INTERNATIONAL STANDARD

**ISO 562**

Second edition
1998-02-01

# Hard coal and coke — Determination of volatile matter

*Houille et coke — Détermination des matières volatiles*

## 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.

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.



International Standard ISO 562 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 562:1981), which has been technically revised.



Annex A of this International Standard is for information only.



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## Introduction

The 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 rate of heating, the final temperature and the overall duration of the test are carefully controlled. 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. The moisture content 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 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.



NOTE — When applying this International Standard for classification purposes, to samples obtained directly from coal seams, special care has to be given to the ash content.



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

# Hard coal and coke — Determination of volatile matter

## 1 Scope

This International Standard 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.



ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method*.



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

ISO 11722:—[1)], *Solid mineral fuels — Hard coal — Determination of moisture in the analysis sample by drying in nitrogen*.

## 3 Principle

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

## 4 Reagent

Cyclohexane of recognized analytical grade.

1) To be published.

## 5 Apparatus

**5.1 Furnace,** heated by electricity, in which a zone of uniform temperature of 900 °C ± 5 °C can be maintained. It may be of the stop-ended type or fitted at the back with a flue approximately 25 mm diameter and 150 mm long (see figure 1).

NOTE — It is important for furnaces with flues that the furnace door 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.

Its heat capacity shall be such that, with an initial temperature of 900 °C, the temperature is regained within 4 min after insertion of a cold stand and its crucibles. The temperature is measured with a thermocouple, as described in 5.2.

Normally the furnace will be 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 zone of uniform temperature shall be at least 160 mm × 100 mm; in the latter case, a zone of diameter 40 mm will be 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 specified tolerance of ± 5 °C in order to compensate for inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

**5.2 Thermocouple,** unsheathed, of wire no thicker than 1 mm. It should be long enough to reach the centre of the underside of each crucible when placed in the zone of uniform temperature on being inserted through the front or rear of the furnace. 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.



If desired, 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.



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

**5.3 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 to 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 no 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.

NOTE — When performing multiple determinations on highly swelling coals, it may be necessary to use taller crucibles; these may be up to 45 mm in height without affecting the determined volatile matter, provided that the specified rate of temperature recovery be maintained.

**5.4 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 as shown in figure 3 a) with ceramic discs, 25 mm diameter and 2 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.

