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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

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Foreword

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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).

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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, *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