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Solid mineral fuels — Determination of carbonate carbon content — Gravimetric method

Combustibles minéraux solides — Dosage du carbone sous forme de carbonate — Méthode gravimétrique

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.

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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 925:1997), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— the normative references have been updated and the dates removed;

— the references in Clause 7 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.

Solid mineral fuels — Determination of carbonate carbon content — Gravimetric method

1 Scope

This document specifies a gravimetric method of determining the carbon in the mineral carbonates associated with solid mineral fuels.

NOTE The result obtained will include any carbon from atmospheric carbon dioxide absorbed by the fuel.

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

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

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 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

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

A known mass of sample is treated with hydrochloric acid, which reacts with the carbonates present to liberate carbon dioxide. The carbon dioxide resulting from the decomposition of the carbonates is absorbed and weighed.

5 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.

NOTE Distilled water can be freed from carbon dioxide by boiling gently for 15 min.

5.1 Hydrochloric acid, approximately 3 mol/l.

5.2 Hydrogen sulfide absorbent, any of the following:

a) copper(II) phosphate, granular, particle size 1,2 mm to 0,7 mm;

NOTE 1 Copper(II) phosphate granules can be prepared as follows.

Mix copper(II) phosphate powdered reagent to a stiff paste with 1 % starch solution. Press through a sheet of metal, perforated with apertures of approximately 1 mm diameter. Dry the extruded material at 110 °C. Sieve to recover the desired size fraction.

b) copper(II) sulfate, deposited on a supporting base of ground pumice;

NOTE 2 A suitable absorbent, based on copper(II) sulfate, can be prepared as follows.

Prepare pumice by crushing and sieving to obtain the 2,8 mm to 0,7 mm fraction. Transfer approximately 60 g of the prepared pumice to an evaporating basin, covering with a saturated solution of copper(II) sulfate, evaporate to dryness with constant stirring, and heat at 150 °C to 160 °C for 3 h to 4 h. Cool in a desiccator and store in a glass-stoppered bottle.

c) silver sulfate, granular.

5.3 Magnesium perchlorate, anhydrous, particle size 1,2 mm to 0,7 mm.

WARNING — Due regard shall be taken of local regulations when disposing of exhausted magnesium perchlorate. It is essential that regeneration of magnesium perchlorate is not attempted, owing to the risk of explosion.

5.4 Sodium hydroxide, on an inert base, preferably of coarse grading, for example 1,7 mm to 1,2 mm, and preferably of the self-indicating type.

5.5 Wetting agent, suitable for use in acid solution.

NOTE A liquid wetting agent at a concentration of 100 ml/l or ethanol (a volume fraction of 95 %) are suitable.

5.6 Check test reagent, either of the following:

a) anhydrous sodium carbonate;

b) anhydrous calcium carbonate.

6 Apparatus

6.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.2 Graduated glassware, conforming to the requirements for Grade A in the International Standards prepared by ISO/TC 48, *Laboratory glassware and related apparatus*.

6.3 Purification tube, consisting of an absorption tube containing sodium hydroxide on an inert base (5.4). Absorption tubes may be U-tubes or Midvale tubes (which reduce back-pressure and, hence, risk of

leakage). The tops of the reagent columns should be covered with a layer of glass wool to guard against entrainment of any fine particles in the circulating air.

6.4 Reaction flask assembly, comprising a 300 ml round-bottomed flask fitted with a tap funnel, a double-surface condenser and a bulbed tube.

6.5 Absorption train, consisting of three absorption tubes a), b) and c) packed, respectively, as follows.

a) Magnesium perchlorate (5.3) to dry the gas.

b) Hydrogen sulfide absorbent (5.2) followed by a protective layer of magnesium perchlorate (5.3). The connection from this tube to tube c) shall be fitted at its outlet end with a stopcock or other means of closure.

c) Sodium hydroxide on an inert base (5.4), to absorb carbon dioxide generated in the reaction flask, followed by a protective layer of magnesium perchlorate (5.3) to absorb water produced in the reaction between carbon dioxide and sodium hydroxide. This tube shall be fitted with stopcocks or other means of closure at the inlet and outlet ends.

6.6 Air circulation equipment, a suction pump, capable of drawing air at a rate of 50 ml/min through the apparatus, connected through a flowmeter to a tee-piece fitted with a stopcock (the air vent).

