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Coal and coke — Determination of chlorine using Eschka mixture

Charbon et coke — Dosage du chlore à l'aide du mélange Eschka

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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).

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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 fourth edition cancels and replaces the third edition (ISO 587:1997), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— updating of referenced documents;

— adding of the provision of terms and definitions.

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 chlorine using Eschka mixture

1 Scope

This document specifies a method of determining the chlorine content of hard coal, brown coals and lignites, and coke using Eschka mixture.

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 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 ignited in intimate contact with Eschka mixture in an oxidizing atmosphere to remove combustible matter and to convert the chlorine to alkaline chlorides. These are extracted with nitric acid or water and determined by either the Volhard or the Mohr method, or by potentiometric titration using an Ion Selective Electrode (ISE).

5 Reagents

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

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

5.1 For all methods

5.1.1 Eschka mixture.

Mix two parts by mass of light, calcined magnesium oxide with one part of anhydrous sodium or potassium carbonate. The mixture shall entirely pass a test sieve of 0,212 mm nominal aperture.

5.1.2 Nitric acid, concentrated, chlorine-free, mass fraction approximately 70 %.

5.2 For the Volhard and ISE methods

5.2.1 Silver nitrate, standard volumetric solution, $c(AgNO_3)$ = 0,025 mol/l.

Heat crushed, crystalline silver nitrate at 125 °C for two to three hours. Dissolve 4,247 g in water and dilute to 1 l. Store in an amber glass bottle.

5.3 For the Volhard method only

5.3.1 3,5,5-Trimethylhexan-1-ol.

5.3.2 *n*-Hexan-1-ol.

5.3.3 Potassium thiocyanate, standard volumetric solution, $c(KSCN) \approx 0{,}025$ mol/l.

Dissolve 2,4 g of potassium thiocyanate in water and dilute to 1 l. Titrate against the silver nitrate solution (5.2.1) and calculate the exact equivalence.

5.3.4 Iron (III) alum (ammonium iron (III) sulfate) indicator, saturated solution.

Saturate 100 ml of water with approximately 125 g of iron (III) alum $[(NH_4)_2SO_4 \cdot Fe_2(SO_4)_3 \cdot 24H_2O]$ and add sufficient nitric acid (5.1.2) to remove the brown colour.

5.4 For the Mohr method only

5.4.1 Silver nitrate, standard volumetric solution, $c(AgNO_3)$ = 0,050 mol/l.

Weigh 8,494 g of silver nitrate, dried as in (5.2.1), dissolve in water and dilute to 1 l. Store in an amber glass bottle.

5.4.2 Potassium chromate, indicator solution.

Dissolve 5 g of potassium chromate in 100 ml of water.

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.

6.3 Electrically heated muffle furnace, capable of being maintained at a temperature of 675 °C ± 25 °C, with adequate ventilation.

6.4 Crucible, of platinum, silica or glazed porcelain, of capacity approximately 25 ml.