INTERNATIONAL STANDARD 562

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Hard coal and coke — Determination of the volatile matter

Houille et coke — Détermination du taux de matières volatiles

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 562 and found it technically suitable for transformation. International Standard ISO 562 therefore replaces ISO Recommendation R 562-1967 to which it is technically identical.

ISO Recommendation R 562 was approved by the Member Bodies of the following countries :

Australia	Iran	Sweden
Austria	Italy	Switzerland
Belgium	Japan	Turkey
Brazil	Philippines	United Kingdom
Chile	Poland	U.S.A.
Czechoslovakia	Portugal	U.S.S.R.
Germany	Romania	Yugoslavia
Greece	South Africa, Rep. of	
India	Spain	

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

France
Netherlands

No Member Body disapproved the transformation of ISO/R 562 into an International Standard.

Printed in Switzerland

Hard coal and coke — Determination of the volatile matter

0 INTRODUCTION

The volatile matter content 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 shall be carefully controlled. The moisture content of the sample shall be 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 test, the magnitude of the loss being dependent on both the nature and the quantity of the minerals present. When the carbonate content of a coal is high or when the result is required for the purpose of classifying the coal, it is necessary to make a correction to the determined volatile matter for the loss of carbon dioxide occurring during the determination. A first approximation to this correction is obtained by the use of the third formula in clause 8.

NOTE — Insufficient experimental evidence is available to justify a correction for loss of carbon dioxide being recommended when determining the volatile matter of coke. On the other hand, the error from this source is unlikely to be great, as carbonates are decomposed in the coke oven in making coke, although some carbonates may be added subsequently if the coke is quenched with waste liquor.

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

1 SCOPE AND FIELD OF APPLICATION

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 REFERENCES

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

ISO 348, *Hard coal — Determination of moisture in the analysis sample — Direct volumetric method.*

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

ISO/R 925, *Determination of carbon dioxide in coal by the gravimetric method.*

ISO/R 1171, *Determination of ash of solid mineral fuels.*

3 PRINCIPLE

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

4 REAGENTS

4.1 Desiccant, fresh or freshly regenerated and preferably self-indicating. Suitable desiccants are silica gel, activated alumina and anhydrous calcium sulphate.

4.2 Benzene (for use with coke samples).

5 APPARATUS

5.1 Muffle furnace, heated by electricity, in which an adequate zone at a constant and uniform temperature of 900 ± 10 °C can be maintained. It may be of the stop-ended type or fitted at the back with a flue approximately 25 mm diameter by 150 mm long.

Its heat capacity shall be such that, with an initial temperature of 900 °C, a minimum temperature of 885 °C is regained within 4 min, preferably within 3 min, of the insertion of a cold stand and its crucible(s), the temperature being measured with an unsheathed thermocouple, as described below. Normally the furnace[1)] will be designed specifically either for receiving one crucible and its stand or for multiple determinations using a number of crucibles in one stand.

NOTE — The temperature of 900 °C shall be attained as closely as possible and the tolerance of ± 10 °C is specified so as to meet inherent errors in the temperature measurement and lack of uniformity in the temperature distribution.

1) A suitable furnace for use with one crucible is specified in the British standard B.S. 1016, Part 3, and a suitable furnace for use with several crucibles is specified in the German standard DIN 51720.

A position for the crucible stand shall be chosen within the zone of uniform temperature and this position used for all determinations.

5.2 Pyrometer. The temperature characteristics of the furnace shall be checked with an unsheathed thermocouple, of wire not thicker than 1 mm. The thermojunction 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, then 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 thermojunction 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 thermojunction maintained at elevated temperatures gradually changes with time.

5.3 Crucible and lid. A cylindrical crucible with a well fitting lid, both of fused silica. The crucible and lid shall weigh between 10 and 14 g and have dimensions approximating to those shown in figure 1. 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.

NOTE – When carrying out 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 content, provided that the specified rate of temperature recovery is maintained.

Dimensions in millimetres

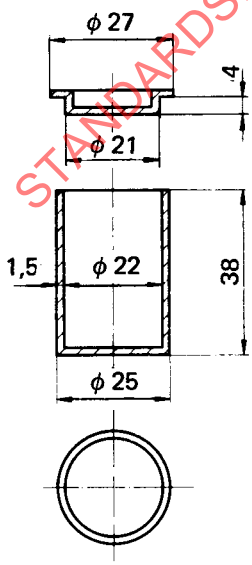


FIGURE 1 – **Silica crucible and lid**

Crucibles of other refractory materials or of platinum may be used if they give the same results as the recommended silica crucible, within the stated tolerances.

