
**Solid mineral fuels — Determination of total
carbon, hydrogen and nitrogen —
Instrumental methods**

*Combustibles minéraux solides — Dosage du carbone, de l'hydrogène et
de l'azote total — Méthodes instrumentales*



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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 3.

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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years with a view to deciding whether it should be confirmed for a further three years, revised to become an International Standard, or withdrawn. In the case of a confirmed ISO/PAS or ISO/TS, it is reviewed again after six years at which time it has to be either transposed into an International Standard or withdrawn.

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

ISO/TS 12902 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

Introduction

Instrumental methods for the analysis of carbon, hydrogen and nitrogen are now in widespread and regular use, often in preference to formerly developed chemical methods for which International Standards exist. These International Standards and/or Certified Reference Materials usually provide the data for the calibration of such instrumental methods. It was considered that the procedures adopted in these instrumental methods should be standardized as far as possible and at this stage would be best presented as a Technical Specification while further data comparing instrumental and chemical methods is gathered and assessed.

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Solid mineral fuels — Determination of total carbon, hydrogen and nitrogen — Instrumental methods

1 Scope

This Technical Specification specifies the procedures to be followed when determining the mass fractions of total carbon (including that present as carbonates), total hydrogen (including that present as water) and nitrogen in hard coal, brown coal and lignite, and coke by instrumental methods.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Technical Specification. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Technical Specification are encouraged to investigate the possibility of applying the most recent editions of the normative documents 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 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method*

ISO 687:1974, *Coke — Determination of moisture in the analysis sample*

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method*

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases*

ISO 1988:1975, *Hard coal — Sampling*

ISO 2309:1980, *Coke — Sampling*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*

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 — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-5:2001, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*

ISO 13909-6:2001, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

ISO 13909-7:2001, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*

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

3 Principle

A known mass of the sample is burnt in oxygen, or in an oxygen/carrier gas mixture, under conditions such that it is converted into ash and gaseous products of combustion, which consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or oxides of nitrogen, oxides and oxyacids of sulfur and hydrogen halides. The products of combustion are treated to ensure that any hydrogen associated with sulfur or halide products of combustion is liberated as water vapour, oxides of nitrogen are reduced to elemental nitrogen, and those products of combustion which would interfere with the subsequent gas-analysis procedures are removed. The carbon dioxide, water vapour and nitrogen mass fractions of the gas stream are then determined quantitatively by appropriate instrumental gas-analysis procedures.

4 Reagents

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity.

4.1 Carrier gas

Helium or other suitable gas as specified by the instrument manufacturer.

4.2 Oxygen

As specified by the instrument manufacturer.

4.3 Additional reagents

Types and qualities as specified by the instrument manufacturer.

4.4 Calibration standards

Pure organic substances such as sulfanilamide and benzylisothiourea.

4.5 Certified reference materials (CRM)

Coal or coke, issued by an internationally recognized authority.

5 Apparatus

No specific designs of systems are presented here because there is a range of components and configurations available which can be used to carry out the test method satisfactorily.

The apparatus shall, however, meet the following functional requirements.

- a) The conditions of combustion of the sample shall be such that all of the carbon (including that in mineral carbonates), the hydrogen (including that in the water of constitution of the minerals), and the nitrogen present, shall be converted into carbon dioxide, water vapour (except for hydrogen associated with oxyacids of sulfur and volatile halides), and gaseous nitrogen and/or oxides of nitrogen, respectively.
- b) The combustion gases, or a representative aliquot, shall be treated to remove and/or separate out any components which would subsequently interfere with the detection and measurement of the carbon dioxide, water vapour or nitrogen in the gas stream.

- c) Hydrogen present as hydrogen halides or sulfur oxyacids shall be liberated, as water vapour, into the gas stream prior to determination of water vapour content.
- d) Any nitrogen oxides produced by the combustion process shall be reduced to nitrogen prior to presentation to the detection system.
- e) The detection systems shall provide responses, that correlate directly with the concentrations of the combustion gases, over the full range applicable and preferably in a linear manner.
- f) If a non-linear response is provided by a detection system, it shall include provisions for evaluating that response in a manner which correlates accurately with the concentration of the combustion gas.
- g) It shall include a means of displaying the detector responses or of calculating and presenting the concentrations of carbon, hydrogen and nitrogen in the sample following the input of other appropriate data as necessary.

NOTE The provisions for the evaluation of the detector response and for the performance of any subsequent calculations required may be integral with the instrument or provided by appropriate auxiliary systems.

