

**BS ISO 562:2010**



BSI Standards Publication

# **Hard coal and coke — Determination of volatile matter**

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**National foreword**

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## Hard coal and coke — Determination of volatile matter

*Houille et coke — Détermination des matières volatiles*



Reference number  
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## Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

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

This third edition cancels and replaces the second edition (ISO 562:1998), of which it constitutes a minor revision.

## Introduction

In this International Standard 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 be 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 can 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, it is required to give special care to the ash.

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 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 referenced documents are indispensable for the application 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 11722, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test 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 mass fraction of volatile matter is calculated from the loss in mass of the test portion after deducting the loss in mass due to moisture.

## 4 Reagents and materials

4.1 **Cyclohexane**, of recognized analytical grade.

## 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 in diameter and 150 mm long (see Figure 1).

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 (5.2).

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 zone of uniform temperature shall be at least 160 mm × 100 mm; in the latter case, a 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 specified tolerance of  $\pm 5$  °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.

When performing multiple determinations on highly swelling coals, it can 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.

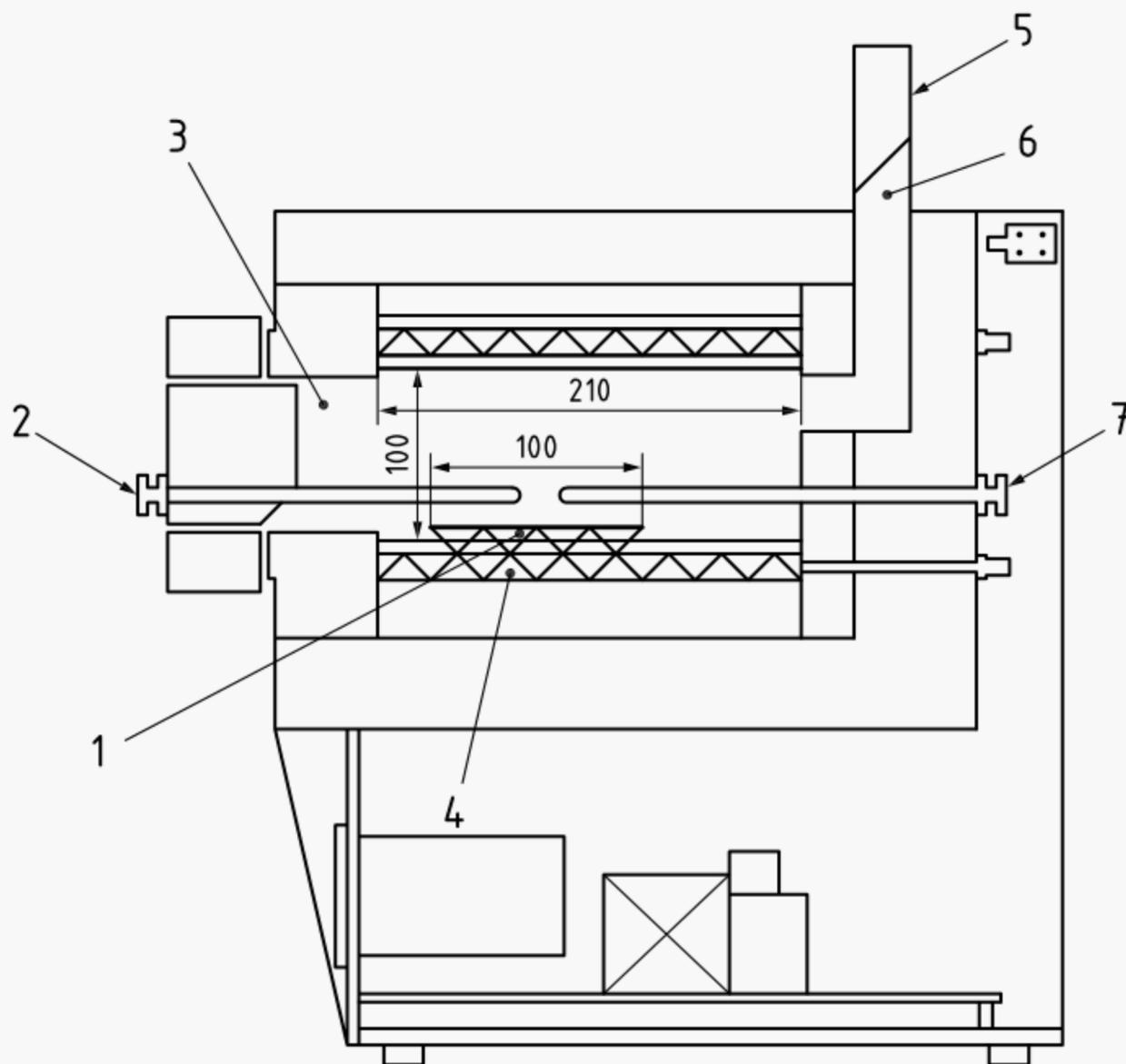
**5.4 Crucible stand**, on which the crucible is placed in the furnace, such that the appropriate rate of heating can be achieved.