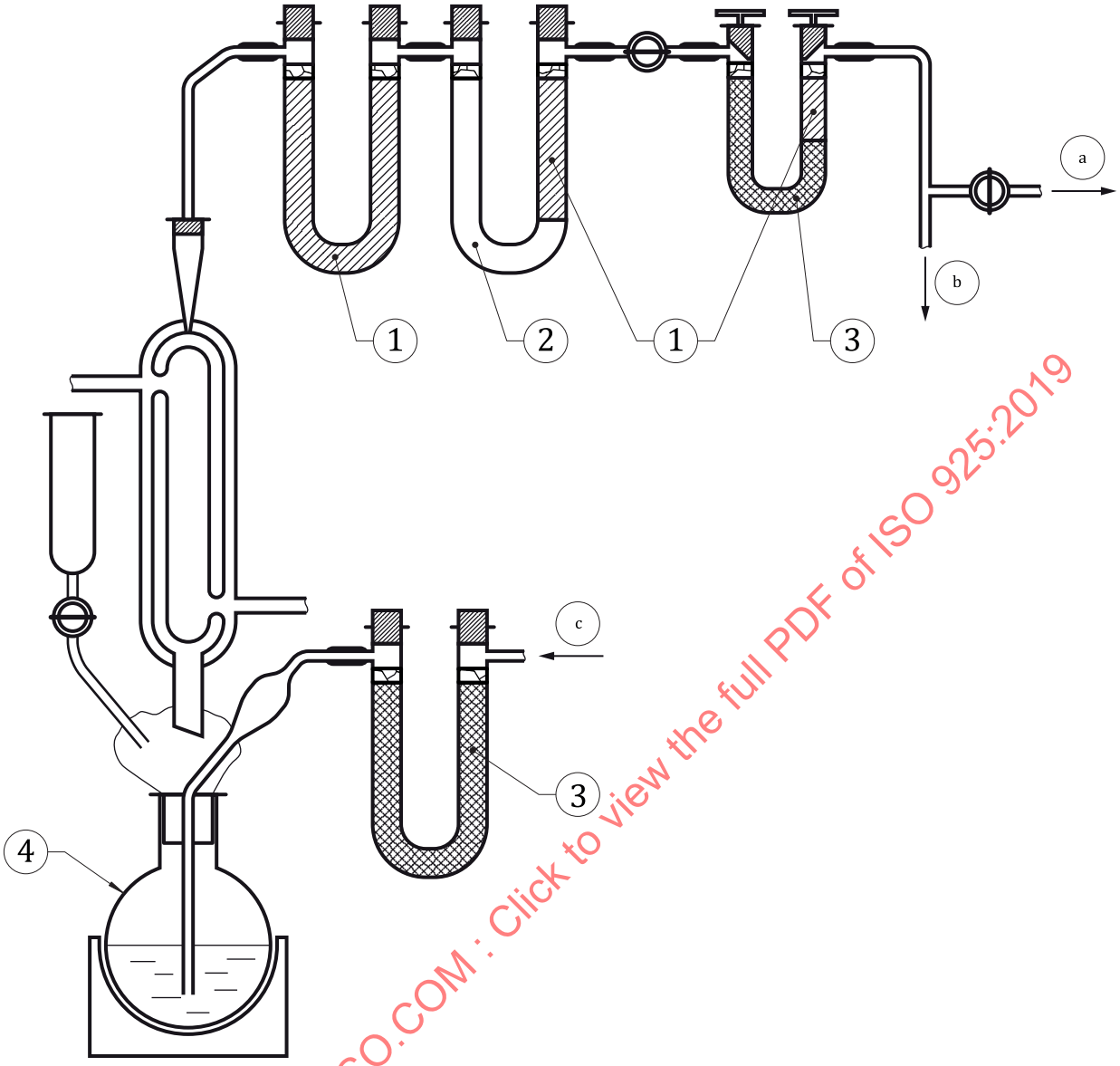
6.7 Heating source, for the reaction flask. Either an electric heating mantle to accommodate a 300 ml flask or a small gas burner. An example of a suitable assembly of the apparatus is illustrated in Figure 1.

7 Preparation of the test sample

The test sample shall be the analysis sample, prepared to a nominal top size of 212 µm. Sample preparation procedures shall be in accordance with ISO 13909-4, ISO 13909-6 or ISO 18283, as appropriate. The moisture content of the sample shall be equilibrated with the laboratory atmosphere by exposure in a thin layer on a tray. Exposure time shall be kept to a minimum.

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

If the results are to be calculated other than on an "air-dried" basis (see Clause 9), then, after weighing the test portion (see 8.2), determine the moisture content using a further portion of the test sample by the method described in ISO 687, ISO 5068-2 or ISO 11722, as appropriate.



Key

1	magnesium perchlorate	a	Air vent.
2	hydrogen sulfide absorbent	b	To flowmeter and suction pump.
3	sodium hydroxide on inert base	c	Air inlet.
4	reaction flask		

Figure 1 — A suitable assembly of the apparatus

8 Procedure

8.1 Check test

Check the air-tightness of the apparatus and condition of the reagents by running a test according to the procedure described in 8.2, but using approximately 60 mg of the check test reagent (5.6), weighed to the nearest 0,1 mg, instead of the test sample.

