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DETERMINATION OF MOISTURE IN THE ANALYSIS SAMPLE OF COAL BY THE DIRECT GRAVIMETRIC METHOD

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BRIEF HISTORY

The ISO Recommendation R 331, *Determination of Moisture in the Analysis Sample of Coal by the Direct Gravimetric Method*, was drawn up by Technical Committee ISO/TC 27. *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (B.S.I.),

Work on this question by the Technical Committee began in 1950 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In February 1958, this Draft ISO Recommendation (No. 211) 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:

- Austria
- Bulgaria
- Burma
- Canada
- Chile
- Czechoslovakia
- Denmark
- France
- Germany
- India
- Israel
- Italy
- Japan
- Netherlands
- New Zealand
- Poland
- Portugal
- Republic of South Africa
- Romania
- Spain
- Turkey
- United Kingdom
- U.S.A.
- U.S.S.R.
- Yugoslavia

One Member Body opposed the approval of the Draft:

Belgium.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 331 August 1963

DETERMINATION OF MOISTURE IN THE ANALYSIS SAMPLE OF COAL BY THE DIRECT GRAVIMETRIC METHOD

1. INTRODUCTION

Two direct methods are available for the determination of moisture in the analysis sample of coal:

(1) a gravimetric method, in which the moisture is determined in an atmosphere of nitrogen, and

(2) a volumetric method,* in which the moisture is determined by distillation.

This ISO Recommendation describes the direct gravimetric method.

Since coal is hygroscopic, its moisture will vary with change of humidity of the atmosphere, and the moisture in the analysis sample, therefore, should be determined whenever portions are weighed out for other analytical determinations, e.g. volatile matter, calorific value, carbon and hydrogen etc. If all the portions taken for analysis are weighed out on the same day and at about the same time, and if the analyses are proceeded with without delay, one determination of moisture will suffice.

2. PRINCIPLE

Coal is heated at 105 to 110 °C in a stream of oxygen-free, dry nitrogen and the moisture driven off is collected in an absorption tube containing a desiccant. The increase in weight of the absorption tube (after deducting the result of a separate blank determination) is due to moisture in the coal (see Note below).

NOTE. - If suitable precautions are taken against re-absorption of moisture by the dried coal, the loss in weight of the sample may be measured and compared with the gain in weight of the absorption tube. This procedure is of advantage in discriminating between coals which are rich in absorbed gases and those which are not.

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* See ISO Recommendation R 348, *Determination of Moisture in the Analysis Sample of Coal by the Direct Volumetric Method.*

3. APPARATUS

The balance used should be sensitive to 0.1 mg.

3.1 **Source of heat,** such that a glass retort tube is maintained at a constant and uniform temperature within the range 105 to 110 °C. A convenient source is e.g. an electrically heated and thermostatically controlled aluminium block oven.

3.2 **Drying tower,** packed with a desiccant to dry the stream of nitrogen entering the retort tube.

3.3 **Flowmeters,** capable of measuring a flow rate sufficient to maintain two atmosphere changes per minute in the retort tube. If a pressure drop over a constriction is used as a means of measuring flow rate, the manometer liquid should be a non-volatile oil.

3.4 **Retort tubes.** Glass retort tubes of about 50 ml capacity, having a suitable inlet for dry nitrogen and an outlet for moisture-laden nitrogen, and capable of holding 1 g of the sample, spread out in a uniform layer either in the retort tube itself or in a boat which is inserted into the tube.

3.5 **Boats** (if employed), of non-oxidizable material, such as glass or glazed porcelain.

3.6 **Absorption tubes,** of a suitable design and able to contain sufficient desiccant to remove the moisture completely from the nitrogen stream.

4. REAGENTS

4.1 *Desiccant.* A suitable desiccant is e.g. dry magnesium perchlorate (see Note below). It is important that the same desiccant be used in both the drying tower and the absorption tubes, since the incoming nitrogen and the gas leaving the system should be dried to exactly the same degree.

