

Soil, sludge and treated biowaste — Trace elements — Method by inductively coupled plasma - mass spectrometry (ICP MS)

Boden, Schlamm Sedimente und behandelte Bio-Abfälle — Spuren Elementen — Methode mit induktiv gekoppeltes Plasma mit Massen- Spectrometer (ICP-MS)

Sol, boue, sediment et bio déchet traité — Éléments trace — Méthode de spectromètre de masse à source plasma (ICP-MS)

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Foreword

This document has been prepared in the framework of the project Horizontal.

This document is a working document.

The following TC's have been involved in the preparation of the standard:

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

[table to be filled and amended by the standards writer]

Material	Validated	Document
Soil	<input type="checkbox"/>	[reference]
Sludge	<input type="checkbox"/>	
Biowaste	<input type="checkbox"/>	
Soil improvers	Not validated yet	

Introduction

This document is developed in the framework of the project 'Horizontal'. It is the result of a desk study "Determination of elements by ICP-AES and ICP-MS" in Phase I and "Trace elements with ICP-MS: ruggedness test" in Phase II and aims at evaluation of the latest developments in assessing measurement of trace elements in sludge, soil, treated biowaste and neighbouring fields. After discussion with all parties concerned in CEN and selection of a number of test methods described in this study the standard has been developed further as a modular horizontal method and has been validated within in the project 'Horizontal'.

1 Scope

This European Standard describes a horizontal method for the determination of the dissolved elements aluminum, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, cesium, calcium, cerium, chromium, cobalt, copper, dysprosium, erbium, europium, gadolinium, gallium, germanium, gold, hafnium, holmium, indium, iridium, iron, lanthanum, lead, lithium, lutetium, magnesium, manganese, mercury, molybdenum, neodymium, nickel, palladium, phosphorus, platinum, potassium, praseodymium, rubidium, rhodium, ruthenium, samarium, scandium, selenium, silicon, silver, sodium, strontium, sulphur, terbium, tellurium, thorium, thallium, thulium, tin, titanium, tungsten, uranium, vanadium, yttrium, ytterbium, zinc, and zirconium in *aqua regia* and nitric acid digests of treated biowaste, soil, sludge and sediments.

The working range depends on the matrix and the interferences encountered.
In soil, sludge and biowaste, the limit of detection is between 0,1 mg/kg dry weight and 2,0 mg/kg dry weight for most elements (see Annex B).

Note: The limit of detection of most elements will be affected their natural abundance, ionization behavior and by blank contamination and this will depend predominantly on the laboratory air-handling facilities available.

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 i.e. clause 8.2 of ISO 17294-1).

2 Normative references

The following normative documents contain provisions, which, through reference in this text, constitute provisions of <standard number>. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on <standard number> are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

ISO 3696:1987, Water for analytical laboratory use. Specification and test methods.

<*Aqua regia* horizontal standard>. Digestion for the determination of selected elements . Part 1: *Aqua regia* digestion.

<Nitric acid horizontal standard>. Digestion for the determination of selected elements . Part 2: Nitric acid digestion.

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

3 Definitions

For the purposes of this standard, the following terms and definitions apply.

3.1 Instrumental limit of detection (IDL):

Three times the repeatability standard deviation ($3 * Sr$) calculated from multiple readings ($n > 8$) of a (matrix) solution within a single run.

3.2 Method detection limit (MDL):

Three times the within laboratory reproducibility standard deviation ($3 * Sw$) calculated from multiple measurements ($n > 8$) on different days of a solution with low analyte concentrations in a relevant matrix.

4 Symbols and abbreviations

4.1 ICS: Interference check solution

5 Principle

This international standard describes the multi-elemental determination of analytes by ICP-MS in (diluted) nitric acid or *aqua regia* digests. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma. The ions produced by 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). Interferences must be assessed and valid corrections applied. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

6 Interferences

6.1 General

Detailed information on spectral and non-spectral interferences is given in clause 6.1 of EN ISO 17294-1.

