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

Combustibles minéraux solides — Dosage du carbone, de l'hydrogène et de l'azote totaux — Méthode instrumentale

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 29541 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This first edition of ISO 29541 cancels and replaces ISO/TS 12902:2001, which has been technically revised.

Introduction

The reliable determination of total carbon, hydrogen and nitrogen is important for engineering calculations applied to the combustion of coal. The precise and accurate determination of the carbon content of coal is essential for carbon accounting purposes.

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

WARNING — The use of this International Standard can involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of total carbon, hydrogen and nitrogen in coal and coke by instrumental methods.

NOTE This International Standard has been validated for coal only in accordance with the principles of ISO 5725-1.

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

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

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

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

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

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

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

ISO 18283, *Hard coal and coke — Manual sampling*

3 Terms and definitions

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

4 Principle

Carbon, hydrogen and nitrogen are determined concurrently in a single instrumental procedure. The quantitative conversion of the carbon, hydrogen and nitrogen into their corresponding gases (CO_2, H_2O, N_2/NO_x) occurs during combustion of the sample at an elevated temperature in an atmosphere of oxygen. Combustion products which would interfere with the subsequent gas analysis are removed. Oxides of nitrogen (NO_x) produced during the combustion are reduced to N_2 before detection. The carbon dioxide, water vapour and elemental nitrogen in the gas stream are then determined quantitatively by appropriate instrumental gas analysis procedures.

5 Reagents

Unless otherwise specified, all reagents shall be of analytical reagent grade.

5.1 Carrier gas, helium or other suitable gas as specified by the instrument manufacturer.

5.2 Oxygen, as specified by the instrument manufacturer.

5.3 Additional reagents, of types and qualities specified by the instrument manufacturer.

5.4 Calibration materials: see Table 1.

Table 1 — Examples of suitable calibration materials and their stoichiometric contents of C, H and N

Name	Formula	Mass fraction %		
		Carbon	Hydrogen	Nitrogen
EDTA (ethylene diamine tetra-acetic acid)	$C_{10}H_{16}N_2O_8$	41,1	5,5	9,6
Phenylalanine	$C_9H_{11}NO_2$	65,4	6,7	8,5
Acetanilide	C_8H_9NO	71,1	6,7	10,4
BBOT (CAS-No 7128-64-5) 2,5-bis (5′-tert-butyl-2-benzoxazolyl) thiophene	$C_{26}H_{26}N_2O_2S$	72,5	6,1	6,5

If these materials are accompanied by a traceable certificate of analysis that includes the uncertainty of the assigned carbon, hydrogen and nitrogen values, then use the certificate values for calibration purposes. If pure compounds (> 99,5 % purity) are available, use the stoichiometric values. Store these substances in a desiccator under conditions that maintain the compounds in a dry state.

Table 1 lists those pure substances that were included in the interlaboratory study (ILS) to determine the calibration requirements and precision of this International Standard. The ILS indicated benzoic acid is not suitable for calibration. Pure substances other than those listed in Table 1 can be used for calibration provided the substances meet the purity and calibration requirements of this International Standard.

5.5 Reference materials.

Reference material coal(s) with a certified composition and uncertainty for carbon, hydrogen and nitrogen may be used as a check to monitor changes in instrument response, which can be affected by constituents not present in the calibration materials, and to verify the acceptability of nitrogen results. Alternatively, coal of a known composition can also be used as a check sample. As the bulk composition of coal can change during storage, coals shall not be used for calibration.

6 Apparatus

6.1 Analytical instrument, consisting of a furnace, gas handling and detection system capable of analysing a test portion of 6 mg or greater.

6.2 Balance, stand-alone or integrated with the instrument, with a resolution of a least 0,1 % of the test portion to be weighed.

7 Preparation of the test sample

The sample shall be the general analysis test sample prepared to a nominal top size of 212 µm using ISO 13909-4, ISO 13909-6, ISO 18283 or ISO 5069-2. Sample preparation procedures are described in ISO 13909-4 for coal, ISO 13909-6 for coke and ISO 5069-2 for brown coal and lignites.

The moisture content of each test sample and reference material shall be determined in accordance with ISO 11722 for coal, ISO 687 for coke or ISO 5068-2 for brown coals and lignites. Alternatively, the test sample and reference material shall be dried prior to analysis.

8 Procedure

8.1 Instrument set-up

Verify that all instrument operation parameters meet the specifications in the instrument operating manual. Verify the condition and quantity of all chemicals currently in use in the instrument to ensure they are satisfactory for the number of samples to be analysed. Prior to any analysis, check for, and if necessary correct, any leaks in the combustion system and carrier gas system.

8.2 Blank analyses

Perform blank analyses daily to establish carbon, hydrogen and nitrogen levels in the combustion and carrier gases. The level of nitrogen in these gases shall not exceed 1 % of the instrument nitrogen response for the lowest mass of calibration material. Repeat blank analyses after changing or renewing gases or other reagents.

8.3 Conditioning and instrument stability check

Condition the instrument in accordance with the manufacturer's instructions (generally by running at least two test portions of a coal, coke or brown coal or lignite that have a composition typical of the general analysis samples).

Select a conditioning sample of similar composition to a typical sample. Carry out four determinations of the conditioning sample. Discard the first determination.

If any maximum difference of three retained repeat determinations for carbon, hydrogen and nitrogen values exceeds 1,2 r, where r is the repeatability limit (see Clause 10) of this International Standard, instrument stability is suspect. In this case, take corrective action before proceeding with calibration.

8.4 Calibration

The instrument shall be calibrated as recommended by the instrument manufacturer, or whenever changes have been made to the equipment and whenever analysis of verification samples (see 8.5) indicates an unacceptable difference between the certified and the measured values.