International Standard 331

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Coal — Determination of moisture in the analysis sample — Direct gravimetric method

Charbon — Détermination de l'humidité de l'échantillon pour analyse — Méthode gravimétrique directe

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Foreword

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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.

International Standard ISO 331 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*.

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Belgium

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Coal — Determination of moisture in the analysis sample — Direct gravimetric method

0 Introduction

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, for example 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 begun without delay, one determination of moisture will suffice.

1 Scope and field of application

This International Standard specifies a method of determining the moisture content of the analysis sample of hard coal, brown coal and lignite using a gravimetric procedure.

NOTE — Alternative volumetric methods are given in ISO 348 and ISO 1015.

2 References

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

ISO 1015, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

3 Principle

The sample of 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 mass of the absorption tube (after deducting the result of a separate blank determination) is due to moisture in the sample of coal.

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

4 Reagents

4.1 Desiccant

A suitable desiccant is dry magnesium perchlorate (see the note). 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 the handling and the disposal of magnesium perchlorate. National requirements must be observed.

4.2 Nitrogen

Maximum oxygen content: 30 ppm (see the annex).

5 Apparatus

The balance used shall be sensitive to 0,1 mg.

5.1 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 an electrically heated and thermostatically controlled aluminium block oven.

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

5.3 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.

5.4 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.

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

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

6 Preparation of sample

The coal used for the determination of moisture content is the analysis sample ground to pass a sieve of 212 µm 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 for at least 1 min, preferably by mechanical means.

7 Procedure

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 readjust 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 min. Close the absorption tube, disconnect, wipe and allow it to stand for 20 min 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).

While 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 to the nearest 0,1 mg 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), close, disconnect and weigh the absorption tube, following exactly the procedure described in the second paragraph above. Stop the flow of nitrogen while 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 min 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 while the loss in mass of the coal is determined (see note, clause 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 1 h is sufficient. For lignites and brown coals, a longer period is required, and the time of heating varies with different coals.

8 Blank determination

Determine the apparatus blank by carrying out the procedure exactly as described in clause 7, 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.

9 Expression of results

The moisture, M, in the coal as analysed, expressed as a percentage by mass, is given by the formula

$$\frac{(m_2 - m_3)}{m_1} \times 100$$

where

m_1 is the mass, in grams, of coal taken;

m_2 is the increase in mass, in grams, of the absorption tube during the test;

m_3 is the increase in mass, in grams, of the absorption tube during the blank determination.

The results (preferably the mean of duplicate determinations, see 10.1) shall be reported to the nearest 0,1 %.

10 Precision of determination

Moisture	Maximum acceptable differences between results	
	Same laboratory (Repeatability)	**Different laboratories** (Reproducibility)
	0,2 % absolute	(see 10.2)

10.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory, by the same operator, with the same apparatus, on representative portions weighed out at the same time from the same analysis sample, shall not differ by more than the above value.

10.2 Reproducibility

Since the results obtained will depend on the humidity conditions in the different laboratories, which will vary, it is not practical to quote a limiting value for reproducibility.

11 Test report

The test report shall include the following particulars:

a) identification of the product tested;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional;

f) the date of the test.

Annex

Nitrogen purification train

(Forms part of the Standard.)

It is essential that the nitrogen used for ventilating the minimum free space oven should be comparatively pure, since even the small residual amount of oxygen which is found in commercial nitrogen may cause oxidation and hence produce a low result in the moisture figure determined by the loss in mass on drying. A good purification train for nitrogen is therefore required; a suitable one is described here which is capable of dealing with at least 600 ml per minute at a pressure of a few millimetres mercury gauge. A convenient source of the gas is a cylinder of compressed nitrogen. The purification train consists of a quartz tube 500 mm long and 37 mm internal diameter, containing 1,2 kg of reduced copper in wire form. The purification tube is heated by a suitable furnace, which encloses the tube to a length of 380 mm and allows the part of the tube containing the copper wire filling to protrude approximately 80 mm at the exit end in order to produce a temperature drop in the gas stream. The reduced copper is heated to approximately 500 °C. The last traces of oxygen are removed by the very large surface area of the copper.

If it is necessary to reduce any copper oxide formed during the purification of the nitrogen, this can be done by passing hydrogen through the tube containing the copper, which is heated to 450 to 500 °C. During the reduction process the water formed is expelled to atmosphere. The tube should be purged with nitrogen before use, the reduced copper emptied from the tube and any agglomerated material broken down, and the tube refilled.

Alternatively, a commercial source of nitrogen may be employed in the absence of a purification train, provided the gas in the cylinder contains not more than 30 ppm of oxygen.

In either case, the purified nitrogen shall be passed through a column of magnesium perchlorate to remove traces of moisture which may be present.