International Standard

ISO 5068

Brown coals and lignites — Determination of moisture content — Indirect gravimetric method

Charbons bruns et lignites — Détermination de l'humidité — Méthode gravimétrique indirecte

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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 5068 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*, and was circulated to the member bodies in January 1982.

It has been approved by the member bodies of the following countries:

Australia	Egypt, Arab Rep. of	South Africa, Rep. of
Austria	Germany, F.R.	Spain
Belgium	India	Thailand
Bulgaria	Japan	Turkey
Canada	New Zealand	USSR
China	Poland	Yugoslavia
Czechoslovakia	Romania	

No member body expressed disapproval of the document.

Brown coals and lignites — Determination of moisture content — Indirect gravimetric method

0 Introduction

Brown coals and lignites are hygroscopic and therefore their moisture content varies with change of humidity of the atmosphere. Hence, the moisture content of the analysis sample is determined whenever test portions are weighed out for other analytical determinations, for example volatile matter, calorific value, carbon and hydrogen, etc.

1 Scope and field of application

This International Standard specifies methods for the determination of the moisture content of brown coals and lignites using an indirect gravimetric method. An alternative direct volumetric procedure is specified in ISO 1015.

This International Standard specifies two basic procedures :

   a) A single-stage method which can be used for determination of the total moisture content of the special moisture sample or of a moisture sample taken from a common sample.

   b) A two-stage method which can be used for determination of the residual moisture content and, if required, also the total moisture content of a special moisture sample or of a moisture sample taken from a common sample.

The moisture content of an analysis sample (see clause 0) is determined using a variation of the single-stage method.

2 References

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

ISO 5069, *Brown coals and lignites — Principles of sampling and sample preparation*

Part 1 : Sampling for determination of moisture content and for general analysis. [1]

Part 2 : Sample preparation for determination of moisture content and for general analysis. [1]

3 Principle

3.1 Single-stage method

A sample, prepared using a closed mill, is dried to constant mass at a tempeature of 105 to 110 °C in a nitrogen oven in an atmosphere of nitrogen free of moisture and oxygen, and the total moisture content calculated from the loss in mass of the sample.

3.2 Two-stage method

A sample is coarsely ground and is then allowed to dry, either in air at ambient temperature or at a higher temperature not exceeding 40 °C, in order to reach equilibrium with the atmosphere. (If the total moisture content of the sample is required, the loss in mass in this initial drying stage is determined.) The sample is then dried to constant mass in the conditions described in 3.1 and the residual moisture content calculated from the loss in mass of the sample during this final drying stage. (The total moisture content is calculated from the losses in mass during the two drying stages.)

3.3 Method for moisture content of an analysis sample

A test portion of the sample to be used for other determinations (see clause 0) is dried to constant mass in the conditions described in 3.1 and the moisture content is calculated from the loss in mass of the test portion. The moisture content is used in calculating the results of the other determinations on a dry basis.

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[1] At present at the stage of draft.

4 Apparatus and materials

4.1 Nitrogen oven, capable of being controlled at a temperature within the range 105 to 110 °C and with provision for passing a current of dry oxygen-free nitrogen through it at a rate sufficient to change the atmosphere 15 times per hour. A suitable oven is illustrated in the figure.

4.2 Drying cabinet, gas or electrically heated and temperature controlled to 35 ± 5 °C, with provision for mechanical venting.

4.3 Non-corrodible tray, of such dimensions that the total sample may be dried in a layer not exceeding 20 mm thick in one tray.

4.4 Weighing vessel, consisting of a shallow vessel, of silica or glass, with ground edges and fitted with ground-on cover, or of a non-corrodible, heat-resistant material with well-fitting lid. The diameter of the vessel shall be such that the mass of the coal layer does not exceed 0,15 g/cm² for 1 to 2 g of the test portion of an analytical sample or 0,30 g/cm² for 10 g of the sample in other cases.

4.5 Balance, of the required accuracy.

4.6 Desiccant, for use in the desiccator.

A suitable desiccant is dry magnesium perchlorate.

NOTE — Attention is drawn to the care needed in the handling and the disposal of magnesium perchlorate. National requirements must be observed.

4.7 Nitrogen, dry, with a maximum oxygen content of 30 ppm (see the annex).

5 Preparation of sample

5.1 Single-stage method

Take a minimum of 500 g of the special moisture sample or of the moisture sample taken from the common sample (see figures 1 and 2 of ISO 5069/2) and crush it in a closed mill until it passes a 3,15 mm sieve.

5.2 Two-stage method

Take a minimum of 500 g (see the note) of the special moisture sample or of the moisture sample taken from the common

sample (see figures 1 and 2 of ISO 5069/2) and coarsely crush it until it passes a 20 mm sieve. (If the total moisture content is required, weigh accurately the sample before crushing it.)

