International Standard

ISO 16965

Environmental solid matrices — Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)

Matrices solides environnementales — Détermination des éléments par spectrométrie de masse avec plasma à couplage inductif (ICP-MS)

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Foreword

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This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization of solid matrices,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 16965 cancels and replaces ISO/TS 16965:2013, which has been technically revised.

The main changes are as follows :

— the contents of ISO/TS 16965 and EN 16171 have been merged;

— the scope has been expanded to include treated biowaste, waste, sludge and sediment;

— performance data from an interlaboratory comparison has been added (Annex A).

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.

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Environmental solid matrices — Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)

WARNING — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — Tests conducted according to this document shall be carried out by suitably trained staff.

1 Scope

This document specifies a method for the determination of the following elements in aqua regia, nitric acid or mixture of hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4)/hydrofluoric (HF) acid digests of soil, treated biowaste, waste, sludge and sediment:

aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), cadmium (Cd), calcium (Ca), cerium (Ce), caesium (Cs), chromium (Cr), cobalt (Co), copper (Cu), dysprosium (Dy), erbium (Er), europium (Eu), gadolinium (Gd), gallium (Ga), germanium (Ge), gold (Au), hafnium (Hf), holmium (Ho), indium (In), iridium (Ir), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), lutetium (Lu), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), palladium (Pd), phosphorus (P), platinum (Pt), potassium (K), praseodymium (Pr), rhenium (Re), rhodium (Rh), rubidium (Rb), ruthenium (Ru), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), terbium (Tb), thallium (Tl), thorium (Th), thulium (Tm), tin (Sn), titanium (Ti), tungsten (W), uranium (U), vanadium (V), ytterbium (Yb), yttrium (Y), zinc (Zn), and zirconium (Zr).

NOTE 1 Details on validation are given in Annex A.

This method is also applicable for the determination of major, minor and trace elements in aqua regia and nitric acid digests and in eluates of construction products (EN 17200[7]).

NOTE 2 Construction products include e.g. mineral-based products, bituminous products, metals, wood-based products, plastics and rubbers, sealants and adhesives, paints and coatings.

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 17294-1, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General requirements*

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/

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4 Principle

Digests of soil, treated biowaste, waste, sludge and sediments with nitric acid, aqua regia (see EN 16173[5], and ISO 54321[1]) or hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4) or hydrofluoric (HF) acid mixture (EN 13656)[2], are analysed by ICP-MS to obtain a multi-elemental determination of analytes.

The method measures ions produced by a radiofrequency inductively coupled plasma. Analyte species originating in the digest solution are nebulised and the resulting aerosol is transported by argon gas into the plasma. The ions produced by the high temperatures of the plasma are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer, sorted according to their mass-to-charge ratios and quantified with a detector (e.g. channel electron multiplier).

NOTE 1 For the determination of tin, only aqua regia extraction applies (e.g. ISO 54321[1], EN 13656[2]).

NOTE 2 When tetrafluoroboric (HBF_4) is used in the acid mixture, boron cannot be determined.

The working range depends on the matrix and the interferences encountered.

The method detection limit of the method is between 0,1 mg/kg dry matter and 2,0 mg/kg dry matter for most elements. The limit of detection will be higher in cases where the determination is likely to be interfered (see Clause 5) or in case of memory effects (see e.g. ISO 17294-1).

The method has been validated for the elements given in Table A.2 (sludge), Table A.3 (compost) and Table A.4 (soil).

5 Interferences

5.1 General

Interferences shall be assessed, and valid corrections applied. Interference correction shall include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

Detailed information on spectral and non-spectral interferences is given in ISO 17294-1.

5.2 Spectral interferences

5.2.1 Isobaric elemental interferences

Isobaric elemental interferences are caused by isotopes of different elements of closely matched nominal mass-to-charge ratio and which cannot be separated due to an insufficient resolution of the mass spectrometer in use (e.g. ^{114}Cd and ^{114}Sn).

Element interferences from isobars can be corrected by considering the influence from the interfering element (see ISO 17294-1). The isotopes used for correction shall be free of interference. Correction options are often included in the software supplied with the instrument. Common isobaric interferences are given in Annex B, Table B.1.

5.2.2 Isobaric molecular interferences and doubly-charged ion interferences

Isobaric molecular interferences and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Examples include $^{40}Ar^{35}Cl^+$ and $^{40}Ca^{35}Cl^+$ ion on the ^{75}As signal or $^{98}Mo^{16}O^+$ ions on the $^{114}Cd^+$ signal. Natural isotope abundances are available from the literature.

The accuracy of correction equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been found to be reliable, e.g. oxide levels can vary with operating conditions. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, this shall be determined by measuring

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the interference solution just before the sequence is started. The validity of the correction coefficient should be checked at regular intervals within a sequence.

Another possibility to remove isobaric molecular interferences is the use of an instrument with collision/reaction cell technology and further extended to triple quadrupole technology facilitating an even more effective use of reactive gases for interference removal. The use of high resolution ICP-MS allows the resolution of these interferences and additionally double-charged ion interferences.

The response of the analyte of interest shall be corrected for the contribution of isobaric molecular interferences and doubly charged interferences if their impact can be higher than three times the detection limit or higher than half the lowest concentration to be reported.

More information about the use of correction factors is given in ISO 17294-1.

5.2.3 Non-spectral interferences

Physical interferences are associated with sample nebulisation and transport processes as well as with ion-transmission efficiencies. Nebulisation and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Solids can be deposited on the nebuliser tip of a pneumatic nebuliser and on the cones.

A low level of total dissolved solids should be kept, to minimise deposition of solids in the sample introduction system of the plasma torch. An internal standard can be used to correct for physical interferences if it is carefully matched to the analyte, so that the two elements are similarly affected by matrix changes. Other possibilities to minimise non-spectral interferences are matrix matching, particularly matching of the acid concentration, and standard addition.

When intolerable physical interferences are present in a sample, a significant suppression of the internal standard signals will be observed. The relative response of the internal standard in all solutions shall be ±30 % of the internal standard in the preceding blank or continuous calibration verification (CCV) solution (11.3). Dilution of the sample (e.g. fivefold) usually eliminates the problem.

6 Reagents

For the determination of elements at trace and ultra-trace level, the reagents shall be of adequate purity. The concentration of the analyte or interfering substances in the reagents and the water should be negligible compared to the lowest concentration to be determined.

6.1 Water, with an electrical conductivity less than 0,1 $mS \cdot m^{-1}$ (equivalent to resistivity greater than 0,01 MΩ m at 25 °C). The water used should be obtained from a purification system that delivers ultrapure water having a resistivity greater than 0,18 $M\Omega \cdot m$ (usually expressed by manufacturers of water purification systems as 18 $M\Omega \cdot cm$).

6.2 Nitric acid, HNO_3, $c(HNO_3)$ approximately 15 mol/l, $w(HNO_3)$ approximately mass fraction of 65 % to 70 %.

6.3 Hydrochloric acid, HCl, $c(HCl)$ approximately 12 mol/l, $w(HCl)$ approximately mass fraction of 35 % to 37 %.

6.4 Tetrafluoroboric acid (HBF_4), $c(HBF_4)$ approximately 6 mol/l, $w(HBF_4)$ approximately mass fraction of 38 % to 48 %.

6.5 Hydrofluoric acid (HF), $c(HF)$ approximately 23 mol/l, $w(HF)$ approximately mass fraction of 40 % to 45 %.

6.6 Boric acid ($B(OH)_3$), solid.

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