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Hard coal — Determination of total moisture

Houille — Détermination de l'humidité totale

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 589 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 589:2003), which has been technically revised.

Introduction

Moisture is an important parameter in respect of coal quality.

The moisture content of coal is not an absolute value and conditions for its determination have to be standardized. It is expected that the results given by the different methods specified here should be comparable within the limits of the tolerance quoted.

It is always necessary that the determination of the total moisture content of hard coals be considered in close connection with sampling. Therefore, this International Standard has been prepared in close relationship with the ISO standards for mechanical sampling ISO 13909 (all parts) and manual sampling ISO 18283.

A major problem with the preparation of test samples for the determination of moisture is the risk of bias due to inadvertent loss of moisture. This is dependent on the tightness of the sealing of sampling containers, the level of moisture content in the sample, the ambient conditions, the type of coal and the reduction and division procedures used. This is described in detail in ISO 13909-4 or ISO 18283.

Depending on the mass, the nominal top size and the facilities available where samples are taken, it is possible to dry the sample directly after sampling (air-drying), then to reduce the particle size and prepare a test sample for determination of moisture in the air-dried sample. Alternatively, the whole sample may be transported to the laboratory and the total moisture determined.

Hard coal — Determination of total moisture

1 Scope

This International Standard describes two methods for determination of the total moisture content of hard coals, a two-stage method and a single-stage method. For either method there is a choice between drying in air and drying in a nitrogen atmosphere. Depending on the coal rank, there may be systematic differences between the results obtained by drying in the different atmospheres on subsamples of the same sample. Drying in a nitrogen atmosphere is suitable for all hard coals, while drying in air is only suitable for hard coals not susceptible to oxidation.

NOTE The term “not susceptible to oxidation” cannot be defined easily. Usually, high-rank coals such as anthracites are not oxidized under the conditions described in this International Standard. For all other types of coal, this has to be verified by experiments.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 11722, *Solid mineral fuels — Hard Coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

ISO 13909-1, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

ISO 13909-3, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*

ISO 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 18283:2006, *Hard coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 apply.

4 Principle

4.1 Method A — Two-stage methods

4.1.1 Method A 1 — Drying under nitrogen in second stage

The sample is dried in air at ambient temperatures or at elevated temperatures not exceeding 40 °C (first or free-moisture stage) and the loss in mass recorded. The air-dried sample is crushed to 2,8 mm nominal top size and subsamples are dried at 105 °C to 110 °C in a nitrogen-flushed oven (second or residual moisture stage).

NOTE Residual moisture is often called moisture in the air-dried sample.

Provided that the result obtained for the determination of moisture in the analysis sample in accordance with ISO 11722 can be shown to give the same result as that for the second-stage moisture determination, the former may be used.

The moisture is calculated from the loss in mass at each of the two stages.

4.1.2 Method A 2 — Drying in air

The sample is dried in air at ambient temperatures or at elevated temperatures not exceeding 40 °C (first or free-moisture stage) and the loss in mass recorded. The air-dried sample is crushed to 2,8 mm nominal top size and subsamples are dried in air at 105 °C to 110 °C (second or residual moisture stage).

The moisture is calculated from the loss in mass at each of the two stages.

NOTE This method is suitable only for hard coals not susceptible to oxidation.

4.2 Method B — Single-stage methods

4.2.1 Method B 1 — Drying under nitrogen

The sample is crushed to a nominal top size of either 11,2 mm or, alternatively, 10 mm. A subsample is dried in a nitrogen-flushed oven at a temperature of 105 °C to 110 °C. The moisture is calculated from the loss in mass.

4.2.2 Method B 2 — Drying in air

The sample is crushed to a nominal top size of either 11,2 mm or, alternatively, 10 mm. A subsample is dried in air at a temperature of 105 °C to 110 °C. The moisture is calculated from the loss in mass.

NOTE This method is suitable only for hard coals not susceptible to oxidation.

5 Reagent

Nitrogen, moisture-free, having an oxygen content of less than 30 µl/l.

NOTE Commercially available nitrogen with a water content of less than 5 µl/l does not require further drying.

6 Apparatus

6.1 Method A

6.1.1 Oven, for first-stage moisture determination, capable of being controlled at a temperature of 30 °C to 40 °C, with a sufficiently rapid rate of atmosphere change (e.g. 5 times per hour). The air velocity shall be such that the sample particles are not dislodged from their tray.

6.1.2 Nitrogen-flushed oven, for second-stage moisture determination, capable of being controlled at a temperature of 105 °C to 110 °C, with the additional provision for passing a current of dry nitrogen through it at a flow rate about 15 times the oven volume per hour. The gas velocity shall be such that the sample particles are not dislodged from their dish.

6.1.3 Oven, for second-stage moisture determination, capable of being controlled at a temperature of 105 °C to 110 °C, with a sufficiently rapid rate of atmosphere change (e.g. 5 times per hour). The air velocity shall be such that the sample particles are not dislodged from their dish.