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 in 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



**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

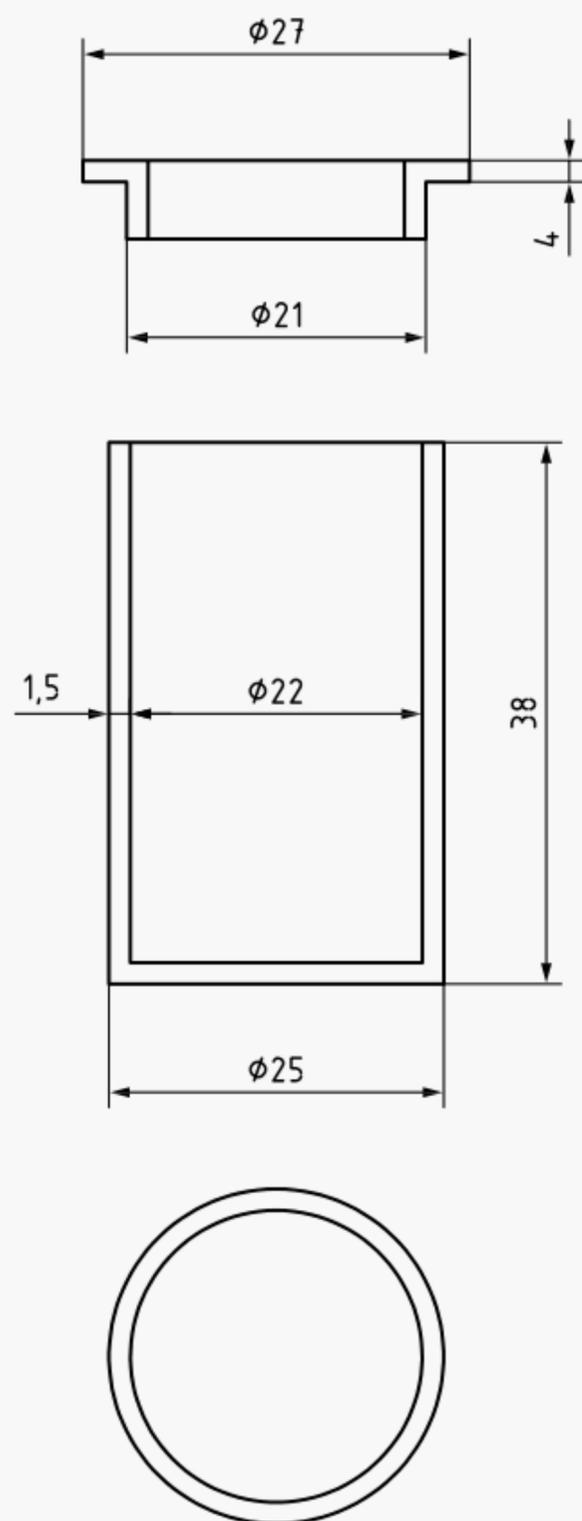
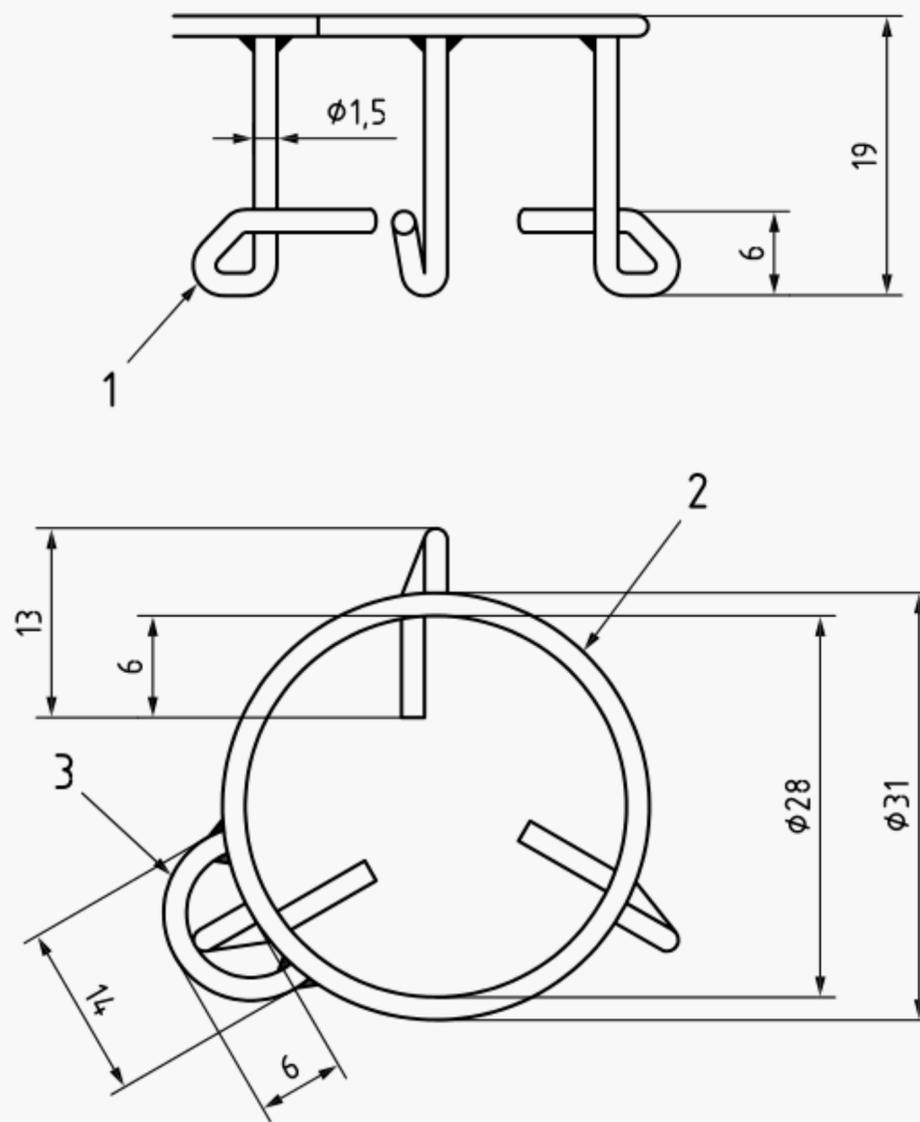
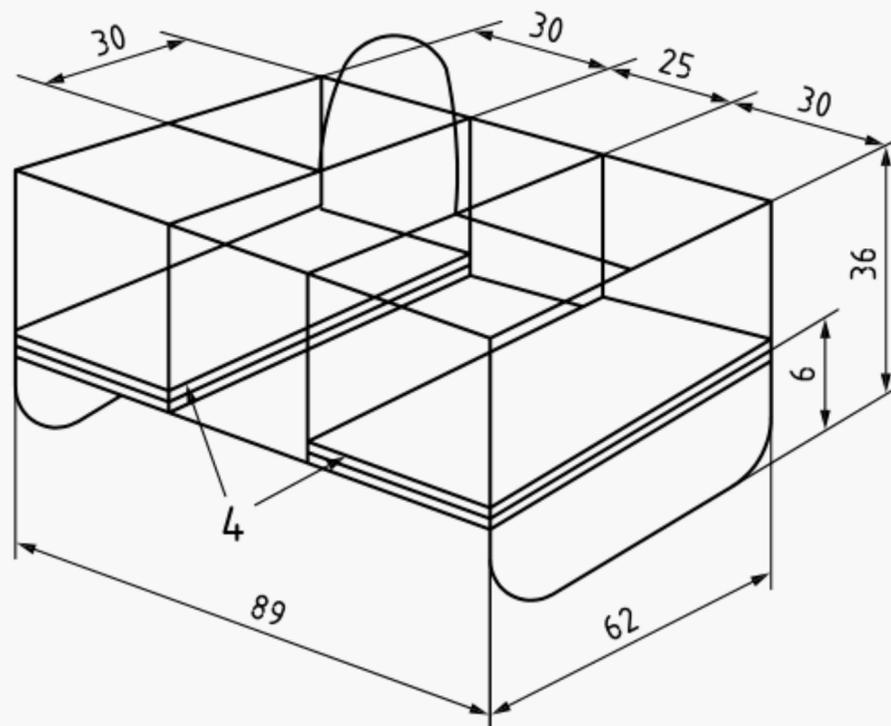