**5.5 Balance,** capable of reading to the nearest 0,1 mg.

Dimensions in millimetres

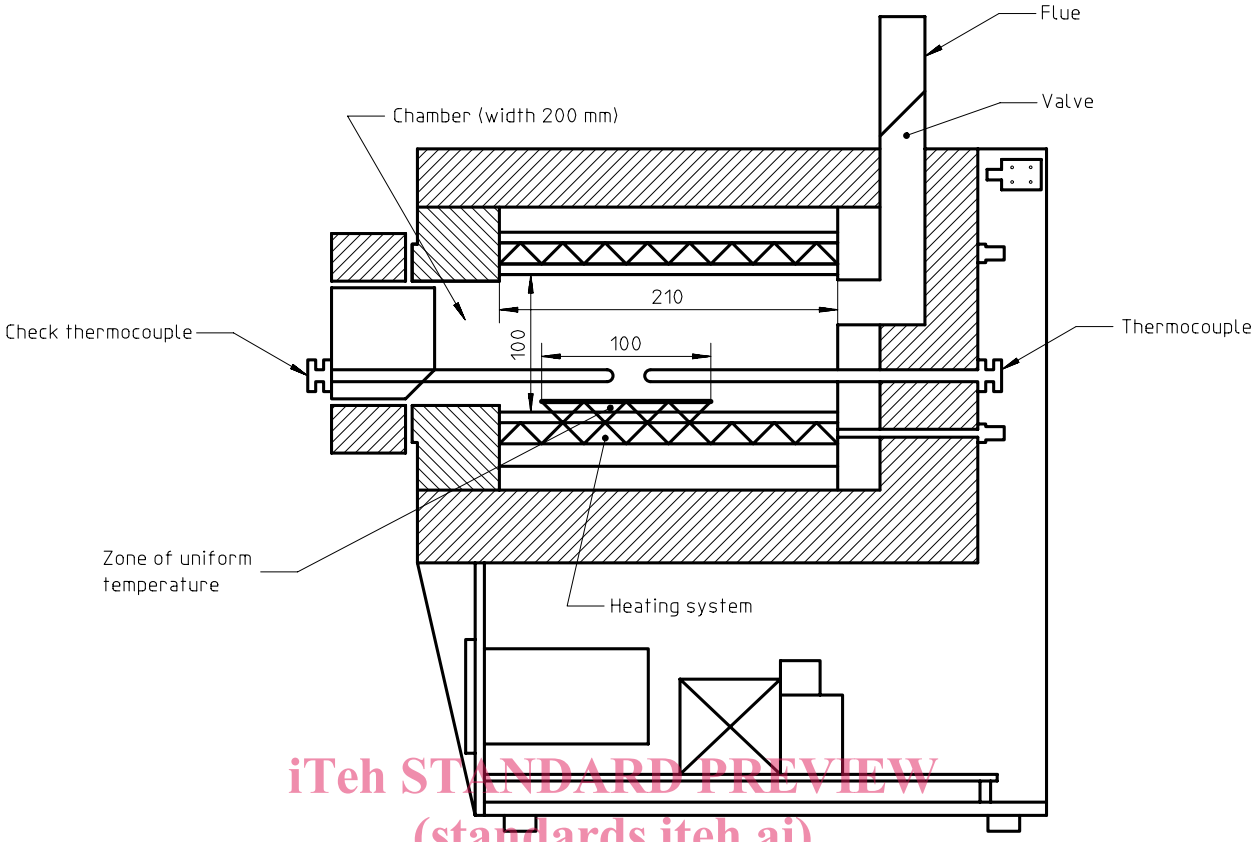





**Figure 1 — Example of suitable furnace**



Dimensions in millimetres

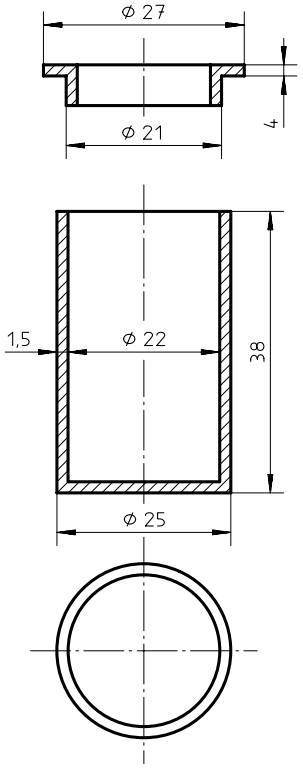
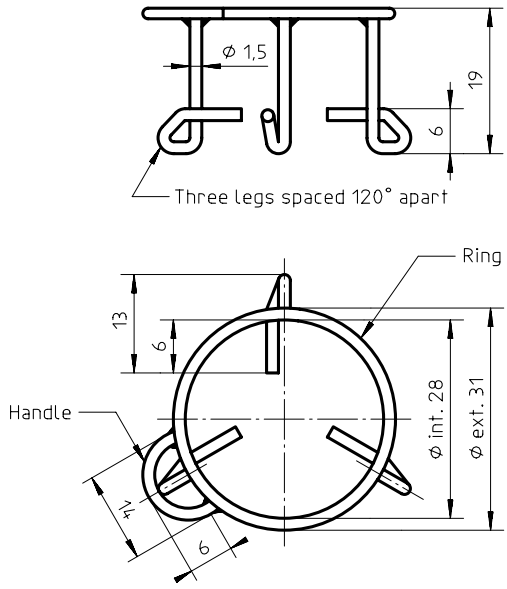


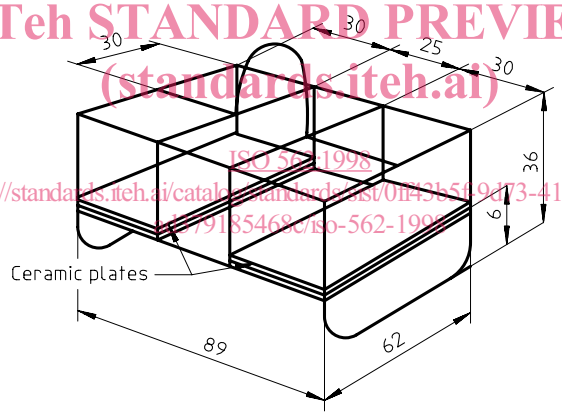

**Figure 2 — Silica crucible and lid**

Dimensions in millimetres




a) Suitable for a single determination






b) Suitable for multiple determinations

**Figure 3 — Crucible stands**

## 6 Preparation of the test sample

The coal or coke used for the determination of volatile matter is the general analysis test sample (ground to pass a sieve of 212 µm aperture).

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 determination of moisture parallel to the determination of volatile matter.

# 7 Procedure

## 7.1 Furnace temperature checking

Adjust the temperature of the zone in the furnace (5.1), containing 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 (5.2). Check that the temperature under each crucible, at the same height, lies within the temperature tolerance of the uniform zone.

NOTE — Temperature checking should be made before starting determinations. However, when several analyses are performed per day, a daily temperature check is sufficient. The check of the temperature recovery criterion (5.1) should be dealt with in a similar way.

## 7.2 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 (fgure 3 b) and insert in the oven. Maintain at 900 °C ± 5 °C for 7 min. Remove the crucible(s) from the furnace and allow to cool to room temperature on a thick metal plate.

As soon as they are cool, weigh each empty crucible and lid and weigh into each crucible, to the nearest 0,1 mg, 1 ± 0,1 g of test sample. 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 of coke, remove the lid of the charged crucible, add 2 to 4 drops of cyclohexane (4) and replace the lid.

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



Place the charged crucible(s) in a cold stand, transfer to the furnace, close the door and leave for 7 min ± 5 s. Remove and allow to cool to room temperature. When cool, weigh the crucible(s) to the nearest 0,1 mg in the same manner as for the empty crucible(s).



NOTES

1 The same treatment of the crucible before and after the determination minimizes the effect of any film of water adsorbed on its surface, while the rapid cooling reduces absorption of moisture by the coal or coke residue.

2 If multiple determinations are being made, any vacant places in the stand should be filled with empty crucibles.

# 8 Expression of results

The volatile matter $V$ in the sample as analysed, expressed as a percentage by mass, is given by the equation:

$$V = \frac{100(m_2 - m_3)}{m_2 - m_1} - M$$

where

$m_1$ is the mass, in grams, of the empty crucible and lid;

$m_2$ is the mass, in grams, of the crucible and lid and test portion before heating;

$m_3$ is the mass, in grams, of the crucible and lid and contents after heating;

$M$ is the moisture, as a percentage by mass, in the sample as analysed, determined according to the method specified in ISO 331 (to be superseded by ISO 11722) or ISO 687.

Report the result, as the mean of duplicate determinations, to the nearest 0,1 % ($m/m$). The results of the determination described in this International Standard are reported on the “air-dried” basis. Calculation of the results to other bases is dealt with in ISO 1170.