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Coke — Determination of moisture in the general analysis test sample

Coke — Détermination de l'humidité de l'échantillon pour analyse

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Foreword

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This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 687:2010), which has been technically revised.

The main change is as follows:

— adding the optional use of nitrogen and a minimum free space oven.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The determination of 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 test portions are taken for other analytical determinations. If test portions for several analytical determinations are taken at the same time, a single simultaneous moisture determination will suffice to correct those analyses.

Coke — Determination of moisture in the general analysis test sample

1 Scope

This document 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

A known mass of the coke is heated in air or nitrogen at a pre-set temperature between (optional 105 °C) 120 °C to 200 °C (optional 320 °C) and maintained at this temperature until constant mass is obtained. The moisture is calculated from the loss in mass of the coke. Coke is not liable to oxidation under the conditions stated.

5 Reagents

5.1 Nitrogen (optional), moisture-free, having a volume fraction of oxygen of less than 30 µl/l.

Commercially available nitrogen with moisture of less than 5 µl/l does not require further drying.

6 Apparatus

6.1 Analytical balance, capable of reading to the nearest 0,1 mg.

6.2 Oven, capable of being controlled at a temperature of 120 °C (optional 105 °C) to 200 °C (optional 320 °C) and with a means to allow flow of air or nitrogen.

6.3 (optional) **Oven**, capable of being controlled at a temperature of 120 °C (optional 105 °C) to 200 °C (optional 320 °C) and with provision for nitrogen to pass through it at a flow-rate of approximately 15 times the oven volume per hour and of lowest practical volume, i.e. minimum free space.

NOTE An example of a suitable oven is given in Annex A.

6.4 **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^2.

6.5 **Cooling ve**ssel, e.g. desiccator, without desiccant, containing a porcelain or a metal plate. The vessel may be provided with the means to pass air or nitrogen through during the cooling period.

7 Preparation of the test sample

The coke used for the determination of moisture is the general analysis test sample (see ISO 1213-2). Ensure that the moisture 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.

8 Procedure

Determine the mass of a clean dry empty dish (6.4) with its cover to the nearest 0,1 mg by weighing on an analytical balance (6.1). Add (1 ± 0,1) g of the coke sample in an even layer and reweigh to the nearest 0,1 mg.

Heat the uncovered dish in the oven (6.2) in air or nitrogen at a pre-set temperature between 120 °C to 200 °C.

If a minimum free space oven (6.3) with a nitrogen atmosphere is used, heating is done while passing nitrogen through at approximately 15 times the oven volume per hour.

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 (6,5) and allow it to cool to room temperature. As soon as room temperature is reached, determine the mass of dish, cover and sample by weighing on an analytical balance (6.1) 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 between successive determinations 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 pre-set 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 lower temperature, e.g. 105 °C to 110 °C as for hard coal. The times required shall be verified by experiments to ensure constancy in mass.

9 Expression of results

The moisture mass fraction in the coke as analysed, $w_{H20.ad}$, expressed as a percentage, is given by Formula (1):

$$w_{H20.ad} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

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;

100 is the conversion factor from dimensionless fraction to per cent, in %.

Report the moisture mass fraction, as the mean of duplicate determinations, to the nearest 0,1 %.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same general analysis sample, should not differ by more than the value given in Table 1.

10.2 Reproducibility limit

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 limit**
0,2 % absolute	See 10.2

11 Test report

The rest report shall include the following information:

a) a reference to this document, i.e. ISO 687:2024;

b) the identification of the sample tested;

c) the results of the determination.

d) the date of the determination.