

BS ISO 29541:2010



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

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National foreword

This British Standard is the UK implementation of ISO 29541:2010. It supersedes DD ISO/TS 12902:2002 which is withdrawn.

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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*



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Foreword

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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)	$\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8$	41,1	5,5	9,6
Phenylalanine	$\text{C}_9\text{H}_{11}\text{NO}_2$	65,4	6,7	8,5
Acetanilide	$\text{C}_8\text{H}_9\text{NO}$	71,1	6,7	10,4
BBOT (CAS-No 7128-64-5) 2,5-bis (5'-tert-butyl-2-benzoxazolyl) thiophene	$\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_2\text{S}$	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 $1,2r$, 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.

Select amounts of one or more calibration materials (see 5.4) based on the regression to be used and the expected ranges of carbon, hydrogen and nitrogen in the test samples to be analysed. See Clause A.1 for recommendations about the number of calibration points and Clause A.2 for recommendations concerning calibration masses.

NOTE Coal is not used for calibration because coal degrades with time and its composition changes.

8.5 Verification of calibration

Before analysis of the test samples, verify the acceptability of the calibration by analysing a calibration material (see 5.4) not used for calibration. In Table 2, the limits for the difference between the measured and stoichiometric or certificate values are listed.

Table 2 — Calibration acceptance limits

Element	Acceptance limit Relative percentage (%)
Carbon	1,20
Hydrogen	2,10
Nitrogen	1,80

If the verification result does not agree within the limits specified in Table 2, an additional verification run shall be conducted. If the results of the additional verification run do not agree with the measured and stoichiometric or certificate value within the limits, the instrument shall be recalibrated.

Clause A.3 gives an example of calibration verification.

8.6 Analysis of test samples

As indicated in Clause 7, determine the moisture content of each test sample concurrently with this analysis.

Carry out an analysis of each test sample by weighing out a test portion with a mass normally employed for analysis. All test samples shall be run in duplicate.

Verify the calibration at the completion of each test batch, and preferably at least every ten samples, in accordance with 8.5.

At the beginning of the appropriate series of test samples, or at the end of the appropriate series of test samples, or both, analyse a reference material as a test sample in duplicate in order to obtain acceptable instrument performance and calibration for the sample matrix.

The duplicate results for carbon, hydrogen and nitrogen, calculated to dry basis in accordance with Clause 9, shall agree within the repeatability limit specified in Clause 10.

If any of these acceptance criteria fail, reject all test results back to the last acceptable result for the certified reference material (CRM). Action shall be taken to identify and correct the assignable cause and, if necessary, the instrument shall be recalibrated in accordance with 8.4.

9 Expression of results

Record the total carbon, hydrogen and nitrogen as analysed, as a percentage by mass.

Report the results (the mean of duplicate determinations) on dry basis to the nearest 0,1 % for carbon and to the nearest 0,01 % for hydrogen and nitrogen.

For the calculation to dry basis, use Equations (1) to (3), in which the subscript “d” indicates dry basis and the subscript “ad” indicates as-determined (as-analysed) basis:

— for the carbon content:

$$w_{Cd} = w_{Cad} \times \frac{100}{100 - w_{Mad}} \quad (1)$$

— for the nitrogen content:

$$w_{Nd} = w_{Nad} \times \frac{100}{100 - w_{Mad}} \quad (2)$$

— for the hydrogen content:

$$w_{Hd} = \left(w_{Had} - \frac{w_{Mad}}{8,937} \right) \times \frac{100}{100 - w_{Mad}} \quad (3)$$

where

w_{Cd} is the content of carbon, dry basis, mass fraction in percentage;

w_{Cad} is the content of carbon as determined (as analysed), mass fraction in percentage;

w_{Nd} is the content of nitrogen, dry basis, mass fraction in percentage;

w_{Nad} is the content of nitrogen as determined (as analysed), mass fraction in percentage;

w_{Hd} is the content of hydrogen, dry basis, mass fraction in percentage;

w_{Had} is the content of hydrogen as determined (as analysed), mass fraction in percentage;

w_{Mad} is the moisture content of the sample as analysed, mass fraction in percentage.

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 test sample, shall not differ by more than the values shown in Table 3.

10.2 Reproducibility limit

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 3.

