SLOVENSKI STANDARD
SIST-TP CEN/TR 16045:2011

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Vsebnost reguliranih nevarnih snovi - Izbira analitskih metod

Construction Products - Assessment of release of dangerous substances - Content of regulated dangerous substances - Selection of analytical methods

Bauprodukte - Bewertung der Freisetzung von gefährlichen Substanzen - Inhalt von geregelten gefährlichen Substanzen - Auswahl von analytischen Verfahren

Produits de construction - Evaluation des émissions de substances dangereuses - Contenu en substances dangereuses réglementées - Sélection des méthodes analytiques

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English Version

Construction Products - Assessment of release of dangerous substances - Content of regulated dangerous substances - Selection of analytical methods

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Contents

Page

Tables

Figures

Foreword

This document (CEN/TR 16045:2010) has been prepared by Technical Committee CEN/TC 351 “Construction Products − Assessment of release of dangerous substances”, the secretariat of which is held by NEN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Under Work Item 6, Mandate M/366 describes the need for CEN standards to test the chemical content of construction products. However, in the first meeting of CEN/BT WG 176, the predecessor to CEN/TC 351 (June 2005, Gouda, The Netherlands), it was decided that a Technical Report (TR) should be drafted first. This TR was for administrative reasons given work item (WI) number 14. The background and guidelines for carrying out this work are outlined in Annex A of this report:

— a wide range of methods exist, focus will be on existing documents;

— one of the criteria for the selection of standard test methods could be the information on validation;

— content determination should not be used for certification of emissions unless this is the only practicable or legally correct solution.

It is emphasized that the focus of the CPD is on the release of dangerous substances, not on content. Content testing may only be applied for product release/emissions certification of a material if emissions testing is prohibitively expensive or when it is specifically required by regulation – for example in the case of banned substances such as certain metals or asbestos.

However, content testing may be useful as a complementary quick screening method for in-house quality control of product emissions (e.g. as a routine check of product uniformity/conformity). Content tests can also be relevant within the scope of the continuous surveillance by the approved bodies or further testing of samples by the manufacturer (see Annex III of the CPD).

Content testing methods may or may not be relevant to predicting the release to soil, surface and groundwater or the emission into indoor air. However, there are precedents for content-testing-type methods being used as a guide to release and emissions.

An example for this concept is the German "regulation" for cementitious materials in contact with drinking water, which includes content values for some trace elements in cements as screening test (German DVGW Worksheet W 347, [2]). These values are not a criterion for exclusion, but the meaning is that leaching tests for trace elements on test pieces (mortar or concrete) are only necessary if the total trace element content in the cement is above these values.

Other examples of standards committees and industries that have followed this route in relation to compounds that could emit into indoor air include paints and varnishes (ref.: EN ISO 17895), wood-based panels (ref.: EN 120 and EN 717-2), toy testing (ref.: EN 71-11:2005, Annex B), the car industry for interior trim components (e.g. Method VDA 278 and similar standards) and hard disk drive manufacturers (various company-specific test protocols for emissions from PC components). Most of these methods use gas extraction at elevated temperatures combined with GC-MS as the analytical approach. This methodology has the advantage that it is similar to the analytical approach used in formal emissions test methods, which means that, in some instances, there is a degree of qualitative and quantitative

correlation between the content-type test method and reference emissions test methods. There are however limitations. The VOC content will unlikely bear any relationship to an emission profile in the case of composite construction products or materials in which the VOCs are encapsulated or otherwise locked-in to the product to prevent emission. Content testing is also not relevant to assessing secondary emissions.

Because of the similarity of the analytical methods for digests and eluates from leaching, for reasons of completeness and efficiency (no separate report necessary) the analysis of eluates from leaching is also covered in this Technical Report. To make a separate report would lead to an almost full duplication of the present report. The additional benefit of addressing both aspects is the coherence that is becoming obvious from Figure 1.

1 Scope

This Technical Report describes appropriate standard test methods for the determination of the content of regulated dangerous substances in construction products. Because of the similarity of the analytical methods for digests and eluates from leaching, the analysis of eluates from leaching is also covered.

This Technical Report is relevant to all substances covered by the provisions of the main body of Mandate M/366, i.e. those included in the work programme for the emission into indoor air, and release to surface water, ground water and soil.

The list of regulated substances provided by the Commission in document "Indicative list of regulated dangerous substances" [1] defines the substances, for which analytical methods for content will in principle be needed. This report will be limited to this list.

