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Solid mineral fuels — Determination of phosphorus content — Reduced molybdophosphate photometric method

Combustibles minéraux solides — Dosage du phosphore — Méthode photométrique au molybdophosphate réduit

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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 on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 27, *Solid minerals fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 622:1981), which has been technically revised. This document incorporates changes related to references and other minor items following its systematic review.

Solid mineral fuels — Determination of phosphorus content — Reduced molybdophosphate photometric method

1 Scope

This document specifies a reduced molybdophosphate photometric method for the determination of the total phosphorus content 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, *Solid mineral fuels — Determination of ash*

ISO 18283, *Hard coal and coke — Manual sampling*

3 Terms and definitions

No terms and definitions are defined in this document.

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

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

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

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 with hydrofluoric and sulphuric acids.

Method 2: Removal of carbonaceous material by repeated oxidation with nitric acid in the presence of sulphuric 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 action on 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 should 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.

5.2 Sulphuric acid, approximately 490 g/l solution.

5.3 Sulphuric acid, concentrated, ρ 1,84 g/ml, approximately 98 % (m/m) solution.

5.4 Nitric acid, concentrated, ρ 1,42 g/ml, approximately 70 % (m/m) 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 solution.

Mix 25 ml of the sulphuric acid solution (5.2), 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 g of P per litre.

Weigh, 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.

1 ml of this standard stock solution contains 0,100 mg of P.

5.10 Phosphorus, standard working solution corresponding to 1 mg of P per litre.

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.

1 ml of this standard working solution contains 1 µg of P.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Muffle furnace, as specified in ISO 1171.

6.2 Dish, of silica, porcelain or platinum, as specified in ISO 1171.