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

*Combustibles minéraux solides — Houille — Détermination de l'humidité de
l'échantillon pour analyse par dessiccation en atmosphère d'azote*



Foreword

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International Standard ISO 11722 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

Annex A of this International Standard is for information only.

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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 dry basis.

Since hard coal is hygroscopic, its moisture will vary with change of 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.

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

1 Scope

This International Standard specifies a method for determining the moisture in the general analysis test sample of hard coal by drying in nitrogen.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, such documents do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 1213-2 apply.

4 Principle

A known mass of the coal is heated in a stream of nitrogen at a temperature between 105 °C and 110 °C and maintained at this temperature until it is constant in mass. The moisture content is calculated from the loss in mass of the coal.

5 Reagent

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

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

6 Apparatus

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

6.2 Oven, capable of being controlled at a temperature of 105 °C to 110 °C and with provision for the 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.

6.3 Weighing dish, shallow, of glass or of corrosion-resistant metal, with well-fitting covers, of such a size that the coal layer does not exceed 0,20 g/cm².

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

7 Preparation of the test sample

The coal used for the determination of mass fraction moisture is the general analysis test sample (see 3.67 of ISO 1213-2:1992). Ensure that the mass fraction moisture in 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

Weigh, on an analytical balance (6.1), a clean, dry, empty weighing dish (6.3) with its cover to the nearest 0,1 mg. Add 1 g ± 0,1 g of the coal sample in an even layer and reweigh. Heat the uncovered dish in the oven (6.2) at 105 °C – 110 °C for at least 60 min while passing nitrogen through at approximately 15 times the oven volume per hour. Do not open the oven door during the drying period.

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 this, 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 (6.4) and allow to cool to room temperature. As soon as room temperature is reached, reweigh to the nearest 0,1 mg.

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

If there is any doubt that drying is not complete, reheat at 105 °C – 110 °C for further 30 min period until any change in mass does not exceed 1 mg.

9 Expression of results

The mass fraction of moisture in the hard coal as analysed, ω_M , in percent, is given by the following formula

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

where

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

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

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

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

10 Precision

10.1 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.

10.2 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

Mass fraction of moisture %	Maximum acceptable differences between results Repeatability limit
Less than 5	0,10
Equal to or more than 5	0,15

11 Test report

The test report shall include the following information:

- reference to this International Standard and its year of publication, i.e. ISO 11722:1999;
- the identification of the sample tested;
- the results of the determination;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional;
- the date of the test.

Annex A

(informative)

Example of a suitable oven

An example of a suitable oven for the determination of moisture in the general analysis test sample is given in figure A.1. It is a so-called "minimum free space oven" which minimizes the amount of nitrogen needed.

However, any other type of oven which meets the criteria given in 6.2 will suffice for this International Standard.

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