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Brown coals and lignites — Determination of the volatile matter in the analysis sample: one furnace method

Charbons bruns et lignites — Détermination des matières volatiles dans l'échantillon pour analyse : méthode avec utilisation d'un four

Reference number
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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 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).

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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*.

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 coals or lignites is heated out of contact with air under specified conditions. Due to the nature of brown coals and lignites, the sample being pressed and cut into small pellets is necessary to minimize the possibility of ejection of sample from the test crucible when the sample is heated at 900 °C, which has been demonstrated for its good precision and accuracy, and applied in GB/T 212 for many years. Results obtained by this method agree with measurements of volatile matter content by ISO 5071-1.

The test of volatile matter 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 are carefully controlled. It is also essential to exclude air from the coal 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.

To arrive at a valid comparison of volatile matter results conducted in different laboratories, it is essential that the moisture condition of the test samples in the two laboratories is within the expected variance of the moisture test. If a sample is re-equilibrated with the laboratory atmosphere or partially dried in one laboratory and not the other then oxidation can and will most definitely occur for brown coals and lignites. Oxidation will alter the, as-determined, volatile matter of a test sample.

The dry basis precision for volatile matter includes a variance contribution from the moisture determination and potentially a covariance component, both of which can influence the precision statistics for volatile matter on a dry basis.

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: one furnace method

1 Scope

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

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, *Hard coal and coke — Manual sampling*

3 Terms and definitions

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

3.1
total volatile matter
$w_{V,T}$
fractional loss in mass, without correction for moisture, when a solid mineral fuel is heated out of contact with air under specified conditions

3.2
volatile matter
loss in mass, corrected for moisture, when a solid mineral fuel is heated out of contact with air under specified conditions

[SOURCE: ISO 1213-2:2016, 3.239]

4 Principle

A portion of the general analysis sample which is pressed and cut into pellets with the side length of about 3 mm 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.

5 Reagent and materials

5.1 Desiccant, fresh or freshly regenerated and preferably self-indicating. Suitable desiccants are magnesium perchlorate and silica gel.

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

6 Apparatus

6.1 Furnace, heated by electricity, in which a zone of uniform temperature of 900 °C ± 5 °C can be maintained (see Figure 1**,** which is the same as the one given in ISO 562:2010, Figure 1).

It may be of the stop-ended type or fitted at the back with a flue approximately 25 mm in diameter and 150 mm in length.

It is important for furnaces with flues that the furnace door seals 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 should be such that, with an initial temperature of 900 °C, the temperature of 900 °C ± 10 °C is regained within 3 min after insertion of a cold stand and its crucibles. The temperature shall be measured with a thermocouple as described in 6.2. The initial temperature of 900°C may be adjusted to a temperature determined by calibration that will guarantee the furnace temperature of 900 °C ± 10 °C is regained within 3 min after insertion of the cold stand and its crucible(s).

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.

6.2 Thermocouple, unsheathed.

The thermo-junction shall be inserted midway between the base of the crucibles in its stand and the floor of the furnace from which there is 20 mm to 30 mm distance. 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 the 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 thus inserted only when necessary.

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

6.3 Crucible and lid, cylindrical, with a well-fitting lid, both made of glazed porcelain. Crucibles of fused silica and other refractory materials can be used, provided that they give the results which agree with the recommended porcelain crucible, within the stated precision of the method (see Clause 10).

The glazed porcelain crucible and lid shall have a mass between 15 g and 20 g, and dimensions approximate to those shown in Figure 2 a), and the fused silica crucible and lid shall have a mass between 10 g and 14 g, and dimensions approximate to those shown in Figure 2 b).

The fit of the lid on the crucible is critical for the determination; 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 lid shall be ground together to give a smooth surface, and they will then be given a common distinguishing mark.

6.4 Crucible stand, made of nichrome wire or other heat-resistant metal, on which the crucible is placed in the furnace, so that the appropriate specified rate of heating can be achieved (see Figure 3).

