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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

ICS 73.040; 75.160.10

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Foreword

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

This second/third/... edition cancels and replaces the first/second/... edition (), [Clause (s) / subClause (s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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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.

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

1 Scope

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

NOTE: This standard has been validated in accordance with the principles of ISO 5725 for coal only.

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, Coke—Determination of moisture in the analysis 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—Indirect gravimetric method, Part 2: Moisture of 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 5725-6, Accuracy (trueness and precision) of measurement methods and results Part 6: Use in practice of accuracy values

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 purpose of this Standard, the 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 (CO₂, H₂O,

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

5.1 General

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

5.2 Carrier gas

Helium or other suitable gas as specified by the instrument.

5.3 Oxygen

As specified by the instrument.

5.4 Additional reagents

Of types and qualities specified by the instrument.

5.5 Calibration materials

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

Name	Formula	Mass fraction in percent		
		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.

NOTE: Table 1 includes those pure substances that were included in the interlaboratory study (ILS) employed to determine the calibration requirements and precision of this standard. This 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 standard.

5.6 Certified reference materials

Certified reference material (CRM) 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 that 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

Analytical instrument

An instrument capable of analysing a test portion of 6 mg or greater. It includes a furnace capable of maintaining a temperature in the range to ensure quantitative recovery of carbon, hydrogen and nitrogen as their corresponding gases (CO_2 , H_2O , N_2/NO_x). The combustion gases or a representative aliquot shall be treated to remove or convert any components that can interfere with the measurement of carbon dioxide, water vapour or nitrogen (produced by the reduction of NO_x prior to measurement). The detection system shall include provisions for evaluation of the response in a manner that correlates accurately with the carbon dioxide, water vapour and nitrogen present in the treated combustion gases.

NOTE Coal contains constituents that can affect the performance of chemicals used to ensure the consistent and uniform conversion and recovery of carbon, hydrogen and nitrogen.

Balance

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

7 Preparation of the test sample

The sample shall be the general analysis sample prepared to a nominal top size of 212 μm using 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 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. Instruments that employ gas to open and close valves shall use extra dry gas for this purpose. 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 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. Re-determine blank analyses, whenever the carrier gas or oxygen supplies are changed or any chemicals are renewed.