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Coal and coke — Determination of phosphorus — Reduced molybdophosphate photometric method

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

This third edition cancels and replaces the second edition (ISO 622:2016), which has been technically revised.

The main changes are as follows:

— the title and scope have been modified to specifically refer to coal;

— the normative references have been updated;

— Formulae (1) and (2) have been modified;

— a new Formula (3) has been added in Clause 9;

— a new test report clause (Clause 11) has been added.

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.

Coal and coke — Determination of phosphorus — Reduced molybdophosphate photometric method

1 Scope

This document specifies a reduced molybdophosphate photometric method for the determination of the total phosphorus mass fraction of hard coal, lignites and coke. Two methods for taking the phosphorus into solution are specified, namely extraction from the coal or coke ash with acid or by repeated oxidation of the coal or coke, by acid, to remove carbonaceous matter.

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 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 1171, *Coal and coke — Determination of ash*

ISO 13909-2, *Coal and coke — Mechanical sampling — Part 2: Sampling of coal from moving streams*

ISO 13909-3, *Coal and coke — Mechanical sampling — Part 3: Sampling of coal from stationary lots*

ISO 13909-4, *Coal and coke — Mechanical sampling — Part 4: Preparation of test samples of coal*

ISO 13909-5, *Coal and coke — Mechanical sampling — Part 5: Sampling of coke from moving streams*

ISO 13909-6, *Coal and coke — Mechanical sampling — Part 6: Preparation of test samples of coke*

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

4.1 Extraction

Method 1: Removal of carbonaceous material by ashing in a muffle furnace under specified conditions, and extraction of phosphorus by treatment of the ash obtained with hydrofluoric and sulfuric acids.

Method 2: Removal of carbonaceous material by repeated oxidation with nitric acid in the presence of sulfuric acid.

4.2 Determination

Addition of ammonium molybdate and ascorbic acid solution to the acid solution. Measurement of the absorbance of the resulting blue solution by a suitable optical instrument.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Hydrofluoric acid, approximately 400 g/l solution.

WARNING — Aqueous hydrofluoric acid is a highly corrosive liquid which attacks glass; the vapour is irritant and toxic. Its reaction with the skin and eyes is strongly corrosive, producing severe and painful burns which may not be immediately evident and which respond slowly to treatment. The solution shall be handled only inside a well-ventilated fume cupboard. In the event of contact or suspected contact, flood with water and seek immediate medical attention. The manufacturer's literature should be consulted for further information.

WARNING — Preparation of solutions using sulfuric acid can generate significant heat. Generation of heat can be reduced by adding sulfuric acid as the last component of a solution mixture.

5.2 Sulfuric acid, concentrated, ρ 1,84 g/ml, approximately 98 % mass concentration solution.

5.3 Sulfuric acid, solution, prepared by diluting approximately 490 g of sulfuric acid (5.2) to a volume of 1 l with water (5).

5.4 Nitric acid, concentrated, ρ 1,42 g/ml, approximately 70 % mass concentration solution.

5.5 Ammonium molybdate, 60 g/l solution.

5.6 Ascorbic acid, 50 g/l solution.

Prepare the solution fresh daily.

5.7 Antimony potassium tartrate ($KSbO{\cdot}C_4H_4O_6$), 1,36 g/l solution.

5.8 Reagent blank solution.

Mix 25 ml of the sulfuric acid solution (5.3), 10 ml of the ammonium molybdate solution (5.5), 10 ml of the ascorbic acid solution (5.6) and 5 ml of the antimony potassium tartrate solution (5.7). Prepare fresh immediately before use.

5.9 Phosphorus, standard stock solution corresponding to 0,100 mg/ml.

Determine the mass by weighing, to the nearest 0,000 1 g, 0,439 2 g of potassium dihydrogen monophosphate (KH_2PO_4) (dried at 110 °C for 1 h) and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

5.10 Phosphorus, standard working solution corresponding to 1 µg/ml.

Transfer 10 ml of the standard phosphorus solution (5.9) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Prepare fresh immediately before use.