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 in thickness, resting on the inner projection of its legs, or

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

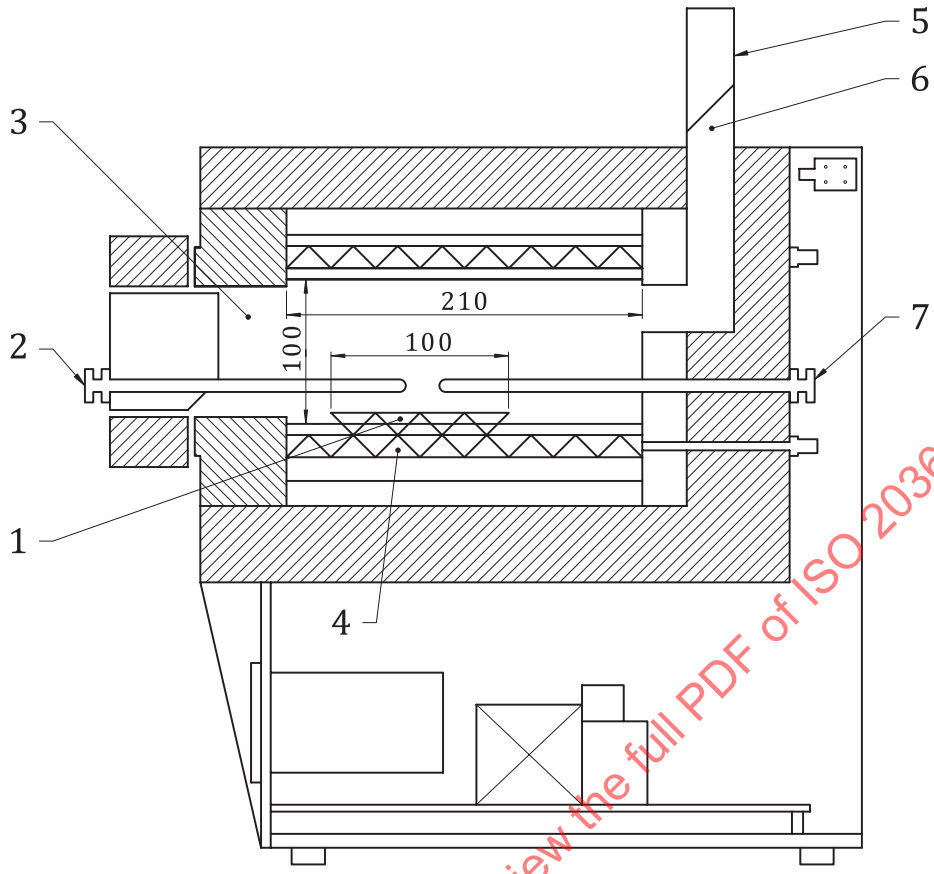
6.5 Crucible stand tong (see Figure 4).

6.6 Analytical balance, with a resolution of at least 0,1 mg.

6.7 Pellet presser, screw or level type (see Figure 5), mechanical or automatic, capable of making a sample cake with the thickness of 1,0 cm to 1,5 cm and about 1,0 cm in diameter under the force created by a mass of over 100 kg (980 N). If the pellets presser can display the force (displayed as mass, in kg) automatically, it can be set between a mass of 200 kg (1 960 N) and a mass of 300 kg (2 940 N), for example a mass of 280 kg (2 740 N).