Table 3 — Precision limits

Element	Maximum acceptable difference between results (dry basis)	
	Repeatability limit <i>r</i>	Reproducibility limit <i>R</i>
Carbon	0,3 % absolute	1,0 % absolute
Hydrogen	0,06 % absolute	0,25 % absolute
Nitrogen	0,03 % absolute	0,15 % absolute

11 Test report

The test report shall contain the following information:

- a) complete identification of the sample tested;
- b) reference to this International Standard (i.e. ISO 29541:2010);
- c) results of the determination for carbon, hydrogen and nitrogen, together with the relevant analysis basis (e.g. dry basis, dry ash free basis).

Annex A (informative)

Recommendations for calibration

A.1 Recommended number of calibration points

Table A.1 gives the recommended number of calibration points for common regressions.

Table A.1 — Recommended number of calibration points for common regressions

Regression fit	Recommended number of calibration points
Linear	6
Quadratic	7
Power	7
Cubic	8

To apply all of these regressions for the instrument calibration standard, a minimum of 8 and preferably 10 calibration points are recommended.

A.2 Recommended calibration masses for carbon

Table A.2 gives the calibrant mass, in milligrams, required to yield the same amount of carbon present in a 100 mg test portion of coal.

Table A.2 — Calibrant masses

Calibrant	Carbon percentage in 100 mg test portion coal											
	35 %	40 %	45 %	50 %	55 %	60 %	65 %	70 %	75 %	80 %	85 %	90 %
	Mass of calibrant in milligrams											
EDTA	85	97	110	122	134	146	158	170	183	195	207	219
Phenylalanine	53	61	69	76	84	92	99	107	115	122	130	138
Acetanilide	49	56	63	70	77	84	91	98	106	113	120	127
BBOT	48	55	62	69	76	83	90	97	104	111	118	125

Calculate the equivalent amounts of calibrant for the test portion, in milligrams, to be used for the coal samples by multiplying the amounts listed in Table A.2 by a factor CF, corresponding to the test portion mass, in milligrams per 100 mg.

For example, if the normal test portion mass in a laboratory is 70 mg, then CF is 0,7. Each of the calibrant masses in Table A.2 are multiplied by 0,7 to obtain the correct masses for calibration.

Select calibration masses based on the recommended number of calibration points specified in Table A.1 that cover the expected range of carbon in the test sample. It is acceptable to run calibration masses in duplicate to meet the requirements in Table A.1. This approach can improve the reliability of the regression.

Table A.3 gives the equivalent hydrogen percentage in coal for the calibrant masses listed in Table A.2.

Table A.3 — Equivalent hydrogen percentage in coal

Calibrant	Carbon percentage in 100 mg test portion coal											
	35 %	40 %	45 %	50 %	55 %	60 %	65 %	70 %	75 %	80 %	85 %	90 %
	Equivalent hydrogen percentage in coal											
EDTA	4,7	5,4	6,1	6,7	7,4	8,1	8,7	9,4	10,1	10,8	11,4	12,1
Phenylalanine	3,6	4,1	4,6	5,1	5,6	6,2	6,6	7,2	7,7	8,2	8,7	9,3
Acetanilide	3,3	3,8	4,2	4,7	5,2	5,6	6,1	6,6	7,1	7,6	8,1	8,5
BBOT	2,9	3,3	3,8	4,2	4,6	5,1	5,5	5,9	6,3	6,8	7,2	7,6

NOTE The amount of hydrogen in the majority of coal employed for power production purposes is in the range of 4,5 % to 6,1 % when the hydrogen in the coal moisture is taken into account. This information is provided to assist in the selection of calibration(s).

Table A.4 gives the equivalent nitrogen percentage in coal for the calibrant masses listed in Table A.2.

Table A.4 — Equivalent nitrogen percentage in coal

Calibrant	Carbon percentage in 100 mg test portion coal											
	35 %	40 %	45 %	50 %	55 %	60 %	65 %	70 %	75 %	80 %	85 %	90 %
	Equivalent nitrogen percentage in coal											
EDTA	8,2	9,3	10,6	11,7	12,9	14,0	15,2	16,3	17,6	18,7	19,9	21,0
Phenylalanine	4,5	5,2	5,9	6,5	7,1	7,8	8,4	9,1	9,8	10,4	11,1	11,7
Acetanilide	5,1	5,8	6,6	7,3	8,0	8,7	9,5	10,2	11,0	11,8	12,5	13,2
BBOT	3,1	3,6	4,0	4,5	4,9	5,4	5,9	6,3	6,8	7,2	7,7	8,1

NOTE The amount of nitrogen in the majority of coal employed for power production purposes is in the range of 0,5 % to 1,8 %. None of the calibrants yield equivalent nitrogen percentage values within the ranges expected for coal. For this reason, CRM coal(s) (see 5.5) are used to check the acceptability of nitrogen results.