Figure 2 — Silica crucible and lid

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determinations

**Key**

- 1 three legs separated by 120°
- 2 ring

- 3 handle
- 4 ceramic plates

**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  $\mu\text{m}$  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 in parallel with 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  $900\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  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.

Temperature checking should be made before starting determinations. However, in routine operation when several analyses are performed per day, a monthly 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 [Figure 3 b)] and insert in the oven. Maintain at  $900\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  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,  $1\text{ g} \pm 0,1\text{ g}$  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 coke, remove the lid of the charged crucible, add 2 drops to 4 drops of cyclohexane (4.1) 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 charged crucible(s) in a cold stand, transfer to the furnace, close the door and leave for  $7\text{ min} \pm 5\text{ s}$ . If multiple determinations are being made, any vacant places in the stand should be filled with empty crucibles.

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).

NOTE 2 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.

## 8 Expression of results

The volatile matter,  $V$ , in the sample as analysed, expressed as a percentage mass fraction, is given by Equation (1):

$$V = \frac{100(m_2 - m_3)}{m_2 - m_1} - \omega_{\text{H}_2\text{O}} \quad (1)$$

where

- $m_1$  is the mass, expressed in grams, of the empty crucible and lid;
- $m_2$  is the mass, expressed in grams, of the crucible and lid and test portion before heating;
- $m_3$  is the mass, expressed in grams, of the crucible and lid and contents after heating;
- $\omega_{H_2O}$  is the moisture, expressed as a percentage mass fraction, in the sample as analysed, determined in accordance with the method specified in ISO 11722 for coal or ISO 687 for coke.

Report the result, as the mean of duplicate determinations, to the nearest 0,1 % mass fraction. 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.

## 9 Precision

### 9.1 Repeatability limit

The results of the duplicate determinations (performed within a short period of time, but not simultaneously) in the same laboratory by the same operator using the same apparatus on two representative test portions taken from the same general analysis test sample, shall not differ by more than the values given in Table 1.

**Table 1 — Repeatability and reproducibility limits for volatile matter**

Type of sample	Maximum acceptable differences between results obtained for volatile matter (calculated to the same moisture content)	
	Repeatability limit	Reproducibility limit
Hard coal < 10 %	0,3 % absolute	0,5 % absolute
Hard coal > 10 %	3 % of the mean result	0,5 % absolute or 4 % of the mean result, whichever is greater
Coke	0,2 % absolute	0,3 % absolute

### 9.2 Reproducibility limit

The means of the results of duplicate determinations, performed in each of two different laboratories on representative test portions taken from the same general analysis test sample shall not differ by more than the values given in Table 1.

## 10 Test report

The test report shall include the following information:

- a) reference to this International Standard, i.e. ISO 562;
- b) identification of the sample tested;
- c) date of the determination;
- d) results of the determination performed, together with the relevant analysis basis (e.g. air dry).

## Bibliography

- [1] ISO 1170, *Coal and coke — Calculation of analyses to different bases*
- [2] ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*







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