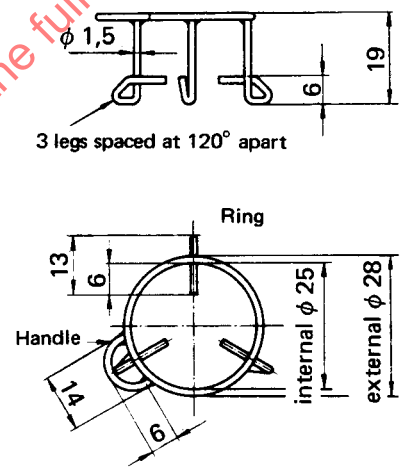
5.4 Stand, on which the crucible is placed in the muffle furnace, such that the appropriate rate of heating can be achieved. For example, it may consist of the following :

a) for single determinations : a ring of heat-resistant steel wire as shown in figure 2a), with two asbestos disks, 25 mm diameter and 1 mm thick, resting on the inner projection of its legs; or

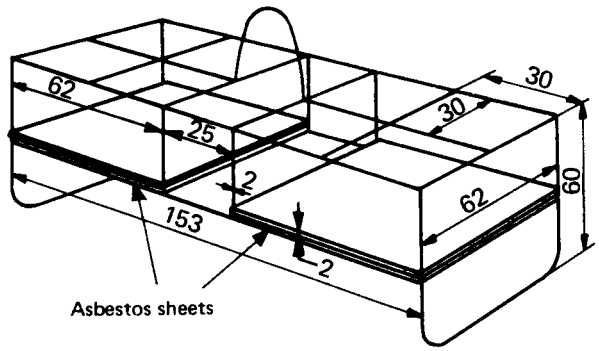
b) for multiple determinations : a tray of heat-resistant steel wire as shown in figure 2 b), of appropriate size, with an asbestos plate 2 mm thick supporting the crucibles.

5.5 Balance, sensitive to 0,1 mg.

Dimensions in millimetres



a) **Suitable for making one determination at a time**



b) **Suitable for making several determinations at a time**

FIGURE 2 – **Crucible stands**

6 PREPARATION OF SAMPLE

The sample used for the determination of volatile matter is the analysis sample ground to pass a sieve of 0,2 mm aperture.

If necessary expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

Before commencing the determination, mix the air-dried sample of coal or coke thoroughly for at least 1 min, preferably by mechanical means.

7 PROCEDURE

Heat at 900 ± 10 °C for 7 min either one crucible and lid or the requisite number of crucibles and lids to fill the multiple stand. Remove from the furnace, cool the crucible(s) first on a metal slab and finally in a desiccator located next to the balance. As soon as these are cool, weigh each empty crucible and lid and weigh accurately into each crucible 1,00 to 1,01 g of sample. 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.

NOTE – Precisely similar treatment of the crucible 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 or coke residue.

Adjust the temperature of the zone in the muffle furnace containing a stand and empty crucible(s), to 900 ± 10 °C as indicated by the correctly located unsheathed thermocouple, or to the equivalent temperature as indicated by the sheathed thermocouple.

Remove the stand and empty crucible(s) and close the door of the muffle furnace to restore steady temperature conditions. If the sample is of coke, remove the lid, add 2 to 4 drops of benzene and replace the lid. Place the charged crucible(s) in another stand, transfer to the muffle furnace and leave for a period of exactly 7 min. Remove, cool and weigh the crucible(s) in the same manner as for the empty crucible(s).

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

8 EXPRESSION OF RESULTS

The volatile matter on dry, ash free basis ($V_{d.a.f.}$)[1], expressed as a percentage, is given by the formulae

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

$$V_{d.a.f.} = \frac{V \times 100}{100 - (M + A)}$$

$$V_{d.a.f.}\ (\text{corrected for } CO_2) = (V - CO_2) \times \frac{100}{100 - (M + A)}$$

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 sample 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, in the sample as analysed, determined in accordance with ISO 331, ISO 348 or ISO 687;

A is the ash, as a percentage, of the sample as analysed, determined in accordance with ISO/R 1171;

CO_2 is the carbon dioxide, as a percentage, in the sample as analysed, determined in accordance with ISO/R 925;

V is the volatile matter, as a percentage, in the sample as analysed.

The results (preferably the mean of duplicate determinations, see clause 9) shall be reported to the nearest 0,1 %.

9 PRECISION OF THE METHOD

Sample	Maximum acceptable differences between results calculated to the same moisture content	
	Repeatability	Reproducibility
Hard coal of volatile matter content < 10 %	0,3 % absolute	0,5 % absolute
Hard coal of volatile matter content ⩾ 10 %	3/100 of the mean result	0,5 % absolute or 4/100 of the mean result, whichever is the greater
Coke	0,2 % absolute	(See 8.3)

9.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus on two representative portions taken from the same analysis sample, shall not differ by more than the above values.

1) Calculation of the results to other bases is dealt with in ISO/R 1170.

9.2 Reproducibility (hard coal)

The means of the results of duplicate determinations, carried out in each of two different laboratories on representative portions taken from the same sample after the last stage of sample preparation, shall not differ by more than the above values.

9.3 Reproducibility (coke)

No value for reproducibility can be quoted for determinations on coke carried out in different laboratories, as insufficient evidence is available for this to be done.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.