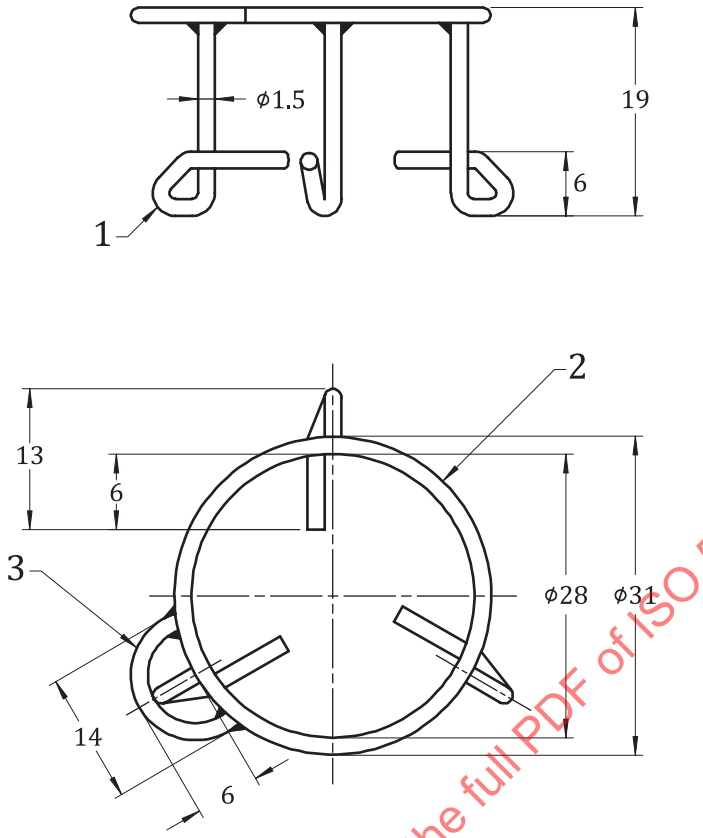
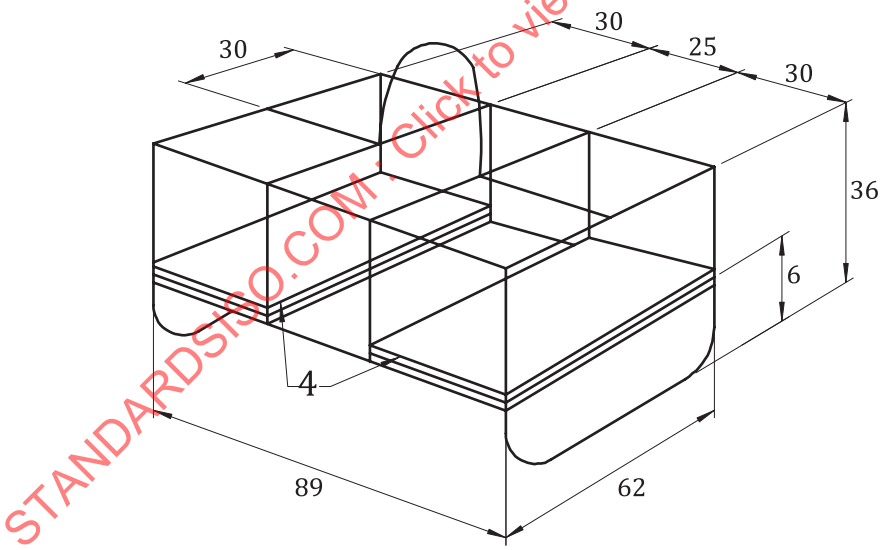


Figure 2 — Example of a silica crucible and lid

Dimensions in millimetres



a) Suitable for a single determination (figure shown without ceramic disc)



b) Suitable for multiple determinations

Key

1	three legs separated by 120°	3	handle
2	ring	4	ceramic plates

Figure 3 — Examples of crucible stands

7 Preparation of the test sample

The sample shall be the general analysis test sample (see ISO 1213-2), prepared to a nominal top size of 212 µm. Sample preparation procedures are specified in ISO 13909-4, ISO 13909-6 or ISO 18283, whatever applies.

The sample shall be well mixed and in moisture equilibrium with the laboratory atmosphere.

A test portion from the same test sample is separated for the determination of moisture in parallel with the determination of volatile matter.

8 Procedure

8.1 General

The test is empirical and, in order to ensure valid and reproducible results, strictly follow the specification given in this document, mainly the specified recovery time of temperature, the start and final temperature and the overall duration of the test. If these temperature requirements (6.1) are not met, stop the test and readjust the temperature settings of the furnace.

It is also essential to exclude air from the coal or coke during heating to prevent oxidation. The fit of the crucible lid is, therefore, critical. Ensure that the requirements in (6.3) are met.

8.2 Furnace temperature checking

Adjust the temperature of the zone in the furnace (6.1), which contains either a stand with one crucible and lid (Figure 3 a)) or a stand with the requisite number of crucibles and lids (Figure 3 b)), to 900 °C ± 5 °C as indicated by the correctly located thermocouple (6.2). Check that the temperature under each crucible, at the same height, lies within the temperature tolerance of the uniform zone.

The temperature should be checked before the determinations are started. However, in routine operation when several analyses are performed per day, a monthly temperature check is sufficient. Temperature recovery (6.1) should be checked in a similar way.

8.3 Volatile matter determination

Fill either a stand with one empty crucible and lid (Figure 3 a)) or a stand with the requisite number of empty crucibles and lids (Figure 3 b)) and insert in the furnace. Maintain at 900 °C ± 5 °C for 7 min. Remove the stand with the crucible(s) from the furnace and allow to cool to room temperature on a thick metal plate.

Once cool, determine the mass of each empty crucible and lid (6.3) to the nearest 0,1 mg by weighing on an analytical balance (6.5). Add (1 ± 0,1) g of the test sample in an even layer and reweigh to the nearest 0,1 mg. Replace the lid and tap each crucible on a clean hard surface until the test portion forms a layer of even thickness on the bottom of the crucible. If the sample is coke, remove the lid of the charged crucible, add 2 drops to 4 drops of cyclohexane (5.1) and replace the lid.

NOTE 1 The addition of cyclohexane prevents the oxidation of the coke but does not prevent adsorption of gases, e.g. nitrogen.

Place the crucible(s) with lid(s) on top in a cold stand, transfer to the furnace, close the door and leave for 7 min ± 5 s. If multiple determinations are being made, any vacant places on the stand should be filled with empty crucibles.

When the heating period is over, remove the stand with the crucible(s) and lid(s) still in place from the furnace and allow to cool to room temperature.