6.2 Spectral interferences

6.2.1 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 may be corrected for taking into account the influence from the interfering element (see Table 1). The isotopes used for correction shall be free of interference if possible. Correction options are often included in the instrument software. Common isobaric interferences are given in Table 2.

6.2.2 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}As signal and $^{98}\text{Mo}^{16}\text{O}^+$ ions on the $^{114}\text{Cd}^+$ signal. Natural isotope abundances are available from the literature. However, the most precise coefficients for an instrument will be determined from the ratio of the net isotope signals observed for a standard solution.

The accuracy of these types of 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, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent.

Other possibilities to correct for isobaric and doubly charged ion interferences are the use of an instrument with collision/reaction cell technology or high resolution ICP-MS.

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 instrumental detection limit or higher than half the lowest concentration to be reported.

6.3 Non spectral interferences

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization 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. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the cones.

It is recommended to keep the level of total dissolved solids below 0.2% (2,000 mg/L) to minimize 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 minimize 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 (to less than 30 % of the signals in the calibration solution) will be observed. Dilution of the sample (e.g. fivefold) will usually eliminate the problem.

7 Reagents

7.1 General

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.

Preferably, nitric acid preservation and digestion should be applied in order to minimize interferences by polyatoms. Bi, Hg, Hf, Mo, Sn, Sb, Te, W and Zr may need hydrochloric acid for preservation and digestion.

7.2 Water

Use water according to Grade 1 of ISO 3696 for all sample preparations and dilutions.

7.3 Nitric acid, $c(\text{HNO}_3) = 16 \text{ mol/l}$.

NOTE Nitric acid is available both as $\rho(\text{HNO}_3) = 1,40 \text{ g/ml}$ ($w(\text{HNO}_3) = 650 \text{ g/kg}$) and $\rho(\text{HNO}_3) = 1,42 \text{ g/ml}$ ($w(\text{HNO}_3) =$

690 g/kg). Both are suitable for use in this method provided there is minimal content of the analytes of interest.

7.4 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$; $\rho \sim 1,18 \text{ g/ml}$.

7.5 Element stock solutions

Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, Ir, K, La, Li, Lu, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, Rh, Ru, S, Sb, Sc, Se, Si, Sm, Sn, Sr, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn, Zr, $\rho = 1\ 000 \text{ mg/l}$ each.

Both single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available.

These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

7.6 Anion stock solutions

Cl, $\text{PO}_4\text{-P}$, $\text{SO}_4\text{-S}$, $\rho = 1\ 000 \text{ mg/l}$ each.

Prepare these solutions from the respective acids. The solutions are as well commercially available.

These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

7.7 Multi-element standard solutions

Depending on the scope, different multi-element standard solutions may be necessary. In general, when combining multi-element standard solutions, their chemical compatibility and the possible hydrolysis of the components shall be regarded. Care must be taken to prevent chemical reactions (e.g. precipitation).

The multi-element standard solutions are considered to be stable for several months, if stored in the dark. This does not apply to multi-element standard solutions that are prone to hydrolysis, in particular solutions of Bi, Mo, Sn, Sb, Te, W, Hf and Zr.

Multi-element standard solution A at the mg/l level may contain the following elements:

Ag, Al, As, B, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, La, Li, Mn, Ni, Pb, Rb, Sr, Th, Tl, U, V, Se, Zn. Use nitric acid for stabilization of standard solution A.

Multi-element standard solution B at the mg/l level may contain the following elements:

Au, Mo, Sb, Sn, W, Zr.

Standard solution B needs hydrochloric acid for stabilization.

Mercury standard solutions can be stabilized by adding 1 ppm Au in nitric acid or by adding hydrochloric acid (up to 0.6%).

Note: When Au is used as modifier the instrument is not suitable for accurate Au determination.

Multi-element standard solutions with more elements are allowed provided that these solutions are stable.

7.8 Multi-element calibration solutions

Prepare in one or more steps calibration solutions at the highest concentration of interest.

