



**International
Standard**

ISO 29541

**Coal and coke — Determination of
total carbon, hydrogen and nitrogen
— Instrumental method**

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

**Second edition
2025-02**



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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 29541:2010), which has been technically revised.

The main changes are as follows:

- the repeatability limits have been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html

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 mass fraction of carbon in coal is essential for carbon accounting purposes.

Coal and coke — Determination of total carbon, hydrogen and nitrogen — Instrumental method

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

1 Scope

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

NOTE This document has been validated for coal only, in accordance with the principles of ISO 5725-1. The suite of samples used in the interlaboratory study (ILS) to determine the precision data did not include coke and therefore there is insufficient data to state precision limits for coke.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 general analysis test sample*

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

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 18283, *Coal and coke — Manual sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

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, N₂/NO_x) occurs during combustion of the sample at an elevated temperature in an atmosphere of oxygen. Combustion products which can interfere with the subsequent gas analysis are removed. Oxides of nitrogen (NO_x) produced during the combustion are reduced to N₂ 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

| Name | Formula | Stoichiometric mass fractions % | | |
|---|-----------------------|------------------------------------|----------|----------|
| | | 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. It is recommended that the relative expanded uncertainty for these materials be no more than 0,2 % for C, 0,5 % for H, and 1 % for N. Assuming $k = 2$, that the relative standard uncertainty be no more than 0,1 % for C, 0,25 % for H, and 0,5 % for N.

If pure compounds (> 99,5 % purity with stated uncertainty) are available, use the stoichiometric values multiplied by the stated purity. Uncertainty specifications similar to those stated above are recommended. 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 document. 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 have a documented purity and uncertainty and meet the purity and calibration requirements of this document. Uncertainty specifications similar to those stated above are recommended for those substances.

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 at least 0,1 % of the mass of the test portion.

7 Preparation of the test sample

The sample shall be the general analysis test sample prepared to a nominal top size of 212 μm in accordance to procedures described in ISO 13909-4 for coal or ISO 18283 for coke.

Ensure that the moisture 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.

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

The moisture mass fraction of each test sample and the reference material shall be determined in accordance with ISO 11722, ISO 687 or ISO 5068-2 as appropriate.

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, this is done by running at least two test portions of a coal sample that has 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 document, instrument stability is suspect. In this case, take corrective action before proceeding with calibration.

8.4 Calibration

The instrument shall be calibrated as instructed 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 specific masses 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 A.1 for recommendations about the number of calibration points and 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 acceptance limits for the difference between the measured and either the stoichiometric or certificate values are listed.

Table 2 — Calibration acceptance limits

| Element | Acceptance limit Relative percentage (%) |
|----------|---|
| Carbon | 0,2 |
| Hydrogen | 0,5 |
| Nitrogen | 1 |

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.

A.3 gives an example of calibration verification.

8.6 Analysis of test samples

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

Test each sample using sample masses that are usually used for this analysis. All test samples shall be analysed 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 in accordance with A.4.

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 mass fraction of total carbon, hydrogen and nitrogen as analysed, expressed as a percentage.

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 Formulae (1) to (3), in which the subscript “d” indicates a dry basis and the subscript “ad” indicates an as-determined (as-analysed) basis:

— for the carbon mass fraction:

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

— for the nitrogen mass fraction:

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

— for the hydrogen mass fraction:

$$w_{Hd} = \left(w_{Had} - \frac{w_{Mad}}{8,936} \right) \times \frac{100}{100 - w_{Mad}} \quad (3)$$

where

- w_{Cd} is the mass fraction of carbon, dry basis, expressed as a percentage, %;
- w_{Cad} is the mass fraction of carbon in the air dried sample, expressed as a percentage, %;
- w_{Nd} is the mass fraction of nitrogen, dry basis, expressed as a percentage, %;
- w_{Nad} is the mass fraction of nitrogen in the air dried sample, expressed as a percentage, %;
- w_{Hd} is the mass fraction of hydrogen, dry basis, expressed as a percentage, %;
- w_{Had} is the mass fraction of hydrogen in the air dried sample, expressed as a percentage, %;
- w_{Mad} is the moisture mass fraction of the sample as analysed, expressed as a percentage, %;
- 100 is the conversion factor from a mass fraction of dimension one to percent, in %;
- 8,936 is the molecular mass conversion factor to convert moisture (H_2O) to hydrogen only.