NOTE. - Attention is drawn to the care needed in handling magnesium perchlorate. When exhausted, it should be washed down the sink with a current of water.

4.2 *Nitrogen.* With a maximum oxygen content of 30 parts per million (see Appendix, page 7).

5. PROCEDURE

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

Adjust the flow rate of the nitrogen (4.2) passing through the drying tower and flowmeter so as to give two atmosphere changes per minute through the retort tube. Connect an empty retort tube to a closed absorption tube and to the nitrogen stream from the flowmeter. Check for leaks in the system, open the absorption tube and re-adjust the flow rate, if necessary.

Insert the retort tube into the oven heated at 105 to 110 °C and pass the nitrogen (4.2) through it for 15 minutes. Close the absorption tube, disconnect, wipe and allow it to stand for 20 minutes in the balance room. Open the tube momentarily to equalize the pressures and then weigh to the nearest 0.2 mg, using a similar tube as a counterpoise (see Note 1 below).

Whilst the absorption tube is standing in the balance room prior to weighing, remove the retort tube from the oven and allow to cool with a stream of dry nitrogen passing through it. When cool, weigh accurately about 1 g of the coal into the tube and spread in a uniform layer not exceeding 0.15 g of coal per square centimetre.

Re-connect the weighed absorption tube, check for leaks as before and, with the nitrogen stream flowing, heat the sample at 105 to 110 °C.

After a suitable time (see Note 2 below), close, disconnect and weigh the absorption tube, following exactly the procedure described in the third paragraph of this section. Stop the flow of nitrogen whilst the absorption tube is disconnected and close the retort tube outlet. After weighing, re-connect the absorption tube, adjust the nitrogen flow rate as before and continue the heating.

Repeat this procedure at intervals of 30 minutes until the absorption tube does not increase in mass by more than 0.2 mg.

Alternatively, the coal may be weighed into a boat, which is inserted directly into the retort tube after the absorption tube has been connected. In this case, there is no need to remove the retort tube from the oven or to cool it between determinations, unless it also functions as a weighing bottle which can be sealed whilst the loss in mass of the coal is determined (see Note, page 3).

NOTES

1. When weighing the absorption tube, a similar tube may be employed as a counterpoise; this tube is treated in the same way as the absorption tube, except that nitrogen is not passed through it. During the determination, the counterpoise is suspended alongside the absorption tube.

2. For anthracites and bituminous coals, heating for one hour is sufficient. For lignites and brown coals, a longer period is required, and the time of heating varies with different coals.

5.1 Blank determination

Determine the apparatus blank by carrying out the procedure exactly as described above, but omitting the sample. The increase in mass of the absorption tube due to the blank determination should not be more than 1 mg. Deduct the value determined by the blank determination from the mass of water collected in each determination.

6. CALCULATION AND EXPRESSION OF RESULTS

If m = mass of coal taken, expressed in grammes,

a = increase in mass of the absorption tube during the test, expressed in grammes.

a_0 = increase in mass of the absorption tube during the blank determination, expressed in grammes,

M = moisture in the coal analysed, expressed in per cent,

then the moisture per cent is given by the formula

$$M = \frac{(a - a_0)}{m} \times 100$$

The results (preferably the mean of duplicate determinations, see clause 7.1 below) should be reported to the nearest 0.1 per cent.

7. ACCURACY OF DETERMINATION

Moisture	Maximum acceptable differences between results obtained	
	in the same laboratory	**in different laboratories**
	0.2 per cent absolute	(see clause 7.2)

7.1 In the same laboratory

The results of duplicate determinations carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same sample after the last stage of the reduction process, should not differ by more than the above value.

7.2 In different laboratories

No tolerance can be quoted for determinations carried out in different laboratories, since the results obtained will depend on the humidity conditions in the different laboratories, which are not necessarily the same.