If more concentration levels are needed prepare those similarly.

Add acids (7.3 – 7.4) to match the acid concentration of samples closely.

If traceability of the values is not established check the validity by comparison with a (traceable) independent standard.

Check the stability of the diluted calibration solutions.

7.9 Internal standard solution

The choice of elements for the internal standard solution depends on the analytical problem. The solution of this/these internal standard(s) should cover the mass range of interest. The concentrations of the selected elements (used as internal standard) should be negligibly low in the digests of samples. The elements In, Lu, Re, Ge and Rh have been found suitable for this purpose.

Generally, a suitable concentration of the internal standard in samples and calibration solutions is 10 - 50 µg/l (or optimized to $\pm 50.000 - 100.000$ counts/s). The use of a collision/reaction cell may require higher concentrations.

7.10 Calibration blank

Prepare the calibration blank by acidifying water (7.2) to the same concentrations of the acids found in the calibration solutions and samples.

7.11 Method blank

The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.12 Optimization solution

The optimization solution serves for mass calibration and for optimization of the instrumental settings, e.g. adjustment of maximal sensitivity with respect to minimal oxide formation rate and minimal formation of doubly charged ions. It should contain elements covering the total mass range, as well as elements prone to a high oxide formation rate or to the formation of doubly charged ions. The composition of the optimization depends on the elements of interest, instrument and manufactures recommendations. An optimization solution containing e.g. Mg, Cu, Rh, In, Ba, La, Ce, U and Pb is suitable. Li, Be and Bi are less suitable because they tend to cause memory effects at higher concentrations. The mass concentrations of the elements used for optimization should allow count rates of more than 10^4 counts per second.

7.13 Interference check solution

The interference check solutions serve to determine the correction factors for the corresponding equations. High demands are made concerning the purity of the basic reagents due to the high mass concentrations. Interference check solutions shall contain all the interferents of practical relevance given in table A.1, at a concentration level at the same range as expected in the samples (see also 12.4).

Leaving out an interferent of table A.1 is permitted if it can be demonstrated that its impact is negligible and lasting.

In extraordinary situations also the other interferents of Table A.1 shall be investigated for relevance.

NOTE

An example of the composition of an interferent check solution is (see ref 1):
 $\omega(\text{Ca}) = 2500 \text{ mg/l}$; $\omega(\text{Cl}^-) = 2000 \text{ mg/l}$; $\omega(\text{P-PO}_4) = 500 \text{ mg/l}$ and $\omega(\text{S-SO}_4) = 500 \text{ mg/l}$ and for digests also $\omega(\text{C}) = 1000 \text{ mg/l}$; $\omega(\text{Fe}) = 500 \text{ mg/l}$; $\omega(\text{Na}) = 500 \text{ mg/l}$ and $\omega(\text{AL}) = 500 \text{ mg/l}$.

8 Apparatus

8.1 General requirements

The stability of samples, measuring, and calibration solutions depends to a high degree on the container material. The material shall be checked according to the specific purpose. For the determination of elements in a very low concentration range (sub ppb), glass or polyvinyl chloride (PVC) should not be used. Instead, it is recommended to use perfluoroalkoxy (PFA), hexafluoroethene propene (FEP) or quartz containers, cleaned with diluted, high quality nitric acid or hot, concentrated nitric acid in a closed system. For the determination of elements in a higher concentration range, HDPE or PTFE containers are also allowed for the collection of samples. Immediately before use, all glassware should be washed thoroughly with diluted nitric acid (e.g. $w(\text{HNO}_3) = 10\%$), and then rinsed several times with water (7.2).

The limit of detection of most elements will be affected by blank contamination and this will depend predominantly on the laboratory air-handling facilities available.

The use of piston pipettes is permitted and also enables the preparation of smaller volumes of calibration solutions. The application of dilutors is also allowed. Every charge of pipette tips and single-use plastic vessels shall be tested for impurities.