Calculate the theoretical mass of carbon dioxide (see A.2) expected to be liberated from the check test portion and compare it with the measured value. The test is satisfactory if the measured value is within

10 % of the theoretical value. Otherwise, check the apparatus carefully to seal any leaks and/or replace the reagents. Re-test until a satisfactory result is obtained.

Carry out check tests after initially setting up the apparatus, after having made any changes to it or to the reagents, and before making a determination or series of determinations.

8.2 Determination

8.2.1 Preparation

Weigh accurately, to the nearest 0,01 g, 5 g of the sample into the reaction flask and add five drops of the wetting agent (5.5) and 100 ml of water. Close the flask by means of a rubber stopper and shake vigorously to wet the sample. Remove the stopper and wash any sample adhering to it back into the flask. Assemble the apparatus as shown in Figure 1, with the air vent closed.

For fuels containing more than 0,5 % carbonate carbon, the sample mass may be reduced pro-rata, to a minimum of 0,5 g.

8.2.2 Conditioning

Draw air through the apparatus at a rate of 50 ml/min for 10 min. Stop the circulation of air and close the ends of the carbon dioxide absorption tube and the outlet end of the hydrogen sulfide absorption tube connected to it. Remove the carbon dioxide absorption tube and wipe it with a clean, dry, lint-free cloth.

Under certain conditions of humidity, wiping the absorbers with a cloth can induce a static charge, which, if significant, could affect the weighing. Consideration should therefore be given to employing static eliminators.

Allow the tube to cool to the balance room temperature and weigh it at 10 min intervals until a constant weight is obtained, i.e. two successive weighings do not differ by more than 1 mg.

Tubes used for the absorption of carbon dioxide, particularly Midvale tubes, cool slowly and up to 60 min should be allowed before weighing. When not connected to the apparatus, the tubes should be protected from atmospheric contamination by closing the taps and fitting guard seals of plugged rubber tubing to the open limbs. It is usual to weigh the absorption tubes without guard seals, after wiping.

8.2.3 Reaction and completion

Reconnect the absorption tube to the apparatus and open the stopcocks or other closures in the absorption train. Place 25 ml of the hydrochloric acid (5.1) in the tap funnel, open the air vent and admit the acid to the reaction flask. Close the air vent and draw air through the system at a rate of about 50 ml/min. Raise the temperature of the liquid in the reaction flask slowly so that it boils after about 15 min. Continue boiling for a further 30 min, the rate of boiling being adjusted so that the condenser is not overloaded. Turn off the heating source, stop the circulation of air, remove the absorption tube, wipe clean and weigh as before (see 8.2.2).

9 Expression of results

Results may be expressed in terms of carbonate carbon content or carbon dioxide content, as appropriate, as follows.

The carbonate carbon content of the sample, W_C, as analysed, expressed as a percentage by mass, is given by Formula (1):

$$W_C = \frac{27{,}29 \times m_2}{m_1} \quad (1)$$

The carbon dioxide content of the sample, W_{CO_2}, as analysed, expressed as a percentage by mass, is given by Formula (2):

$$W_{CO_2} = \frac{100 \times m_2}{m_1} \quad (2)$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the increase in mass, in grams, of the absorption tube.

NOTE See Annex A for the derivation of factors used in calculations in this document.

Report the results as the mean of duplicate determinations to the nearest 0,01 %.

The results of the determination described in this document are reported on an "air-dried" basis. Calculation of results to other bases shall be in accordance with ISO 1170.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out at different times within a short interval, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same analysis sample, should not differ by more than the values shown in Table 1 or Table 2, as appropriate.

10.2 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, should not differ by more than the values shown in Table 1 or Table 2, as appropriate.

Table 1 — Precision as carbonate carbon content

Carbonate carbon	Maximum acceptable differences between results (calculated to the same moisture content)	
	Same laboratory (repeatability)	Different laboratories (reproducibility)
Up to 0,4 % 0,4 % and over	0,02 % absolute 5 % relative	0,04 % absolute 10 % relative

Table 2 — Precision as carbon dioxide content

Carbon dioxide	Maximum acceptable differences between results (calculated to the same moisture content)	
	Same laboratory (repeatability)	Different laboratories (reproducibility)
Up to 1 % 1 % and over	0,05 % absolute 5 % relative	0,1 % absolute 10 % relative

11 Test report

The test report shall include the following:

a) reference to this document, i.e. ISO 925;

b) an 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 document, or regarded as optional;

f) the date of the test.