6.8 Desiccator, with fresh desiccant (5.1) to assure that the air in it is dry.

Dimensions in millimetres

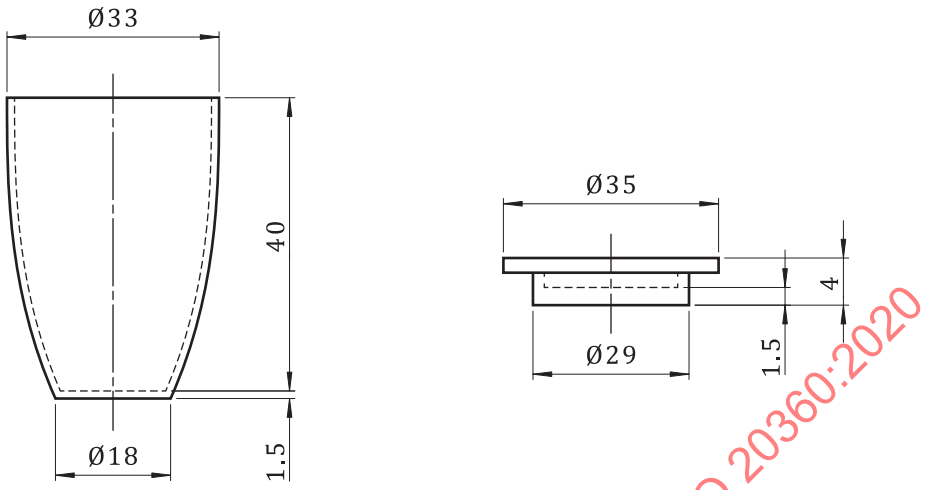


Key

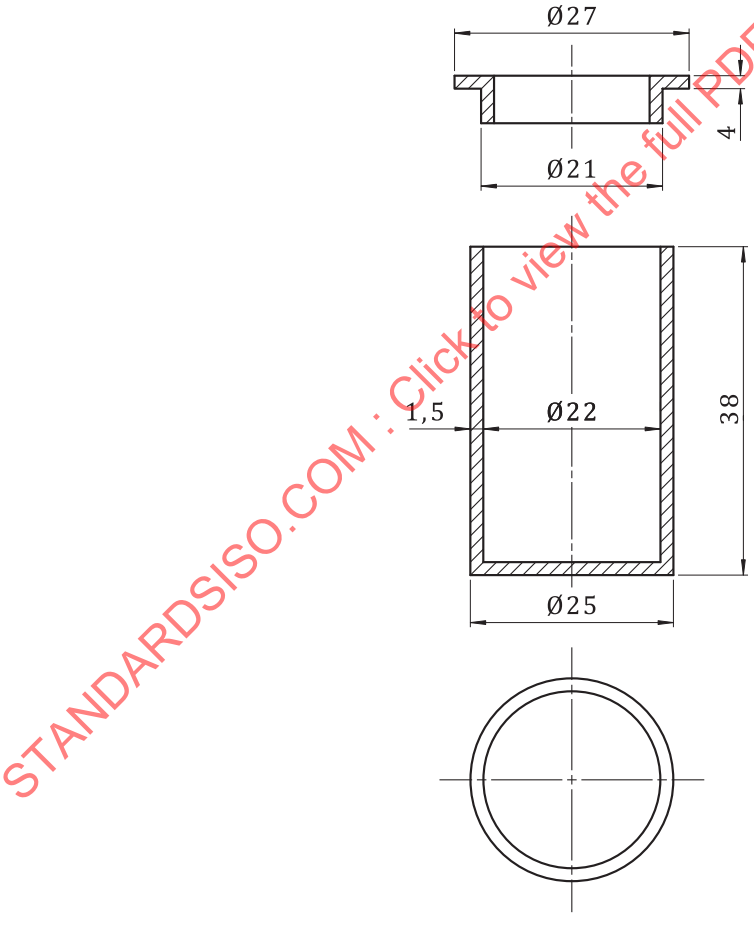
1	zone of uniform temperature	5	flue
2	check thermocouple	6	valve
3	chamber (width 200 mm)	7	thermocouple
4	heating system		