NOTE: Dispensing of a volume of less than 50 μl by means of a pipet should be avoided.

For more detailed information on the instrumentation see clause 5 of ISO 17294-1.

8.2 Mass spectrometer

A mass spectrometer with inductively coupled plasma (ICP) suitable for multi-element and isotope analysis is required. The spectrometer should be capable of scanning a mass range from 5 m/z (AMU) to 240 m/z (AMU) with a resolution of at least 1 m_r/z peak width at 5% of peak height (m_r = relative mass of an atom species; z = charge number). The instrument may be fitted with a conventional or extended dynamic range detection system.

Quadrupole ICP-MS, high-resolution ICP-MS and collision/reaction cell ICP-MS instrumentation is fit for purpose.

8.3 Mass-flow controller

A mass-flow controller on the nebulizer gas supply is strongly recommended. Mass-flow controllers for the plasma gas and the auxiliary gas are preferred. A cooled spraychamber (cold water or Peltier) may be of benefit in reducing some types of interferences (e.g. from polyatomic oxide species).

NOTE The plasma is very sensitive to variations in the gas flow rate. For older instruments a flowrestrictor in combination with elevated pressure may be used for nebulizer flow adjustment but makes an internal standard obligatory.

8.4 Nebulizer with variable speed peristaltic pump

Note: The speed of the pump shall not be too low and the number of rolls as high as possible to provide a stable signal.

8.5 Gas supply

Argon gas with high purity grade, i.e. > 99.99 %
Reaction gas: He, H₂ high purity (i.e. > 99.99 %)

8.6 Glassware

Volumetric flasks;

Erlenmeyer flasks;
Pipettes.

8.7 Storage bottles for the stock, standard, calibration and sample solutions

Preferably made from perfluoroalkoxy (PFA) or hexafluoroethene propene (FEP). For the determination of elements in a higher concentration range (>sub ppb) glass, HDPE or PTFE bottles may be sufficient for the storage of samples.

9 Procedure

9.1 Test sample

The test sample is an acidified aqueous sample ($\text{pH} \leq 1$) free of particles or the digest obtained by *<Horizontal standard nitric acid digestion>* or *<Horizontal standard aqua regia digestion>*.

9.2 Test portion

The test portion may be directly obtained from the digest or may be diluted to accommodate the measurement range or to dilute the matrix. The acidity of the test portion must match the acidity of calibration solutions.

Ensure that all elements are present in a non-volatile form. Volatile species must be converted to non-volatile ones e.g. sulfide oxidation by hydrogen peroxide.

9.3 Instrument set up

Adjust the instrumental parameters of the ICP-MS system in accordance with the manufacturer's manual. A guideline for method and instrument set up is given in ISO 17294-1.

Define the isotopes and the need for corresponding corrections. See clause 6.3.2 of ISO 17294-1 for a method to determine these factors. Alternatively, apply multivariate calibration procedures.

Define the rinsing times depending on the length of the flow path; in the case of wide working range of analyte mass concentrations in the measuring solutions, allow longer rinsing periods.

The use of an internal standard is recommended. Add the internal standard solution (7.9) to the interference check solution (7.13), to all multi-element calibration solutions (7.8), to the blank calibration solutions (7.10), and to all measuring solutions. On-line dilution and mixing of the sample flow with internal standard solution by means of the peristaltic pump of the nebuliser is commonly used. In such case the calibration solutions are diluted the same way as the sample solutions.

The mass concentration of the reference-elements shall be the same in all solutions. A mass concentration of 10 - 50 $\mu\text{g/l}$ (or optimized to $\pm 50.000 - 100.000$ counts/s) is often suitable.

About 30 min prior to measurement, adjust the instrument to working condition.

Before each series of measurement check the sensitivity and the stability of the system and minimize interference e.g. by using the optimization solution (7.12).

Check the resolution and the mass calibration as often as required by the manufacturer.