A.3 Calibration verification

Verify the acceptability of the calibration by determining the carbon, hydrogen and nitrogen content of a pure substance (see 5.4) not used for calibration (see Clause A.2).

Table A.5 lists the relative percentage by which the verification result for carbon, hydrogen and nitrogen shall agree with the certificate value of the verification material.

If any of the verification results are not within the limits specified in Table A.5, conduct an additional verification run with a mass of verification calibrant that yields the same carbon content as the mass of calibrant employed at the extremes of the calibration (see Table A.2). If the additional verification run is within the limits specified in Table A.5, then it is acceptable to proceed with the analysis of the test samples. Otherwise, reject the calibration as well as all test sample results back to the last acceptable verification. Check instrument set-up (see 8.1), conduct blank analyses (see 8.2) and condition the instrument (see 8.3). Calibrate the instrument in accordance with Clauses A.1 and A.2 before proceeding with the analysis.

Table A.5 — Calibration relative percentage acceptance limits

Element	Acceptance limit Relative percentage (%)
Carbon	1,20
Hydrogen	2,10
Nitrogen	1,80

NOTE These limits are developed from the calibration data supplied by the 14 laboratories that took part in the interlaboratory study (ILS) employed to validate this International Standard. The calibration data include results from the pure substances listed in 5.4 and five instrument configurations covering two phases of the ILS conducted one year apart. These limits can serve as reasonable performance criteria for calibrations conducted in the same or different laboratories over an extended period of time.

Table A.6 — Example of calibration verification with EDTA

Parameter	Certificate mass fraction %	Relative limit	Absolute limit mass %	Verification result	Absolute difference mass fraction %
Carbon	41,08	1,20%	0,49	41,27	0,19
Hydrogen	5,51	2,10%	0,12	5,59	0,08
Nitrogen	9,60	1,80%	0,17	9,50	0,11

Since the absolute difference between the verification result and the certificate value are within the absolute limit for carbon, hydrogen and nitrogen, the laboratory can proceed with the analysis of the test samples without recalibrating the instrument.

A.4 Acceptance of results for reference material coal(s)

A.4.1 Calculate the as-determined carbon, hydrogen and nitrogen results to dry basis in accordance with Clause 9.

A.4.2 Verify that the dry basis duplicate results for carbon, hydrogen and nitrogen are within the repeatability limits specified in Clause 10.

For nitrogen, verify that each dry basis nitrogen result agrees with the certificate value within 0,11 mass %.

NOTE 1 The 0,11 % acceptance limit is derived from the reproducibility critical difference for nitrogen cited in Clause 10, in accordance with ISO 5725-6:1994, 4.2.3 ("Comparison with a reference value for one laboratory").

For carbon and hydrogen, it is recommended that a record be kept of the dry basis results. Each dry basis carbon result should agree with the dry basis certificate value within 0,9 mass %. Each dry basis hydrogen result should agree with the dry basis certificate value within 0,3 mass %. The certifying agency should be notified when more than seven consecutive dry basis results for carbon, or for hydrogen, or for both carbon and hydrogen fall outside these limits. The certifying agency should be provided with the purchase date, in-service date and expiry date of the CRM coal.

NOTE 2 The comparison limits for carbon and hydrogen are derived from the reproducibility critical difference cited in Clause 10, in accordance with in ISO 5725-6:1994, 4.2.3 ("Comparison with a reference value for one laboratory").

If any of these acceptance criteria fail, then reject all test results back to the last acceptable certified reference material result(s). Check instrument set-up (see 8.1), conduct blank analyses (see 8.2) and condition the instrument (see 8.3). Calibrate the instrument in accordance with Clauses A.1 and A.2 before proceeding with the analysis.

Bibliography

- [1] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- [2] ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

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