6.2 Method B

6.2.1 Nitrogen-flushed oven, for method B 1, capable of being controlled at a temperature of 105 °C to 110 °C, with the additional provision for passing a current of dry nitrogen through it at a flow rate about 15 times the oven volume per hour. The nitrogen velocity shall be such that the sample particles are not dislodged from their tray.

6.2.2 Oven, for method B 2, capable of being controlled at a temperature of 105 °C to 110 °C and with a sufficiently rapid rate of atmosphere change (e.g. 5 times per hour). The air velocity shall be such that the sample particles are not dislodged from their tray.

6.3 Methods A and B

6.3.1 Weighing tray, made of heat- and corrosion-resistant material of such dimensions that the loading of the coal layer does not exceed 1 g/cm^2.

6.3.2 Weighing dishes, shallow vessels of glass, silica or corrosion-resistant metal with well-fitting covers of such a size that the loading of the coal layer does not exceed 0,3 g/cm^2.

6.3.3 Apparatus for size reduction (to 11,2 mm or 10,0 mm and 2,8 mm), without significant loss in moisture content.

6.3.4 Balance, capable of weighing to 0,1 g.

6.3.5 Analytical balance, capable of weighing to the nearest 1 mg.

6.3.6 Sample divider, e.g. riffle divider.

7 Sample

7.1 General

Depending on the mass, the nominal top size and the facilities available where samples are taken, it is possible to dry the sample (air-drying) directly after sampling, then to reduce the particle size and prepare a test sample for determination of moisture in the air-dried sample (“on-site treatment”). Alternatively, the whole sample may be transported to the laboratory and the moisture determined.

7.2 Sampling and sample preparation

Sampling shall be made in accordance with ISO 13909-1, ISO 13909-2 and ISO 13909-3 for mechanical sampling, or ISO 18283 for manual sampling. On-site sample preparation shall be carried out in accordance with ISO 13909-4 for mechanical sampling, or ISO 18283 for manual sampling.

NOTE If convenient, samples for moisture determination may be extracted from a common sample for both moisture and general analysis; see ISO 13909-4 or ISO 18283.

7.3 Precautions against loss of moisture

One of the main difficulties in determining total moisture is that of minimizing changes in the moisture content of the sample while preparing the final sample. Every precaution shall be taken to minimize change of moisture due to the use of unsuitable containers and by evaporation during handling, particularly if the coal is extremely wet. All moisture samples shall be kept in sealed containers in a cool place before and after preparation, as well as during any interval between stages of sample preparation.

Care needs to be taken to minimize the change of moisture during particle size reduction, by using equipment in which there is no appreciable heating and by reducing to a minimum the amount of air passing through the mill. Machines that crush are preferable to those that grind, as the latter have a greater tendency to generate heat.

Care should also be taken to minimize change of moisture when carrying out sample division, and all such operations should be carried out as quickly as possible.

7.4 Moisture determination with on-site air-drying — Method A only

If the sample has already been air-dried directly after sampling (in accordance with ISO 13909-4, alternatively ISO 18283), it can then be crushed to 2,8 mm nominal top size. The amount of the crushed sample shall not be less than 650 g and step 8.1.1 can be skipped in the laboratory.

It is necessary to check that the air-dried sample does not take up or lose moisture when it is transported to the laboratory.

7.5 Moisture determination without on-site air-drying — Methods A and B

Samples for the determination of moisture shall be received in air-tight containers. The sample mass shall not be less than the minimum mass stated in ISO 13909-4:2001, Table 1 and in ISO 18283:2006, Table 3 (which are the same).

If the coal is so wet that water separates from the coal in the sample container, the whole of the sample and the container shall be air-dried until this condition no longer applies and the loss in mass is recorded.

8 Procedure

8.1 Methods A 1 and A 2 — Two-stage methods

8.1.1 First-stage moisture — Free moisture

Weigh a dry, empty tray (6.3.1), transfer the sample (7.5) to the tray and spread evenly, so that the loading of the coal layer does not exceed 1 g/cm^2. Weigh the sample to the nearest 0,1 g. If the loading is too high for one tray, use two or more.

Weigh the tray(s) plus sample and place it/them in the drying oven at ambient temperature. Remove the trays when a constant mass is reached. To shorten the time required for air drying, the drying oven may be heated to a maximum of 40 °C. In this case the coal sample shall re-equilibrate to ambient temperature before re-weighing to the nearest 0,1 g.

NOTE Constancy in mass is defined as a change in mass not exceeding 0,2 % absolute during a further period of not less than 25 % of the initial drying period.

Drying of lower-rank hard coal should not be excessive because of oxidation; the drying time should not exceed 18 h.

The first-stage moisture (free moisture), M_1, of the sample, expressed as a percentage by mass, is given by Equation 1:

$$M_1 = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where

m_1 is the mass of the empty tray(s), expressed in grams;

m_2 is the mass of the tray(s) plus sample before drying, expressed in grams;

m_3 is the mass of the tray(s) plus sample after drying, expressed in grams.