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 | 0,45 % absolute | 1,0 % absolute |
| Hydrogen | 0,10 % absolute | 0,25 % absolute |
| Nitrogen | 0,05 % absolute | 0,15 % absolute |
| NOTE The suite of samples used in the interlaboratory study (ILS) to determine this precision data did not include coke and therefore there is insufficient data to state precision limits for coke. | | |

11 Test report

The test report shall contain the following information:

- complete identification of the sample tested;
- reference to this document (i.e. ISO 29541:2025);
- the date of the determination;
- 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 (normative)

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, needed to yield the same amount of carbon present in a 100 mg test portion of coal.

Table A.2 — Calibrant masses

| Calibrant | Carbon mass fraction, in % 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. 100 mg of EDTA ($C_{10}H_{16}N_2O_8$), based on stoichiometry, contains 41,1 % C, 5,5 % H, and 9,6 % N. Therefore, on a weighted proportional basis, 85 mg of EDTA whose mass is recorded as 100 mg in the instrument will yield mass fractions of 35 % for C, 4,7 % for H and 8.2 % for N.

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 preferable 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 mass fraction, in % in coal for the calibrant masses listed in [Table A.2](#).

Table A.3 — Equivalent hydrogen mass fraction, in % in coal

| Calibrant | Carbon mass fraction, in % in 100 mg test portion coal | | | | | | | | | | | |
|---------------|--|------|------|------|------|------|------|------|------|------|------|------|
| | 35 % | 40 % | 45 % | 50 % | 55 % | 60 % | 65 % | 70 % | 75 % | 80 % | 85 % | 90 % |
| | Equivalent hydrogen mass fraction, in % 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 mass fraction 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 mass fraction, in % in coal for the calibrant masses listed in [Table A.2](#).

Table A.4 — Equivalent nitrogen mass fraction, in % in coal

| Calibrant | Carbon mass fraction, in % in 100 mg test portion coal | | | | | | | | | | | |
|---------------|--|------|------|------|------|------|------|------|------|------|------|------|
| | 35 % | 40 % | 45 % | 50 % | 55 % | 60 % | 65 % | 70 % | 75 % | 80 % | 85 % | 90 % |
| | Equivalent nitrogen mass fraction, in % 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 mass fraction 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 mass fraction % of a pure substance (see [5.4](#)) not used for calibration (see [A.2](#)).

[Table A.5](#) lists the relative mass fraction, in % by which the verification result for carbon, hydrogen and nitrogen shall agree with the certificate value of the verification material. [Table A.6](#) lists examples of calibration verification with EDTA.

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 mass fraction 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 [A.1](#) and [A.2](#) before proceeding with the analysis.

Table A.5 — Calibration relative percentage acceptance limits

| Element | Acceptance limit Relative percentage (%) |
|----------|---|
| Carbon | 0,2 |
| Hydrogen | 0,5 |
| Nitrogen | 1 |

NOTE These limits are developed from the calibration data supplied by the 14 laboratories that took part in the ILS employed to validate this document. 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. Acceptance limits that are no more than about one-third of the value of r minimise bias caused by the calibrants.

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 a mass fraction of 0,11 %.

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.

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 a mass fraction of 0,9 %. Each dry basis hydrogen result should agree with the dry basis certificate value within a mass fraction of 0,3 %.

In accordance with good laboratory practice the laboratory should notify the certifying agency if it suspects that the reference material's oxidation sensitive parameters, C and H, have deteriorated from the published values, for example, 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 ISO 5725-6:1994, 4.2.3.

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 [A.1](#) and [A.2](#) before proceeding with the analysis.

Bibliography

- [1] ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*
- [2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- [3] ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*
- [4] ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal – Sampling from moving streams*



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