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**Kakovost tal - Določevanje elementov v sledovih z masno spektrometrijo z induktivno sklopljeno plazmo (ICP-MS)**

Soil quality - Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)

Qualité du sol - Détermination des éléments en traces par spectrométrie de masse avec plasma induit par haute fréquence (ICP-MS)

**Ta slovenski standard je istoveten z: ISO/TS 16965:2013**

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# TECHNICAL SPECIFICATION

# ISO/TS 16965

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## Soil quality — Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)

*Qualité du sol — Détermination des éléments en traces par spectrométrie  
de masse avec plasma induit par haute fréquence (ICP-MS)*

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## ISO/TS 16965:2013(E)

### 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. [www.iso.org/directives](http://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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

## Introduction

ISO/TS 16965 is based upon CEN/TS 16171, *Sludge, treated biowaste and soil — Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)*, which was developed by CEN/TC 400, *Project Committee — Horizontal standards in the fields of sludge, biowaste and soil*.

This Technical Specification is applicable and validated for several types of matrices as indicated in [Table 1](#).

**Table 1 — Matrices for which this Technical Specification is applicable and validated**

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost
Soil	Sludge-amended soils

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# Soil quality — Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)

**WARNING** — Persons using this Technical Specification should be familiar with usual laboratory practice. This Technical Specification 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 Technical Specification be carried out by suitably trained staff.

## 1 Scope

This Technical Specification specifies a method for the determination of the following elements in *aqua regia* or nitric acid digests or other extraction solutions of sludge, treated biowaste and soil:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), cadmium (Cd), calcium (Ca), cerium (Ce), cesium (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 limit of detection 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 have interferences (see [Clause 4](#)) or in the case of memory effects (see e.g. 8.2 of ISO 17294-1:2004).

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11466, *Soil quality — Extraction of trace elements soluble in aqua regia*

ISO 16729, *Soil quality — Digestion of nitric acid soluble fractions of elements*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 17294-1:2004, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

## 3 Principle

Digests with nitric acid or *aqua regia* of samples of sludge, treated biowaste or soil (see ISO 11466 and ISO 16729) are analysed by ICP-MS to get a multi-elemental determination of analytes.

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The method measures ions produced by a radio-frequency 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).

### 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 Clause 6 of ISO 17294-1:2004.

#### 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.  $^{114}\text{Cd}$  and  $^{114}\text{Sn}$ ).

Element interferences from isobars may be corrected by taking into account the influence from the interfering element (see ISO 17294-1:2004). 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 A.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  $^{40}\text{Ar}^{35}\text{Cl}^+$  and  $^{40}\text{Ca}^{35}\text{Cl}^+$  ion on the  $^{75}\text{As}$  signal or  $^{98}\text{Mo}^{16}\text{O}^+$  ions on the  $^{114}\text{Cd}^+$  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. The use of high resolution ICP-MS avoids 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.

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

##### 4.2.3 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