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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 third edition cancels and replaces the second edition (ISO 589:1981), 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. Results given by the different methods specified here should be comparable within the limits of the tolerance quoted.

The determination of the total moisture content of hard coals is always to be considered in close connection with sampling. Therefore this International Standard has been prepared in close relationship with the series ISO 13909, *Hard coal and coke — Mechanical sampling.*

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.

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 the determination of the total moisture content of hard coals. Depending on the coal rank, there may be systematic differences between the results obtained by applying different methods on subsamples of the same sample. Methods using a nitrogen atmosphere are suitable for all hard coals; methods with drying in air are only suitable for hard coals not susceptible to oxidation.

NOTE The term "not susceptible to oxidation" cannot be defined easily. Usually, coals very high in rank are not oxidized under the conditions described in this 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 1988, *Hard coal — Sampling*

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

ISO 13909-1:2001, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2:2001, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

ISO 13909-3:2001, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*

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

ISO 13909-8:2001, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*

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

The sample is dried in air at ambient temperatures or at elevated temperatures not exceeding 40 °C (first stage or free moisture) 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 - 110 °C in a nitrogen-flushed oven (second stage or residual moisture).

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 stage or free moisture) and the loss in mass in recorded. The air-dried sample is crushed to 2,8 mm nominal top size and subsamples are dried at 105 - 110 °C (second stage or residual moisture).

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

NOTE This method is only suitable 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 11,2 mm nominal top size. A subsample is dried in a nitrogen-flushed oven at a temperature of 105 - 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 11,2 mm nominal top size. A subsample is dried at a temperature of 105 - 110 °C. The moisture is calculated from the loss in mass.

NOTE This method is only suitable 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

Methods A

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

6.2 Nitrogen-flushed oven (second-stage moisture), capable of being controlled at a temperature of 105 - 110 °C and 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

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

Methods B

6.4 Nitrogen-flushed oven (B 1), capable of being controlled at a temperature of 105 - 110 °C and 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.

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

Methods A and B

6.6 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.

6.7 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.

6.8 Apparatus for size reduction (to 11,2 mm and 2,8 mm, respectively) without significant loss in moisture content.

6.9 Balance, capable of weighing to the next 0,1 g.

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

6.11 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 carried out according to ISO 13909, Parts 1-3 or ISO 1988, respectively. On-site sample preparation shall be carried out according to ISO 13909-4.

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

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 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 treatment (method A only)

The sample has already been air-dried directly after sampling (according to ISO 13909-4). It is then crushed to 2,8 mm nominal top size; the sample shall not be less than 650 g. The step described in 8.1.1 can be skipped in the laboratory.

It has to be checked that the air-dried sample does not take up or lose moisture when it is transported to the laboratory (see 7.3).

7.5 Moisture determination without on-site treatment (methods A and B)

Samples for the determination of moisture shall be received in airtight containers. The sample mass shall not be less than the minimum mass stated in ISO 13909-4:2001, Table 1.

If the mass of the sample is so large that transport and subsequent drying is impractible, the samples shall be crushed and divided at the place of sampling. Crushing shall be kept to the minimum necessary to allow division to a manageable mass. The absence of bias due to these procedures shall be checked using the procedures given in ISO 13909-8, by comparison with the method of drying samples without reduction.

NOTE For practical reasons, it may be useful to crush the sample to, for example, 11,2 mm nominal top size, resulting in a minimum mass for moisture determination of 2 500 g. This sample can be transported to the laboratory. However, this reduction needs to be tested for relevant bias using the procedures given in ISO 13909-8, by comparison with the method of drying samples without reduction.

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 (6.6), transfer the sample (sample mass see Table 1 in ISO 13909-4:2001) 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 another or more.

Weigh the tray(s) plus sample and place it (them) in the drying oven at ambient temperature. Remove when constancy in 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 reweighing to the nearest 0,1 g.

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

Drying of low-rank coal should not last too long because of oxidation; the drying time should not exceed 18 h.

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

$$M_1 = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

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

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

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

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 reweighing 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 (6.7). 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 at 105 - 110 °C.

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

When constant in mass, 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 reweigh to the nearest 1 mg.

NOTE Constancy in mass is defined as a change in mass not exceeding 0,2 % of the total loss in mass 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 the equation:

$$M_2 = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

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

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

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

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

Use the same procedure as described in 8.1.2 with the exception of using air instead of nitrogen at a flow-rate about 5 times the oven volume per hour.