NOTE 1 Sampling for content analysis is addressed by applying the relevant product standards and or by applying WI 00351013, *Construction products — Assessment of release of dangerous substances — Complement to sampling* (TR 4) [7] in case the sampling protocol for technical properties does not adequately address requirements in testing posed by the assessment of release to soil, surface and groundwater.

NOTE 2 Based on this selection of appropriate test methods from other fields, horizontal test methods for analysing the chemical content of construction products will be developed as ENs.

NOTE 3 In Annex B a compilation is given of the content regulations for construction products for health or environmental reasons.

2 Abbreviations, terms and definitions

NOTE In this paragraph some of the abbreviations and terms used in this report, are defined.

2.1 Abbreviations

AES	atomic emission spectrometry
CPD	construction products directive
DOC	dissolved organic carbon
DS	dangerous substances
HPLC	high-performance liquid chromatography, high-pressure liquid chromatography
ICP	inductively coupled plasma
GC	gas chromatography
MS	mass spectrometry
OES	optical emission spectrometry
SVOC	semi-volatile organic compounds
TD	thermal desorption
TOC	total organic carbon
VOC	volatile organic compounds

VVOC very volatile organic compounds

NOTE There are several definitions of VVOC, VOC and SVOC in use:

a) by the World Health Organization (WHO):

VVOC
boiling point is in the range from < 0 °C (50 °C to 100 °C)

VOC
boiling point is in the range from (50 °C to 100 °C) to (240 °C to 260 °C)

SVOC
boiling point is in the range from (240 °C to 260 °C) to (380 °C to 400 °C)

b) by European Collaborative Action Report No 18:

VVOC
all compounds which, in a capillary column coated with 100 % dimethylpolysiloxane, are eluted before n-hexane

VOC
all volatile organic compounds which, in a capillary column coated with 100 % dimethylpolysiloxane, are eluted with a retention range between n-hexane and n-hexadecane

SVOC
all semi-volatile organic compounds which, in a capillary column coated with 100 % dimethylpolysiloxane, are eluted with a retention range between n-hexadecane and n-docosane

c) by some national regulations and RL 2004/42/EG:

VOC
any organic compound having an initial boiling point less than or equal to 250 °C measured at a standard pressure of 101,3 kPa

2.2 Terms and definitions

For the purposes of this document, the terms and definitions in the CEN/TC 351 document on Terminology (CEN/TC 351 N 0218, under development) and the following apply.

2.2.1
digest
solution resulting from acid digestion of a test sample

2.2.2
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part (as completely as possible) when reacted with a reagent mixture

NOTE Usually done with a strong, concentrated acid like aqua regia or nitric acid to solve inorganic substances for chemical analysis.

2.2.3
eluate
solution obtained from a laboratory leaching test of a test sample

2.2.4
extract
solution resulting from extraction of a test sample with a solvent

2.2.5
extraction
dissolution of substances from a sample into a solvent for subsequent chemical analysis

NOTE Usually done with an organic solvent to extract organic substances for chemical analysis or for special analysis of inorganic substances.

2.2.6
product matrix
main composition of the product dictating the manner of sample pre-treatment and the type of digestion or extraction for later chemical analysis

NOTE For construction products for example the following product matrices could be distinguished: silica-based products, bituminous products, metals, wood-based products, plastics and rubbers, sealants and adhesives, and paints and coatings.

3 Approach

The first question was how to group content test methods by type of target compound and sample matrix. The separation into different matrix types is related to the nature of the matrix, the ease of size reduction, mode of sample handling and the suitability for digestion by acids or extraction by organic solvents.

It was suggested that most construction products and materials fall into one or more of the following main categories of product matrices:

— silica-based products (S) (includes calcerous and stony like materials);

— bituminous products (B);

— metals (M);

— wood-based products (W);

— plastics and rubbers (P);

— sealants and adhesives (A);

— paints and coatings (C).

EXAMPLES Examples of products that could fall into more than one category are:

1) structured wall paper may contain wood-based material (paper) and plastics (PVC-P foam);

2) fibre-reinforced concrete containing silica (concrete) and wood-based (wood-fibres);

3) painted steel containing metal and paint or a coating;

4) pipes made from mineral-fibre-reinforced PVC (PVC-GF) containing plastics and silica.

When the materials can be separated easily, the product may be considered as consisting of two separately quantified constituents. When a material is made inseparable from the composite it can be regarded as one product to be tested as a whole. In the examples above example 1) and 3) would fall in the first group and 2) and 4) in the second group.