6 Preparation of the test sample

The test sample is the general analysis test sample prepared in accordance with ISO 1988, ISO 2309, ISO 5069-2, or the appropriate part of the ISO 13909 series. If appropriate to the requirements of the test instrument, further grind the test sample to pass through a sieve of 75 μm . 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. In the case of a test sample ground to pass through a 75 μm sieve, exposure to the laboratory atmosphere is essential to re-establish moisture equilibrium disturbed by the grinding process.

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

7 Procedure

7.1 Preparation of the test portion

Weigh, to the nearest 0,05 % (relative), a quantity of the test sample recommended by the instrument manufacturer as appropriate to the type of instrumentation and the expected concentrations of carbon, hydrogen and nitrogen. The test portion shall be weighed directly into the sample capsule in the case of a micro- or semi-micro-analyser. Otherwise it may be weighed directly or transferred from a suitable weighing container.

If results are to be calculated other than on the "air-dried" basis (see clause 8), then concurrently weigh the test portion for analysis and a further portion for the determination of the moisture content in accordance with the method described in ISO 331, ISO 687, ISO 1015 or ISO 5068, as appropriate.

NOTE Pelletising samples of low rank coals with a low mass fraction of nitrogen to express air trapped in the pores, has been found to give more reliable results.

7.2 Calibration of the apparatus

Condition the apparatus by analysing two test portions with concentrations within the expected ranges of values. Discard these results.

Select calibration standards (4.4), or certified reference materials (CRM) (4.5), representing the levels of carbon, hydrogen and nitrogen in the samples to be tested, and preferably covering the expected ranges. Make five determinations of each standard or CRM selected, operating the instrument in the manner described in the manufacturer's instructions.

Check that the five determinations meet the precision requirements of this test method by comparing the range of results with the appropriate repeatability limits (see Table 1). The range should not exceed the repeatability limit by a factor greater than 1,4. If it does exceed that value, ensure that the instrument is in proper working order, review the test procedure used and repeat the calibration test.

Using the average value of the five determinations for each element, calibrate the instrument as described in the manufacturer's instructions.

When the instrument has been calibrated, verify the calibration by analysing (see 7.3), as a test sample, a portion of a suitable calibration standard (4.4) or CRM (4.5). The calibration is acceptable if the measured value for this determination differs from the standard value by no more than the repeatability limit for the test method (see clause 9). Otherwise repeat the calibration procedure (7.2).

In addition to the initial setting-up, the instrument shall be calibrated after any changes have been made to the equipment and whenever routine analysis of standard samples (see 7.3) indicates a difference between the standard and measured values in excess of the repeatability limit for the test method.

7.3 Analysis of test samples

Analyse test portions (7.1) of the test samples in accordance with the manufacturer's instructions.

At intervals between batches of samples, analyse a calibration standard (4.4) or CRM (4.5). If the measured value differs from the known value by more than the repeatability limit of the test method (see clause 9), recalibrate the instrument as in clause 7.2.

If CRMs have not been used in 7.2 or for the standard in the preceding paragraph, analyse a CRM at the beginning and end of an appropriate series of analyses to obtain traceability and to document the accuracy of results.

8 Expression of results

The total carbon, total hydrogen and nitrogen mass fractions of the sample, as analysed, shall be expressed as a percentage by mass.

Report the results, as the mean of duplicate determinations, to the nearest 0,1 % for carbon and 0,01 % for hydrogen and nitrogen.

The results of the determination described in this Technical Specification are reported on the "air-dried" basis. Calculation of results to other bases is dealt with in ISO 1170.

9 Precision

9.1 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 test sample, shall not differ by more than the values shown in Table 1.

9.2 Reproducibility critical difference

The means of the results of duplicate determinations, carried out in each of two laboratories, on representative portions taken from the same sample after the last stage of sample preparation, shall not differ by more than the values shown in Table 1.

Table 1

Element	Maximum acceptable difference between results (calculated to the same moisture contents)	
	Repeatability limit	Reproducibility critical difference
Carbon	0,5 % absolute	1,5 % absolute
Hydrogen	0,10 % absolute	0,3 % absolute
Nitrogen	0,08 % absolute	0,15 % absolute

10 Test report

The test report shall include the following information:

- a) the method used by reference to this Technical Specification;
- b) the identification of the sample tested;
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this Technical Specification, or regarded as optional;
- f) the date of the test.