Figure 1 — Example of a suitable furnace

Dimensions in millimetres



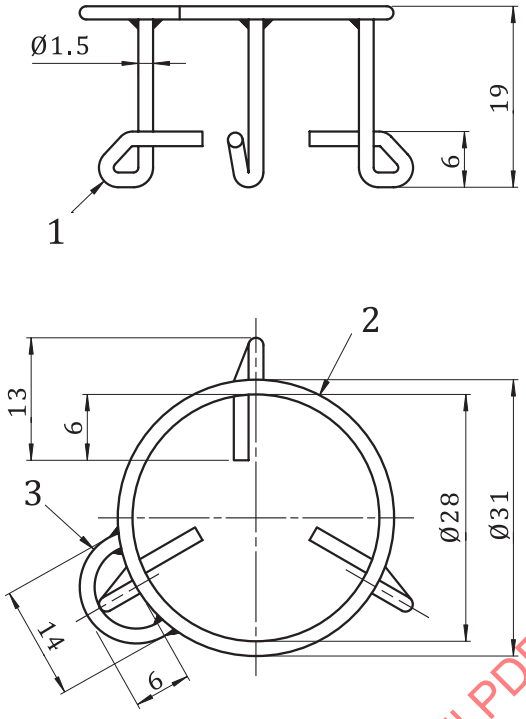
a) Glazed porcelain crucible and lid



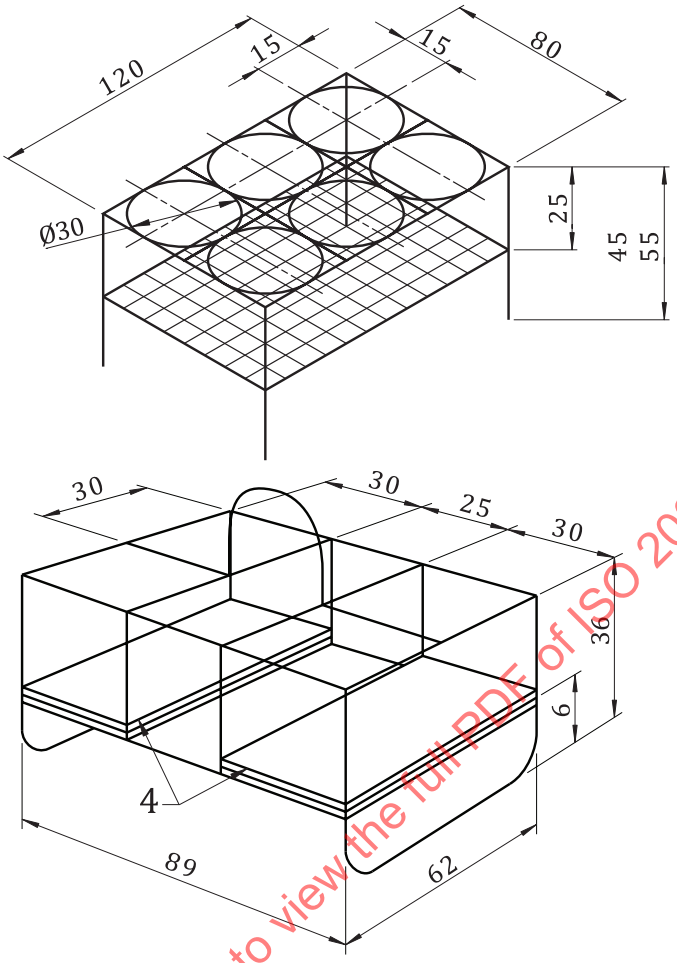
b) Fused silica crucible and lid

Figure 2 — Crucible and lid

Dimensions in millimetres



a) Suitable for a single determination



b) Suitable for multiple determination

Key

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

Figure 3 — Crucible stands

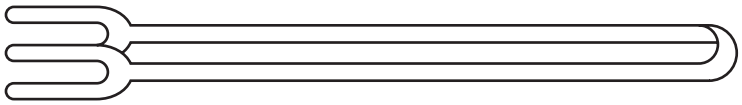
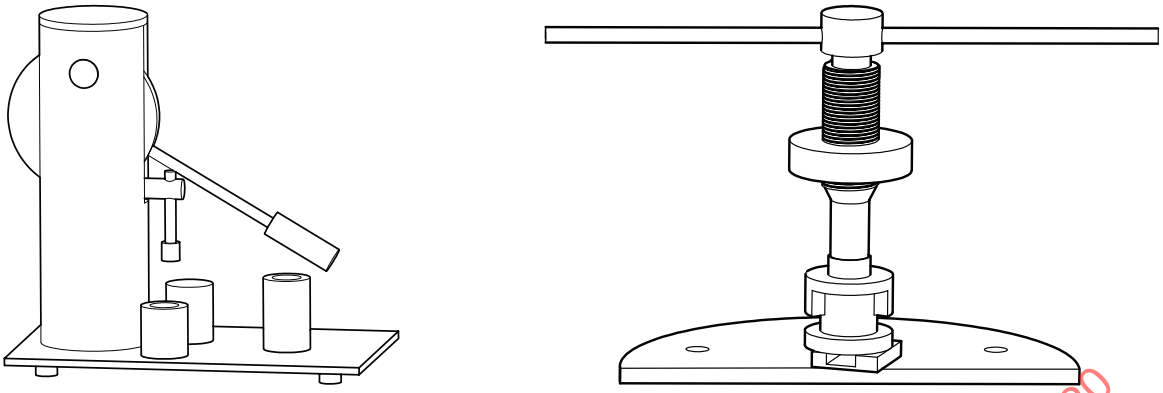


Figure 4 — Example of a crucible stand tong



a) Level type

b) Screw type

Figure 5 — Examples of a pellet presser

7 Preparation of test sample

7.1 Preparation of the general analysis test 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.

7.2 Preparation of the sample pellets

Samples are pressed into cakes and then cut into pellets for analysis. Take an appropriate amount of analysis test sample (about 1,0 g to 1,5 g for each cake) and press it into a cake by using the pellet presser (6.7). The cake is expected to have a thickness of 1,0 cm to 1,5 cm and 1,0 cm in diameter. Store each pressed cake in a glass bottle with ground edges and fitted with a ground-glass cover. Cut the pressed cakes into small pieces (pellets) with a side length of about 3 mm size by using a clean knife on a mat that neither absorbs water nor releases water. Immediately weigh the pellets into a pre-processed crucible (8.2) to a mass of 0,99 g ± 1,01 g to provide duplicates of each sample for the determination of volatile matter.

8 Procedure

8.1 Furnace temperature check

Adjust the temperature zone in the furnace (6.1), 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 (6.2). 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 (6.1) should be dealt with in a similar way.