Similarly, the various target chemical analytes can be grouped as follows:

— volatile organics (primary interest for emissions to indoor air);

- semi- and non-volatile organics (interest for both emissions to indoor air and impact to soil and groundwater);
- inorganic substances – Metals and salts (primarily of interest for impact to soil and groundwater).

Product matrices and groups of analytes can also be categorised according to the typical preparation and analytical methods. These include:

- gas extraction of volatile organics from the sample matrix and analysis by GC-MS or HPLC;
- solvent extraction of organics from the sample matrix and analysis by GC-MS or HPLC;
- analysis of metals and salts almost always involves some form of matrix digestion followed by ICP-AES or ICP-MS;
- specific procedures of sample dissolution and analysis are required for some specific inorganic analysed items such as Cr (VI);
- other content properties such as fibres are described as a separate group.

These classifications are used to categorise the various available content test methods in the sections that follow.

Since the methods for analysing the chemical content of digests (inorganic substances) and organic extracts (organic substances) are not very different from the analytical methods needed for analysis of eluates from leaching tests, it is therefore most practical to integrate a summary of possible analytical methods for eluate analysis in the present document (see Figure 1). It must be realised, however, that water extracts from leaching normally require an additional solvent extraction step to concentrate the organic substances of interest for analysis. In addition, the analytical methods used for the analysis of VOC, VVOC and SVOC are similar to the methods used for analysing organic substances in extracts. In Figure 1 below the possible options for content analysis, digestion and extraction as well as trapping volatiles on sorbents is schematically shown.

NOTE 1 There are many standards in the field of the metal alloy composition. In Annex C a list is given. These standards are **not** considered in this report, as these methods are not focused on environmental questions, but rather on impurities of the metal.

NOTE 2 The water quality standards for metal content determination are given in Table 14 "Analytical methods for inorganic substances in eluates" (see 5.2).