NOTE ICP-MS has excellent multi-element capability. Nevertheless it does not mean that all elements can be analyzed during one measurement run. The sensitivity of determination depends on numerous parameters (nebulizer flow, radio-frequency power, lens voltage, lens voltage mode etc.). The optimal instrument settings can not be reached

for all elements at once.

9.4 Calibration

9.4.1 Linear calibration function

If more than two concentration levels, including zero, are used apply weighted linear regression to obtain the linear calibration function.

NOTE ICP-MS provides a large measurement range. The dispersion of blank measurements is usually much smaller than the dispersion at full scale. Ordinary linear regression assumes that the dispersion is constant over the entire range. As a consequence a much higher percentage of the calculated intercepts is out of the range expected from the spread of blanks: a non-zero blank value is calculated that is actually not there. Weighted linear regression forces the line through points of low dispersion, resulting in the expected intercept dispersion. Unfortunately, many data systems cannot handle weighted regression. An alternative, but less efficient, approach is ordinary linear regression where the line is forced through the blank value or through zero. The most inexpensive way however is a (single or multiple) two point calibration. If calibration lines are linear, as they are usually in ICP-MS, this procedure is valid and efficient.

If weighted linear regression is not possible apply linear regression forced through the blank value or through zero. In the latter case check regularly by running a blank whether the assumption on the absence of a blank value is justified.

NOTE: weighted linear regression is not suitable for lower element concentrations.

A two point calibration is allowed if the calibration function is linear, which usually is the case. Check regularly with a calibration solution of known dilution whether the assumption on linearity is justified. Instead of one measurement per level more measurements can be performed to reduce the uncertainty of the calibration line.

9.4.2 Standard addition calibration

Add a known amount of standard solution (V_s) of the analyte and an equal amount of blank solution (V_b) to two separate but equal portions of the sample solution (or its dilution). Minimise dilution or correct for spike dilution. The added amount of standard solution should be between 0.4 and 2 times the expected sample mass concentration. Measure both solutions as a sample solution. Determine the 'measured spike concentration' as the difference in mass concentration between the two spiked sample portions. Use the ratio 'true spike concentration' versus 'measured spike concentration' as a correction factor for the initially measured concentration of the sample portion.

9.4.3 Determination of correction factors

The need for the use of correction factors is determined during method development. In order to evaluate and to update the correction factors, measure the interference check solutions (7.13) at regular intervals within a measuring cycle.

9.4.4 Variable isotope ratio

Take into account the possible discrepancies in the isotope composition between the calibration solutions and the measuring solutions (e.g. relevant for Li, Pb, U).

9.5 Sample measurement

Run one or more calibration solutions and a calibration blank.

Run the interference check solution(s) to establish interference correction or to check presence of interference.

Every 25 samples or less and at the beginning and end, run a calibration blank and a calibration check solution of an independent source (7.8).

Every (e.g.) 50 samples and at the end of a run, analyze an interference check solution (ICS).

Run all samples including one or more method blanks.

Run at least one post digestion spiked sample from the series to check recovery.

NOTE 1 If standard addition calibration is applied to all samples leave out recovery check.

Some elements (for example Ag, B, Be, Hg, Li, Th) are rinsed very slowly from the sample inlet system. Check whether a high sample count rate has an effect on the next measurement result.

Whenever a unknown sample matrix is encountered check:

- matrix effects by running the spike sample or
- matrix effects by running a fivefold diluted sample and
- inter-element interference analysing a different isotope.

10 Calculation

Calculate the element concentration in the digested solid sample:

$$w = (\rho_1 - \rho_0)f_a.V.100/(m.dw)$$

where:

ρ_1	is the concentration of the element in the test sample in $\mu\text{g/l}$;
ρ_0	is the concentration of the element in the blank in $\mu\text{g/l}$;
f_a	is the dilution factor of the test portion;
w	is the mass fraction of the element in the solid sample in $\mu\text{g/g}$ or mg/kg ;
V	is the volume of the test sample (digest) in litres;
m	is the mass of the digested sample in g.
dw	is the dry weight percentage of the sample.