8.2 Crucible and lid process

Fill either a stand with one empty crucible and lid or a stand with the requisite number of empty crucibles and lids, and insert it in the furnace set at 900 °C. Maintain at 900 °C for 7 min. Remove the crucible(s) from the furnace by using the crucible stand tong (6.5) and cool them first on a thick metal plate and finally in a desiccator (6.8) filled with desiccant to room temperature. As soon as it is cool, weigh each paired empty crucible and lid to the nearest 0,000 1 g.

8.3 Volatile matter determination

Accurately weigh 0,99 g to 1,01 g to the nearest 0,000 1 g of the pelleted sample into each pre-processed crucible, replace the lid and shake each charged crucible slightly to make the test pellets form a layer on the bottom of the crucible.

Place the charged crucible(s) in a cold stand and insert into the furnace at 900 °C; close the door and leave for 7 min ± 3 s. The temperature of 900 °C ± 10 °C should be regained within 3 min after insertion of a cold stand and its crucible(s), then keep the temperature of 900 °C ± 10 °C, otherwise this run is invalid. Heating time (7 min ± 3 s) includes temperature recovery time (3 min). Remove the crucible(s) from the furnace by using the crucible stand tong (6.5) and allow to cool to room temperature in the same manner as done for the empty crucible(s). When they have cooled, weigh crucible(s) to the nearest 0,000 1 g in the same manner as for the empty crucible(s). The duplicate determinations are performed within a short period of time, but not simultaneously.

NOTE 1 The initial temperature of 900 °C can be adjusted to a temperature determined by calibration that will guarantee the furnace temperature of 900 °C ±10 °C is regained within 3 min after insertion of the cold stand and its crucible(s)

NOTE 2 The similar treatment of the crucible(s) before and after the determination minimizes the effect of any film of water absorbed on its surface, while the rapid cooling reduces absorption of moisture by the coal residue.

If multiple determinations are being made, fill any vacant places in the stand with empty crucibles.

9 Expression of results

9.1 The total volatile matter in the sample as analysed, $w_{V,T}$, expressed as a percentage by mass, is given by the following Formula (1):

$$w_{V,T} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where

$w_{V,T}$ is the mass fraction of the total volatile matter in the sample as analysed without correction for moisture, expressed as a percentage, %;

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

m_2 is the mass of the crucible, lid and sample pellets taken before heating, expressed in grams;

m_3 is the mass of the crucible, lid and sample pellets after heating, expressed in grams;

100 is a conversion factor from a dimensionless mass fraction to percentage, expressed as a percentage, %.

9.2 The volatile matter on an air-dried basis, $w_{V,ad}$, expressed as a percentage by mass, is given by the following Formula (2):

$$w_{V,ad} = w_{V,T} - w_{M,ad} \quad (2)$$

where

$w_{V,ad}$ is the mass fraction of volatile matter on air-dried basis, expressed as a percentage, %;

$w_{M,ad}$ is the mass fraction of moisture in the air-dried sample, expressed as a percentage, %.

9.3 The volatile matter on a dry basis, $w_{V,d}$, expressed as a percentage by mass, is given by the following Formula (3):

$$w_{V,d} = \frac{100}{100 - w_{M,ad}} \times w_{V,ad} \quad (3)$$

9.4 The results (the mean of duplicate determinations) shall be reported to the nearest 0,1 %. The results to other bases shall be calculated by the specification in ISO 1170.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus within a short interval of time on representative portions taken from the same analysis sample, shall not differ by more than the values of the repeatability limit, *r*, shown in Table 1.

10.2 Reproducibility limit

The mean of the results of duplicate determinations, carried out in each of the two laboratories, on the representative portions taken from the same sample at the last stage of sample preparation, shall not differ by more than the values of the reproducibility limit, *R*, shown in Table 1.

Table 1 — Repeatability limit and reproducibility limit

Test parameter %	Maximum acceptable differences between results % absolute	
	Repeatability limit	Reproducibility limit
$w_{V,T}$	$r_{ad} = 0{,}33$	$R_{ad} = 0{,}088\,1\,w_{M,ad} + 0{,}009\,94\,w_{V,T} - 0{,}704$
$w_{V,d}$	$r_{d} = 0{,}33$	$R_{d} = 0{,}104\,w_{M,ad} - 0{,}022\,5\,w_{V,d} + 0{,}924$

11 Test report

The test report shall include the following information:

a) identification of the sample tested;

b) the method used by reference to this document, i.e. ISO 20360:2020;

c) the results and the method of expression used;

d) any deviations from the procedure;