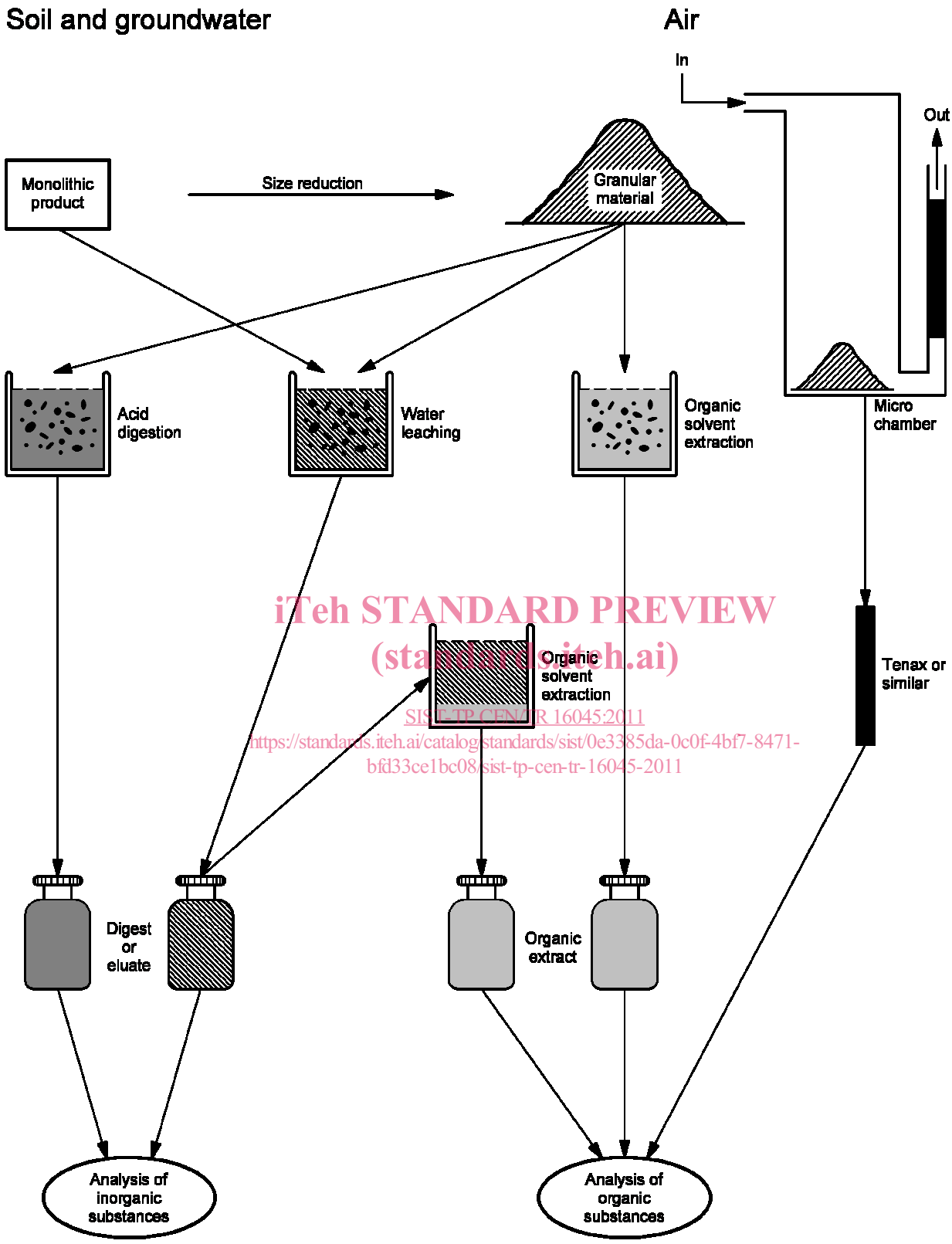

Figure 1 — Principles of horizontal analytical methods

4 Available content test methods

4.1 Introduction

The following tables of test methods are arranged following the approach outlined above.

The order of standards is EN, EN ISO, ISO, national and other documents. National and other methods are only mentioned when there is no EN, EN ISO or ISO available.

NOTE The documents named "CSS xxxxx" have been prepared by CEN/TC 400 "Project Committee − Horizontal standards in the fields of sludge, biowaste and soil" (formerly CEN/BT TF 151). They currently are in the process of being transposed into EN standards.

4.2 Inorganic substances

4.2.1 Major, minor and trace elements

4.2.1.1 General

The inorganic substances comprise all major, minor and trace elements listed in the "Indicative list of regulated dangerous substances" [1].

The methods given will generally be applicable to a wider range of inorganic substances than those listed in the "Indicative list of regulated dangerous substances" [1]. Table 1 is a draft list of metals and their compounds to be considered. The availability of methods for digestion and for analysis is indicated. The methods themselves are presented in more detail in the next paragraphs. 4.2.1.2 deals with digestion methods (Table 2); 4.2.1.3 with analytical methods (Table 3).

NOTE 1 For the analysis of inorganic substances in digests many regulations refer to the methods for the analysis of inorganic substances in water, even though digests have a much higher ionic strength. The methods for the analysis of inorganic substances in water are collected in 5.2.

NOTE 2 Some elements may not be relevant from a regulatory point of view, but may be very important for the characterization of the product. Hence methods for these elements are also given in this table.

4.2.1.2 Digestion methods for major, minor and trace elements

The digestion methods for major, minor and trace elements are given in Table 2. In the table seven different matrices are identified, for which the expected suitability of the listed method or uncertainties therein are indicated. Explanations are given in footnotes at the bottom of the table.

Several methods developed in the environmental field are multi-element methods. So instead of listing all elements in the table "major, minor and trace elements" is given as indication that the digestion methods are suitable for many elements, which may not all have been analysed. Chemical forms cannot be assessed, as the chemical form changes after relative aggressive digestions, such as aqua regia or nitric acid digestion, e.g. Cr (VI) cannot be assessed.

Methods for separation and subsequent analysis of minor or trace substances in metals have not been developed to address environmental concerns, but are mainly intended to measure impurities affecting the technical quality of a material. The methods generally have a narrow scope with the exception of EN 24242. In case of a small impurity of, for instance Ni in Zn, the main component Zn is the one to concentrate on from an environmental perspective and the Ni is of minor importance from an environmental perspective.

