INTERNATIONAL STANDARD

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Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample

Combustibles mineraux solides — Coke — Détermination de l'humidité de l'échantillon pour analyse

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

This second edition cancels and replaces the first edition (ISO 687:1974), which has been technically revised.

Introduction

The determination of the moisture in the general analysis test sample is required to correct the results of certain analytical determinations, e.g. volatile matter and hydrogen, for the effect of water in the determination and to allow all determinations to be corrected to a dry basis.

Since coke is hygroscopic, its moisture will vary with a change in humidity of the atmosphere, and the moisture in the general analysis test sample should therefore be determined whenever portions are weighed out for other analytical determinations. If test portions for several analytical determinations are weighed out at the same time, a single simultaneous moisture determination will suffice to correct those analyses.

Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample

1 Scope

This International Standard specifies a method for determining the moisture in the general analysis test sample of coke. It can be used for the determination of moisture in blast-furnace coke, foundry-coke and other high-temperature carbonization products.

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 1213-2:1992, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2:1992 apply.

4 Principle

A known mass of the coke is heated in air at 120 °C to 200 °C and maintained at this temperature until constant mass is obtained. The moisture content is calculated from the loss in mass of the coke. Coke is not liable to oxidation under the conditions stated.

5 Apparatus

5.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.2 Oven, capable of being controlled at a temperature of 120 °C to 200 °C and provided with a means to allow the flow of air or nitrogen.

5.3 Weighing dish, shallow, of glass or of corrosion-resistant metal, with well-fitting covers, of such a size that the coke layer does not exceed 0,20 g/cm^3.

5.4 Cooling vessel, e.g. desiccator, without desiccant, containing a porcelain or metal plate, preferably of aluminium or copper. The vessel may be provided with a means to pass air or nitrogen through it during the cooling period.

6 Preparation of the test sample

The coke used for the determination of moisture content is the general analysis test sample (see ISO 1213-2:1992). Ensure that the moisture content of the sample is in equilibrium with the laboratory atmosphere, exposing it, if necessary, in a thin layer for the minimum time required to achieve equilibrium.

Before commencing the determination, thoroughly mix the equilibrated test sample for at least 1 min, preferably by mechanical means.

7 Procedure

Weigh a clean, dry, empty weighing dish with its cover to the nearest 0,1 mg. Add (1 ± 0,1) g of the coke sample in an even layer and reweigh. Heat the uncovered dish in the oven at 120 °C to 200 °C.

When the drying period is complete, remove the dish with the dried sample from the oven and replace the cover immediately. If the size of the oven allows, replace the cover while the dish is still in the oven. Allow the dish to cool on a thick metal plate for 10 min. At the end of the 10 min cooling period, transfer the dish to a cooling vessel (5.4) and allow to cool to room temperature. As soon as room temperature is reached, reweigh to the nearest 0,1 mg.

NOTE 1 If a cooling vessel with air or nitrogen flow is used, the dish can be transferred directly without cooling on a metal plate.

If there is any doubt that drying is complete, reheat at 120 °C to 200 °C for further 30 min periods until any change in mass does not exceed 1 mg.

For a particular oven, the times required to ensure constancy in mass shall be verified by experiments.

NOTE 2 Heating for 4 h is normally sufficient.

The time taken for the determination can be considerably shortened if drying is carried out at a temperature of 320 °C in a nitrogen atmosphere, when heating for 1 h will usually suffice. For this procedure, a minimum-free-space oven may be used.

If appropriate, the drying can be done at a lower temperature, e.g. 105 °C to 110 °C as for hard coal. The times required to ensure constancy in mass have to be verified by experiments.

8 Expression of results

The moisture in the coke as analysed, M_A, expressed as a percentage by mass, is given by the following equation

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

where

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

m_2 is the mass, in grams, of the dish plus cover plus coke before heating;

m_3 is the mass, in grams, of the dish plus cover plus coke after heating.

Report the result, as the mean of duplicate determinations, to the nearest 0,1 %.

9 Precision

9.1 Repeatability limit

The results of duplicate determinations (carried out over a short period of time, but not simultaneously) in the same laboratory, by the same operator, with the same apparatus on two representative portions taken from the same analysis sample, should not differ by more than the values shown in Table 1.

9.2 Reproducibility critical difference

Since the humidity conditions in different laboratories vary, it is not practical to quote a limiting value for reproducibility.

Table 1 — Precision of moisture determination

Maximum acceptable differences between results	
Repeatability limit	**Reproducibility critical difference**
0,2 % absolute	See 9.2

10 Test report

The test report shall include the following information:

a) a reference to this International Standard and its year of publication, i.e. ISO 687:2004;

b) the identification of the sample tested;

c) the results of the determination;

d) the date of the determination.