Dry the crushed sample in the non-corrodible tray (4.3) in air at ambient temperature to bring it into approximate equilibrium with the atmosphere. This process can be accelerated by placing the sample on a tray in the drying cabinet (4.2) at a temperature not exceeding 40 °C and not less than 30 °C. Allow a sample dried at this temperature to attain equilibrium with the atmosphere until the loss in mass of the sample over a 2 h period is less than 0,3 % of the original mass.

Crush the sample so dried to pass a 3,15 mm aperture sieve. (If the total moisture content of the sample is required, weigh accurately the sample and calculate the loss in mass W_{ex} by air drying).

NOTE — The sample mass in kilograms should be not less than 0,1 of the maximum grain size in millimetres for grain sizes between 3 and 20 mm and in any case not less than 500 g.

5.3 Method for moisture content of the analysis sample

Take a test portion of at least 150 g from the analysis sample.

NOTE — The analysis sample for determination of tar and benzene-soluble extract[*] should be below 1 mm in particle size and for other determinations below 0,212 mm.

6 Procedure

6.1 Single-stage method

Weigh, to the nearest 0,002 g, about 10 g of the sample into the clean, dry weighing vessel (4.4). Place the uncovered vessel in the drying oven (4.1), previously heated to 105 to 110 °C. Dry the sample at this temperature for exactly 60 min. Remove the vessel from the oven, replace the cover and, after a rapid cooling in air on a metal plate, transfer the vessel to a desiccator containing the desiccant (4.6). After attaining the balance-room temperature, i.e. approximately 20 min, re-weigh the vessel. To ensure complete drying, re-dry for further 30 min periods until the difference between two successive weighings of the sample is less than 0,01 g. For the calculation, use the higher loss in mass of these successive weighings.

6.2 Two-stage method

Follow the procedure specified in 6.1, using a portion of the sample prepared in accordance with 5.2.

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[*] It is proposed to replace benzene by toluene in ISO 975.

Dimensions in millimetres

A-A

Figure — Suitable nitrogen oven

6.3 Method for moisture content of an analysis sample

Weigh, to the nearest 0,002 g, 1 to 2 g of the sample (5.3) into the clean, dry weighing vessel (4.4) and place it in the drying oven (4.1), preheated to a temperature of 105 to 110 °C and dry at this temperature for 60 min. Remove the vessel from the oven, replace the cover and, after a rapid cooling in air on a metal plate, transfer the vessel to a desiccator containing the desiccant (4.6) and after attaining the balance-room temperature, re-weigh with an accuracy of 0,002 g. To ensure complete drying, re-dry for 30 min periods until the difference between two successive weighings of the sample is less than 0,001 g. For the calculation, use the higher loss in mass of these successive weighings.

7 Expression of results

Calculate the total moisture content, W_t^r, determined by the single-stage method, residual moisture content, W_h, when predrying the sample using the two-stage method and moisture content in the analytical sample, W^a, as percentages, using the formula

$$W = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the coal before drying;

m_1 is the loss of mass, in grams, of the coal after drying.

NOTE — When using the two-stage method, the total moisture content in the "as delivered state", W_t^r, is calculated, as a percentage, using the formula

$$W_t^r = W_{ex} + W_h \times \left(1 - \frac{W_{ex}}{100}\right)$$

where

W_{ex} is the loss of mass of the sample below 20 mm by air-drying, expressed as a percentage by mass;

W_h is the residual moisture in the air-dried sample below 3 mm, expressed as a percentage by mass.

Take as the final result of the test the arithmetic mean of the results of two or three determinations (see 8.1), provided that the requirements for repeatability are satisfied. Report the results of the test to the nearest 0,1 %.

8 Precision of method

8.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory, by the same operator, with the same apparatus, on two test portions of the same sample, shall not differ by more than the values given in the table.

If the difference is greater than that given in the table, a third determination shall be carried out and the arithmetic mean of the two nearest values within the acceptable difference shall be taken as the result of the test. If the value of the third determination is within the acceptable difference to each of the first two determinations, the arithmetic mean of all the three determinations shall be taken as the result of the test.

8.2 Reproducibility

The results of the test carried out in two laboratories, on representative test portions of the same sample shall be regarded as suspect if they differ by more than the values given in the table.

9 Test report

The test report shall include the following particulars :

a) an identification of the product tested;

b) the reference of the method used;

c) the result 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.

Table — Maximum acceptable differences between results

Moisture content			Maximum acceptable differences between results	
			Same laboratory (Repeatability)	Different laboratories (Reproducibility)
Less than 10 %	Residual moisture,	W_h	0,3 % absolute	—
	Moisture in the analysis sample,	W^a	0,2 % absolute	—
10 % and over	Total moisture,	W_t^r	3 % of result (one-stage method)	5 % of result
	Residual moisture,	W_h	0,4 % absolute	—
	Moisture in the analysis sample,	W^a	2,0 % of result	—