11 Expression of results

State as many significant figures as are acceptable according to the precision of the measuring values, but not more than three significant figures.

12 Performance characteristics

12.1 Blank

Result of the calibration blank check shall be less than 3 times the instrumental limit of detection or 0.5 times the lowest concentration to be reported.

12.2 Continuing calibration verification

Result of the calibration check standard shall not deviate more than 10% otherwise recalibrate.

12.3 Internal standard response

The relative response of the internal standard in a sample shall be between 70 and 130% of the response of the internal standard blank or calibration check solution. If not, clean the cones and recalibrate.

12.4 Interference

The contribution of corrected and uncorrected isobaric molecular and doubly charged shall not be higher than three times the instrumental detection limit in trace analysis or lower than half the value of the lowest concentration to be reported.

In high matrix measurements the limit of detection of the analytes of interest should be determined in ICS containing relevant interferences.

Note: Assume that the concentration for calcium in an interference check solution (ICS) based upon a relevant matrix is 200 mg/L. As long as the contribution of interference of Ca on the interfered isotope (Ni60) is lower than three times the instrumental detection limit or lower than 0.5 times the lowest concentration to be reported (and if the concentration of the interferent (Ca) in the testsample is LOWER than 200 mg/L) the concentration of the interfered isotope (Ni60) can be reported.

Assume that the contribution of interference by the same 200 mg/L calcium solution is e.g. 0.5 µg/L and the maximum allowed contribution is 1 µg/L (either 3* IDL or 0.5 * lowest concentration to be reported) than the maximum concentration of the interferent (Ca) in the testsample on the interfered isotope (Ni60) is 400 mg/L (= 1/0.5 * 200 mg/L assuming a linear relation). Otherwise Ni60 can NOT be reported prior to further investigation of the interference.

12.5 Recovery

Spike recovery shall be between 75% and 125% or difference between results for the original sample and the fivefold-diluted sample shall be less than 20% when the concentration in fivefold-diluted sample is higher than three times the instrumental detection limit or twice the lowest concentration to be reported. Spike concentration should be within 0.4 and 2.5 times the analyte concentration.

12.6 Precision

For precision data see Annex B

13 Test report

The test report shall make reference to this international standard and contain the following details:

- a) Complete identification of the sample;
- b) Expression of results as indicated in clause 9;
- c) Sample pre-treatment, if appropriate;
- d) Any deviations from this method, and details of all circumstances which could have affected the result.

Annex A Informative

Table 1. Selected isotopes, spectral interferences and detection limits for quadrupole ICP-MS instruments

