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DETERMINATION OF THE VOLATILE MATTER OF HARD COAL AND OF COKE

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March 1967

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 562, *Determination of the Volatile Matter of Hard Coal and of Coke*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1950 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In October 1962, this Draft ISO Recommendation (No. 550) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

Two Member Bodies opposed the approval of the Draft:

France,
Netherlands

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 562 March 1967

DETERMINATION OF THE VOLATILE MATTER OF HARD COAL AND OF COKE

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 should be carefully controlled. The moisture content of the sample should 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 section 6.

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

This ISO Recommendation describes the method of determining the volatile matter of hard coal and of coke. It is not applicable to brown coals and lignites.

2. PRINCIPLE

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

3. REAGENTS

3.1 **Desiccant.** Fresh or freshly regenerated and self indicating. Suitable desiccants are silica gel, activated alumina and anhydrous calcium sulphate.

3.2 **Benzene** (for use with coke samples).

4. APPARATUS

4.1 **Muffle furnace,** heated by gas or electricity in which an adequate zone of 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 is such that, with an initial temperature of 900 °C, a minimum temperature of 885 °C is regained within 4 minutes, preferably within 3 minutes, 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 * 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 should 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.

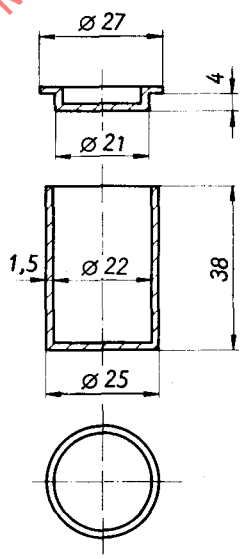
A position for the crucible stand is chosen within the zone of uniform temperature and this position is used for all determinations.

4.2 **Pyrometer.** The temperature characteristics of the furnace are checked with an unsheathed thermocouple, of wire not thicker than 1 mm. The thermojunction is 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 is 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 should 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.

4.3 **Crucible and lid.** A cylindrical crucible with a well fitting lid, both of fused silica. The silica crucible and lid should 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 should 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 should 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

FIG. 1. — **Silica crucible and lid**

* 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.

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.

4.4 Stand. The stand on which the crucible is placed in the muffle furnace should be 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 2(*a*), 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.

4.5 Balance, sensitive to 0.1 mg.

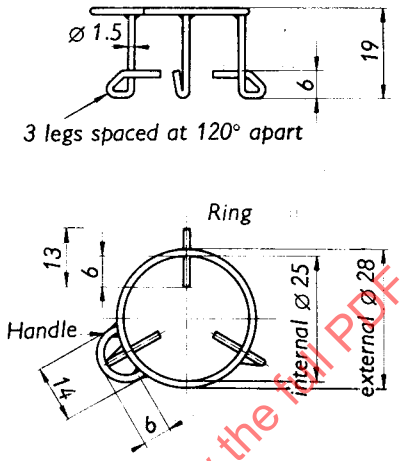
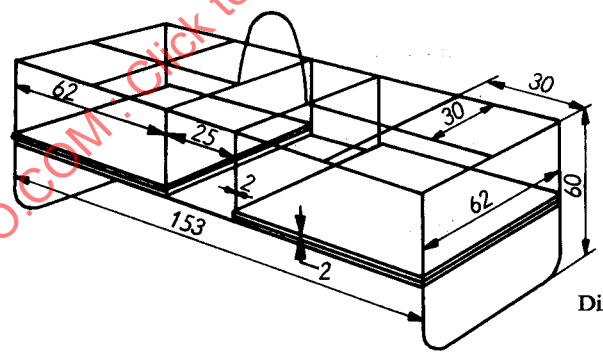


FIG. 2 (*a*). — **Suitable for making one determination at a time**



Dimensions in millimetres

FIG. 2 (*b*). — **Suitable for making several determinations at a time**

FIG. 2. — **Crucible stands**

5. SAMPLE

The sample used for the determination of volatile matter should be the analysis sample ground to pass a sieve of 0.2 mm aperture.

The sample should be exposed to the laboratory atmosphere in a thin layer for the minimum time necessary for the moisture content to reach approximate equilibrium.

6. PROCEDURE

Before commencing the determination, mix the air-dried sample of coal or coke, ground to pass a sieve of 0.2 mm aperture, thoroughly for at least 1 minute, preferably by mechanical means.

Heat at 900 ± 10 °C for 7 minutes 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, whilst 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 minutes. 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.

7. CALCULATION AND EXPRESSION OF RESULTS

If m_1 = mass of empty crucible and lid, expressed in grammes;

m_2 = mass of crucible and lid and sample before heating, expressed in grammes;

m_3 = mass of crucible and lid and contents after heating, expressed in grammes;

M_1 = moisture in the sample as analysed, expressed as a percentage; *

A = ash of the sample as analysed, expressed as a percentage; *

CO_2 = carbon dioxide in the sample as analysed, ** expressed as a percentage;

V = volatile matter in the sample as analysed, expressed as a percentage;

$V_{d.a.f.}$ = volatile matter on dry, ash free basis, expressed as a percentage;

then

$$V = \frac{100\,(m_2 - m_3)}{(m_2 - m_1)} - M_1$$

$$V_{d.a.f.} = \frac{V \times 100}{100 - (M_1 + A)}$$

$$V_{d.a.f.}\ (\text{corrected for } CO_2) = (V - CO_2) \times \frac{100}{100 - (M_1 + A)}$$

* The appropriate ISO methods of analysis should be used, as follows:

ISO/R 331, *Determination of Moisture in the Analysis Sample of Coal by the Direct Gravimetric Method;*

ISO/R 348, *Determination of Moisture in the Analysis Sample of Coal by the Direct Volumetric Method;*

ISO/R ..., *Determination of Moisture in the General Analysis Sample of Coke*, at present Draft ISO Recommendation No. 682;

ISO/R 158, *Determination of Ash of Hard Coal;*

ISO/R ..., *Determination of Ash of Coke*, at present Draft ISO Recommendation No. 680;

** ISO/R ..., *Determination of Carbon Dioxide in Coal*, at present at the stage of draft proposal. See Note of Introduction.