SLOVENSKI STANDARD
oSIST prEN ISO 16965:2024

01-september-2024

Trdni matriksi v okolju - Določanje elementov z masno spektrometrijo z induktivno sklopljeno plazmo (ICP-MS) (ISO/DIS 16965:2024)

Environmental solid matrices - Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS) (ISO/DIS 16965:2024)

Feststoffe in der Umwelt - Bestimmung von Elementen mittels Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) (ISO/DIS 16965:2024)

Matrices solides environnementales - Détermination des éléments par spectrométrie de masse avec plasma induit par haute fréquence (ICP-MS) (ISO/DIS 16965:2024)

Ta slovenski standard je istoveten z: **prEN ISO 16965**

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13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

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ISO/DIS 16965

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

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization.*

This second edition cancels and replaces the first edition (ISO/TS 16965:2013), which has been technically revised.

The main changes compared to the previous edition are as follows :

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

— The scope has been widened to include treated biowaste, waste, sludge and sediment;

— The document has been developed parallel with CEN according to the Vienna Agreement;

— validation data has been added:

— repeatability and reproducibility data have been added from a European interlaboratory comparison organized by the German Federal Institute for Materials Research and Testing BAM in 2013 (see Annex A).

— repeatability and reproducibility data have been reported as a result of the European interlaboratory comparison organized by VITO NV in Mol (Belgium) and Synlab Analytics & Services B.V. in Rotterdam (The Netherlands) for the validation of EN 13656, *Digestion with a hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4) or hydrofluoric (HF) acid mixture for subsequent determination of elements*. The validation was performed on the following types of matrices: city waste incineration ash (BCR176/ BCR176R), ink waste sludge (organic matrix), electronic industry sludge (“metallic” matrix), sediment, coal fly ash, steel slag, copper slag, city waste incineration fly ash ("oxidised" matrix), city waste incineration bottom ash ("silicate" matrix), sewage sludge (BCR 146R)

— repeatability and reproducibility data have been reported as a result of the European interlaboratory comparison organized by VITO NV in Mol (Belgium) and Synlab Analytics & Services B.V. in Rotterdam (The Netherlands) for the validation of EN ISO 54321, *Soil, treated biowaste, sludge and waste — Digestion of aqua regia soluble fraction of elements*. The validation was performed on the following types of matrices: Municipal sludge, Industrial sludge, Sludge from electronic industry, Ink waste sludge, Sewage sludge, Biowaste, Compost, Composted sludge, Agricultural soil, Sludge amended soil, Waste City waste incineration fly ash ("oxidised" matrix), City waste incineration bottom ash ("silicate" matrix), Ink waste

sludge (organic matrix), Electronic industry sludge ("metallic" matrix), BCR 146R (sewage sludge), BCR 176 (city waste incineration ash)

— a reference is made to repeatability and reproducibility data obtained from a European interlaboratory comparison organized by CEN TC 351 for the validation of EN 17200, *Construction products: Assessment of release of dangerous substances — Analysis of inorganic substances in digests and eluates — Analysis by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS).*

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.

Introduction

This document is applicable and validated for several types of matrices as indicated in Table 1 (see Annex A for the results of validation of sludge, biowaste and soil).

Table 1 — Matrices for which this document is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge Industrial sludge Sludge from electronic industry Ink waste sludge Sewage sludge
Biowaste	Compost Composted sludge
Soil	Agricultural soil Sludge amended soils
Waste	City waste incineration fly ash ("oxidised" matrix) City waste incineration bottom ash ("silicate" matrix) Ink waste sludge (organic matrix) Electronic industry sludge ("metallic" matrix) BCR 146R (sewage sludge) BCR 176 (city waste incineration ash)
Sediments	ISE 859 (Sediment from de Bilt / Netherlands)

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 — It is absolutely essential that tests conducted according to this document 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).

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 4) or in case of memory effects (see e.g. EN ISO 17294-1).

The method has been validated for the elements given in Table A.1 (sludge), Table A.2 (compost) and Table A.3 (soil). The method is applicable for the other elements listed above, provided the user has verified the applicability.

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)[2].

NOTE 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.

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

3 Principle

Digests of soil, treated biowaste, waste, sludge and sediments with nitric acid, aqua regia (see EN 16173, and EN ISO 54321) or hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4) or hydrofluoric (HF) acid mixture (EN 13656) are analysed by ICP-MS to get 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 For the determination of tin only aqua regia extraction applies (e.g., EN ISO 54321, EN 13656).

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

4 Interferences

4.1 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 EN ISO 17294-1.

4.2 Spectral interferences

4.2.1 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 EN ISO 17294-1). The isotopes used for correction shall be free of interference if possible. Correction options are often included in the software supplied with the instrument. Common isobaric interferences are given in Table B.1.

4.2.2 Isobaric molecular and doubly-charged ion interferences

Isobaric molecular 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 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 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.