
**Solid mineral fuels — Determination of
extractable metals in dilute hydrochloric
acid**

*Combustibles minéraux solides — Détermination des métaux
extractibles dans l'acide chlorhydrique dilué*

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Published in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 1952 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 1952:1976), which has been technically revised.

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Introduction

Sodium, potassium, calcium, iron and magnesium are commonly present in solid mineral fuel. They can contribute significantly to coal-utilization problems, such as boiler-tube fouling and ash disposal; they also affect product quality and gaseous emissions from combustion processes.

Research indicates that the amounts of alkaline and earth-alkaline metals extractable with dilute hydrochloric acid are more closely related to the fouling/slugging properties of solid mineral fuel than are the total amounts of the metals.

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Solid mineral fuels — Determination of extractable metals in dilute hydrochloric acid

1 Scope

This International Standard describes the extraction from solid mineral fuels by dilute hydrochloric acid and determination of extractable sodium, potassium, calcium, iron and magnesium.

2 Normative references

The following referenced documents are indispensable for the application 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 5069-2, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*

ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

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*

3 Principle

A portion of the sample is extracted with hot, dilute hydrochloric acid after wetting of the solid mineral fuel with alcohol. All forms of sodium, potassium, magnesium, calcium, iron that are exchangeable or extractable with the hydrochloric acid are extracted from the coal. After removal of the residual solid mineral fuel by filtration, the concentrations of the elements in the filtrate are determined by atomic absorption spectrometry.

The concentrations of the elements may also be determined by ICP-OES if the instrument has been validated for the quantification in an appropriate manner.

4 Reagents and materials

Use analytical reagent grade chemicals. Other grades can be used, provided it is documented that the reagent is of sufficiently high purity to permit its use without adversely affecting the accuracy of the determination.

4.1 Water, high-purity deionized water as defined in ISO 3696, or as defined by the equivalent national standard.

4.2 Ethanol.

4.3 Hydrochloric acid (HCl).

4.4 Hydrochloric acid, concentrated, $\rho_{20} = 1,19$ g/ml.

4.5 Hydrochloric acid solution, prepared by mixing 1 part concentrated hydrochloric acid (4.4) with 1 part deionized water (4.1).

DANGER — The addition of the strong acid, HCl, to water liberates a significant amount of heat and can cause a violent reaction.

4.6 Nitric acid (HNO₃), concentrated, $\rho_{20} = 1,42$ g/ml.

4.7 Lanthanum oxide (La₂O₃).

4.8 Lanthanum solution.

Dissolve 23,4 g of La₂O₃ (4.7) in 350 mL of deionized water (4.1). Slowly add 50 mL of concentrated nitric acid. Stir until solution is completely mixed, transfer to a 1 l volumetric flask, dilute to the mark with water.

DANGER — The addition of the strong acid, HNO₃, to water liberates a significant amount of heat and can cause a violent reaction.

NOTE The lanthanum solution is used as an ionization suppressant during calcium determination. It is possible to use other commonly used ionization suppressants, e.g. cesium, as an alternative.

4.9 Magnesium perchlorate, anhydrous, $-2,0$ mm $+0,7$ mm, used as a desiccant.

DANGER — Regeneration of magnesium perchlorate by oven drying should not be attempted because of the risk of explosion. Magnesium perchlorate is a strong oxidant, and should be kept away from organic materials such as oils, greases and organic solvents.

4.10 Stock solutions

4.10.1 Sodium chloride (NaCl).

4.10.2 Sodium solution, 1 000 mg/L.

Dissolve 2,542 g of sodium chloride (4.10.1) (previously dried at 110 °C for 1 h) in 200 mL of deionized water (4.1), stir until solution is completely mixed, transfer to a 1 l volumetric flask, dilute to the mark with water.

4.10.3 Potassium chloride (KCl).

4.10.4 Potassium solution, 1 000 mg/L.

Dissolve 1,907 g of potassium chloride (4.10.3) (previously dried at 110 °C for 1 h) in 200 ml of water (4.1), transfer to a 1 l volumetric flask, dilute to the mark with water.

4.10.5 Calcium carbonate (CaCO₃).

4.10.6 Calcium carbonate solution, 1 000 mg/L.

Dissolve 2,497 g of calcium carbonate (4.10.5) (previously dried at 110 °C for 1 h) in a solution containing 10 ml of concentrated hydrochloric acid (4.4) and 200 ml of deionized water (4.1), stir until solution is

completely mixed. Bring to a gentle boil to remove excess carbon dioxide. Cool the solution, transfer to a 1 l volumetric flask, dilute to the mark with water.

4.10.7 Magnesium oxide.

4.10.8 Magnesium solution, 1 000 mg/L.

Place 1,658 g of magnesium oxide (4.10.7) (previously heated at 1 000 °C for 1 h) in solution containing 10 ml of concentrated hydrochloric acid (4.4) and 200 ml of deionized water (4.1), stir until solution is completely mixed. Warm until dissolved, cool, transfer to a 1 l volumetric flask, dilute to the mark with water.

4.10.9 Iron, minimum purity 99,8 % Fe.

4.10.10 Iron solution, 1 000 mg/L.

Place 1,000 g of pure iron (4.10.9) in a solution containing 20 ml of concentrated hydrochloric acid (4.4) and 20 ml of deionized water (4.1), stir until solution is completely mixed. Heat to boiling and gently simmer until dissolved. Cool, transfer to a 1 l volumetric flask, dilute to the mark with deionized water.

4.10.11 Commercial stock solutions, optional, certified stock solutions with a concentration of the ion of interest of 1 000 mg/l that can be used as an alternative to preparing stock solutions.

4.11 Calibration solutions, prepared with the volumes of reagents given in Table 1.

Prepare calibration solution A, by adding 40 ml of lanthanum solution (4.8) and 32 ml of hydrochloric acid solution (4.5) to a 1 l volumetric flask. Add 1 ml of each stock solution and stir until the solution is completely mixed. Dilute to the mark with water.

Prepare calibration solutions B, C, D, E and the calibration BLANK in the same way as calibration solution A using the volumes of each stock solution shown in Table 1.

Additional calibration solutions within the range of those described here may be prepared in the same manner, in order to increase the number of points on the calibration graph. This can be necessary because of the wide ranges of analyte concentrations likely to be encountered, and also because of the variations in spectrometer responses.

The calibration solutions should be stored in screw-top polyethylene bottles. They should be discarded after three months.

Table 1 — Volumes of reagents for calibration solutions

Calibration solution	Concentration of calibration solution mg/l	Volume of stock solution ml	Volume of lanthanum solution ml	Volume of hydrochloric acid solution ml
A	1	1	40	32
B	2	2	40	32
C	3	3	40	32
D	4	4	40	32
E	5	5	40	32
BLANK	0	0	40	32

4.12 Filter paper, medium-textured filter paper.