

PD ISO/TS 16965:2013



BSI Standards Publication

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

**bsi.**

...making excellence a habit.™

### **National foreword**

This Published Document is the UK implementation of ISO/TS 16965:2013.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2013.  
Published by BSI Standards Limited 2013

ISBN 978 0 580 74389 4  
ICS 13.080.10

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 October 2013.

### **Amendments/corrigenda issued since publication**

<b>Date</b>	<b>Text affected</b>
-------------	----------------------

---

**TECHNICAL  
SPECIFICATION**

**ISO/TS  
16965**

First edition  
2013-09-15

---

---

**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)*



Reference number  
ISO/TS 16965:2013(E)

© ISO 2013



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2013

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Principle</b> .....	<b>1</b>
<b>4 Interferences</b> .....	<b>2</b>
4.1 General.....	2
4.2 Spectral interferences.....	2
<b>5 Reagents</b> .....	<b>3</b>
<b>6 Apparatus</b> .....	<b>5</b>
6.1 General requirements.....	5
6.2 Mass spectrometer.....	6
6.3 Mass-flow controller.....	6
6.4 Nebuliser with variable speed peristaltic pump.....	6
6.5 Gas supply.....	6
6.6 Storage bottles, for the stock, standard, calibration and sample solutions.....	6
<b>7 Procedure</b> .....	<b>7</b>
7.1 Test sample solution.....	7
7.2 Test portion solution.....	7
7.3 Instrument set up.....	7
7.4 Calibration.....	8
7.5 Sample measurement.....	8
<b>8 Calculation</b> .....	<b>9</b>
<b>9 Expression of results</b> .....	<b>9</b>
<b>10 Performance characteristics</b> .....	<b>10</b>
10.1 General.....	10
10.2 Blank.....	10
10.3 Calibration check.....	10
10.4 Internal standard response.....	10
10.5 Interference.....	10
10.6 Recovery.....	10
10.7 Precision.....	10
<b>11 Test report</b> .....	<b>11</b>
<b>Annex A (informative) Selected isotopes and spectral interferences for quadrupole ICP-MS instruments</b> .....	<b>12</b>
<b>Bibliography</b> .....	<b>13</b>

## 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. [www.iso.org/directives](http://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. [www.iso.org/patents](http://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 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**

<b>Matrix</b>	<b>Materials used for validation</b>
Sludge	Municipal sludge
Biowaste	Compost
Soil	Sludge-amended soils

# 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.