Table 1 — Major, minor and trace elements to be considered under the CPD and available test methods

Substance or substance subgroup	Chemical Abstract Service Number (CAS)	Test methods for digestion available [a]	Test methods for analysis available
Multi-element, multi-matrix methods			
Major, minor and trace elements	n/a	CSS 99025A CSS 99025B	 CSS 99026 CSS 99027
Multi-element, single-matrix methods			
Major, minor and trace elements	n/a	CSS 99025A CSS 99025B DIN 53770-16 EN 12506 EN 13346 EN 13656 EN 13657 EN 14242 ISO 6713 ISO 11466 ISO 14869-1 ISO 14869-2 ISO 11047	 CSS 99026 CSS 99027 DIN 53770-16 EN 12506 EN 14242 ISO 11047
Single element methods [b]			
Chromium	7440-47-3	ISO 3856-6	ISO 3856-6
Selenium	7782-49-2	ISO 20280	ISO 20280
Lead and its compounds	7439-92-1	EN 12441-3 IEC 62321 ISO 3856-1 ISO 6503	 ISO 3856-1 ISO 6503
Cadmium	7440-43-9	EN 1122 EN 12441-3 IEC 62321 ISO 3856-4	EN 1122 ISO 3856-4
Nickel and its compounds	7440-02-0	EN 12441-9	EN ISO 17294-2
Mercury and its compounds	7439-97-6	CSS 99030 IEC 62321 ISO 16772	CSS 99030 ISO 16772
Tin	7440-31-5	EN 12441-7 EN 12441-8	EN 12506
Antimony	7440-36-0	EN 13656 ISO 3856-2 ISO 20280	EN ISO 17294-2 ISO 3856-2 ISO 20280
Arsenic	7440-38-2	ISO 20280	ISO 20280
Aluminium	7429-90-5	EN 12441-6 ISO 14869-1 ISO 14869-2	CSS 99026 CSS 99027
Calcium	7440-70-2	ISO 14869-2	CSS 99026 CSS 99027
Silicon	7440-21-3	EN 12441-2 ISO 14869-2	CSS 99026 CSS 99027

[a] Not all methods for analysis can be combined with all methods for extraction mentioned. Individual standards need to be checked.

[b] All multi-element methods can be applied to quantify single elements. Not all methods for analysis go with all methods for extraction mentioned.

Table 2 — Digestion methods for major, minor and trace elements

Standard	Title	Substance(s)	Digest from product matrices [a] [b]						
			S	B	M	W	P	A	C
Multi-element, multi-matrix methods									
CSS 99025A (To be published)	Digestion of soil sludge, biowaste and waste for the extraction of nitric acid soluble elements	Major, minor and trace elements	+	+	+	+	+ #	+ #	+
CSS 99025B (To be published)	Digestion of soil sludge, biowaste and waste for the extraction of aqua regia soluble elements	Major, minor and trace elements	+	+	+	+	+ #	+ #	+
Multi-element, single-matrix methods									
EN 13346	Characterization of sludges – Determination of trace elements and phosphorus – Aqua regia extraction methods	As, Cd, Cr, Cu, Hg, Ni, Pb, Se, Zn, P	+	+	+	+	+ #	+ #	+
EN 13656 [c]	Characterization of waste – Microwave assisted digestion with hydrofluoric (HF), nitric (HNO_3) and hydrochloric (HCl) acid mixture for subsequent determination of elements	Major, minor and trace elements	+	+	+	+	+ #	+ #	+
EN 13657	Characterization of waste – Digestion for subsequent determination of aqua regia soluble portion of elements	Major, minor and trace elements	+	+	+	+	+ #	+ #	+
EN 14242	Aluminium and aluminium alloys – Chemical analysis – Inductively coupled plasma optical emission spectral analysis	Si, Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, Ga, V, Be, Bi, Ca, Cd, Co, Li, Na, Pb, Sb, Sn, Sr, Zr	0	0	+ @	0	0	0	0
EN 62321	Electrotechnical products – Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)	Pb, Hg, Cd, Cr (VI), PBB, PBDE	0	0	+?	0	0	0	0
ISO 6713	Paints and varnishes – Preparation of acid extracts from paints in liquid or powder form	Acid extracts	0	0	0	0	0	0	+
ISO 11047	Soil quality – Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc – Flame and electrothermal atomic absorption spectrometric methods	Cd, Cr, Co, Cu, Pb, Mn, Ni, Zn	+	0	0	0	0	0	0
ISO 11466	Soil quality – Extraction of trace elements soluble in aqua regia	Trace elements	+	0	0	0	0	0	0
ISO 14869-1	Soil quality – Dissolution for the determination of total element content – Part 1: Dissolution with hydrofluoric and perchloric acids	Al, Ba, Cd, Ca, Cs, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Sr, V, Zn	+	0	0	0	0	0	0
ISO 14869-2	Soil quality – Dissolution for the determination of total element content – Part 2: Dissolution by alkaline fusion	Na, K, Mg, Ca, Ti, Mn, Fe, Al, Si	+	0	0	0	0	0	0