8.1.2 Method A 1 — Second-stage moisture (residual moisture) under nitrogen

Immediately after air-drying, the sample is crushed by a suitable apparatus to 2,8 mm nominal top size. At least two test portions for the determination of second-stage moisture content are taken as quickly as possible to avoid losses.

If an air-dried sample has been prepared on site after sampling, check that it has not taken up or lost moisture during transport to the laboratory by re-weighing and correct accordingly. This sample is also reduced to 2,8 mm nominal top size.

Weigh, to the nearest 1 mg, a clean dry empty weighing dish with cover (6.3.2). Take (10 ± 1) g of sample and spread evenly into the sample dish. Weigh the uncovered dish plus its cover to the nearest 1 mg and place the uncovered dish plus its cover in the oven, preheated to between 105 °C and 110 °C.

Dry at 105 °C to 110 °C while flushing the oven with nitrogen at a flow rate of about 15 times the oven volume per hour.

When the mass is constant, replace the cover (if possible while the dish is still in the nitrogen-flushed oven, otherwise immediately after removal from the oven) and remove the covered dish. Cool to ambient temperature and re-weigh to the nearest 1 mg.

NOTE Constancy in mass is defined as a change in mass not exceeding 0,2 % absolute during a further period of not less than 25 % of the initial drying period.

The second-stage moisture content, M_2, of the sample, expressed as a percentage by mass, is given by Equation 2:

$$M_2 = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (2)$$

where

m_1 is the mass of the empty weighing dish with cover, expressed in grams;

m_2 is the mass of the weighing dish with cover plus sample before drying, expressed in grams;

m_3 is the mass of the weighing dish with cover plus sample after drying, expressed in grams.

The result for the second-stage moisture is given as the mean of duplicate determinations.

8.1.3 Method A 2 — Second-stage moisture (residual moisture) in air

The procedure is as described in 8.1.2 with the exception that air instead of nitrogen is used at a flow rate about five times the oven volume per hour.

8.1.4 Calculation and expression of results

The total moisture content, M_T, is calculated from the first-stage moisture (free moisture) content, M_1, and the second-stage moisture, M_2 (residual moisture), using Equation (3):

$$M_T = M_1 + \frac{M_2 \times (100 - M_1)}{100} \quad (3)$$

The result shall be reported to the nearest 0,1 %.

If it has been proven that the second-stage moisture content, M_2, equals the value of the moisture in the general analysis test sample, M_A, the total moisture, M_T, can be calculated from the first-stage moisture, M_1, and the moisture in the general analysis test sample, M_A, using Equation (4):

$$M_T = M_1 + \frac{M_A \times (100 - M_1)}{100} \quad (4)$$

8.2 Method B — Single-stage method

8.2.1 Method B 1 — Drying under nitrogen

Crush the sample to a nominal top size of either 11,2 mm or, alternatively, 10 mm. Extract at least two portions of minimum 600 g each from the crushed sample.

Weigh a dry, empty tray to the nearest 0,1 g, transfer the sample to the tray and spread evenly such that the loading of the coal layer does not exceed 1 g/cm^2. Weigh the sample to the nearest 0,1 g. If the loading is too high for one tray, use two or more.

Place the loaded tray(s) in the heated oven, set to 105 °C to 110 °C while flushing the oven with nitrogen at a flow rate of about 15 times the oven volume per hour. Heat the sample until mass is constant. Weigh as soon as possible (within 5 min) and while still hot to avoid adsorption of moisture during cooling.

NOTE Constancy in mass is defined as a change in mass not exceeding 0,2 % absolute during a further period of not less than 25 % of the initial drying period.

8.2.2 Method B 2 — Drying in air

The procedure is the same as described in 8.2.1 with the exception that air instead of nitrogen is used at a flow rate about five times the oven volume per hour.

8.2.3 Calculation and expression of results

The total moisture, M_T, in the sample, expressed as a percentage of mass, is given by Equation (5):

$$M_T = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (5)$$

where

m_1 is the mass of the empty tray(s), expressed in grams;

m_2 is the mass of the tray(s) plus sample before drying, expressed in grams;

m_3 is the mass of the tray(s) plus sample after drying, expressed in grams.

The result for total moisture is given as the mean of duplicate determinations and shall be reported to the nearest 0,1 %.

9 Precision

9.1 Repeatability limit

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 sample, should not differ by more than the values given in the second column of Table 1.

9.2 Reproducibility critical difference

Since the humidity conditions in different laboratories vary, it is not possible to quote a limiting value for reproducibility critical difference for first-stage moisture. For total moisture, the results from two different laboratories should not differ by more than the values given in the third column of Table 1.

Table 1 — Precision

Property	Repeatability limit %	Reproducibility critical difference %
Second-stage moisture	0,3	Not applicable
Total moisture	0,5	1,5

10 Test report

The test report shall include the following:

a) reference to this International Standard and its year of publication, i.e. ISO 589:2008;

b) identification of the sample tested;

c) results of the determination.

Bibliography

[1] ISO 13909-5, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*

[2] ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

[3] ISO 13909-7, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*

[4] ISO 13909-8, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*

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