Element	Isotope	Abundance (%)	Potential interferences		Interference with practical relevance	Best usable isotopes	
			Inter-element	Polyatomic ions		Preference	Reason
Ag	107	51,8		ZrO YO	⁹¹ Zr ¹⁶ O	x	least interference
	109	48,2		ZrO ZrOH NbO			
Al	27	100		BeO BO CN HCN		x	
As	75	100	Sm ²⁺ Eu ²⁺ Nd ²⁺	ArCl CoO Ar ₂ H ArK CaO ₂ NaCAr CPO ₂ CaCl	⁴⁰ Ar ³⁵ Cl	x	
Au	197	100		HfO TaO		x	
B	10	19,9				x	free of interference, low background
	11	80,1		BH			
Ba	135	6,6					
	137	11,2				x	highest abundance, least interference
	138	71,7	La Ce		La, Ce		
Be	9	100				x	
Bi	209	100				x	
Ca	43	0,14	Sr ²⁺	MgO BO ₂ AlO CaH CNO CO ₂	Sr ²⁺	x	low background, least interference
	44	2,1	Sr ²⁺	CaH MgO AlO BO ₂ CNO SiO CO ₂ N ₂ O	Sr ²⁺ , ¹² C ¹⁶ O ₂	x	lowest det. limit
Cd	111	12,8		MoO MoOH ZrOH K ₂ O ₂ H	⁹⁴ Zr ¹⁶ O ¹ H, ⁹⁵ Mo ¹⁶ O		
	113	12,2	In	MoO ZrOH Ca ₂ O ₂ H Ar ₂ O ₂ H RuO	In, ⁹⁷ Mo ¹⁶ O	x	for high Sn
	114	28,7	Sn	MoO MoOH RuO	Sn, ⁹⁸ Mo ¹⁶ O	x	lowest det. limit, least interference
Ce	140	88,5				x	
Co	59	100	Sn ²⁺	CaO CaOH MgCl ArNa ArOH ArF	⁴³ Ca ¹⁶ O	x	
Cr	52	83,8		SO ArO ArC ArN ClO HClO ClN ArNH	⁴⁰ Ar ¹² C	x	for low C and high Cl
	53	9,5		H ₂ O ArC HClO ClO ArOH ArN ArNH SO	³⁷ Cl ¹⁶ O	x	for high C and low Cl
Cs	133	100		RuO ₂		x	
Cu	63	69,2		TiO PO ₂ ArNa MgCl NaCa CaOH ArCNH NCCl COCl	⁴⁷ Ti ¹⁶ O, ⁴⁰ Ar ²³ Na	x	for low Na and Ti, lowest det. limit
	65	30,8	Ba ²⁺	TiO PO ₂ SO ₂ SO ₂ H ArMg CaOH ArN ₂ H S ₂ COCl	⁴⁹ Ti ¹⁶ O, ³² S ¹⁶ O ₂ ¹ H, ⁴⁰ Ar ²⁵ Mg	x	for medium Mg, S and Ti and high Na
Dy	163	24,9		NdO SmO		x	
Er	166	33,6		SmO NdO		x	
Eu	151	47,8		BaO			
	153	52,2		BaO		x	
Fe	54	5,8	Cr	HClO ArO ArN ArNH ArOH SO ClO	ArN Cr	x	abundance and background deter- mine choice
	57	2,2		CaO ArO ArOH CaC CaN CaOH MgO ₂ ArF	⁴⁰ Ar ¹⁶ O ¹ H ⁴⁰ Ca ¹⁶ O ¹ H	x	
Ga	69	60,1	Ce ²⁺ Ba ²⁺ La ²⁺	ClO ₂ ArP VO ArS SO ₂ S ₂	Ba ²⁺		
	71	39,9	Nd ²⁺ Ce ²⁺	ArP ClO ₂ ArCl SO ₂ ArS CrO		x	least interference
Gd	157	15,7		CeO PrO LaO BF		x	least interference
	158	24,8	Dy	CeO PrO NdO	Dy		

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Element	Isotope	Abundance (%)	Potential interferences		Interference with practical relevance	Best usable isotopes	
			Interelement	Polyatomic ions		Preference	Reason
Ge	74	35,9	Se Sm ²⁺ Nd ²⁺	ArS Cl ₂ Ar ₂	Se	x	
Hf	178	27,3		GdO DyO ErO		x	
Hg	200	23,1		WO	¹⁸⁴ W ¹⁶ O		
	201	13,2		WO	¹⁸⁴ W ¹⁷ O	x	least interference of WO
	202	29,9		WO	¹⁸⁶ W ¹⁶ O		
Ho	165	100		SmO		x	
In	115	95,7	Sn	RuO	Sn	x	
Ir	193	62,7		HfO LuO		x	
K	39	93,3		ArH		x	
La	139	99,9				x	
Li	6	7,5					
	7	92,5				x	lowest det. limit
Lu	175	97,4	Hf	GdO TbO	Hf	x	
Mg	24	79,0	Ti ²⁺ Ca ²⁺	LiO NaH C ₂		x	lowest det. limit, least interference
	25	10,0	Ti ²⁺	LiO BeO C ₂ C ₂ H			
	26	11,0		BeO BO CN C ₂ H ₂ C ₂ H		x	for higher Mg concentrations
Mn	55	100		HClO ArN ClO NaS ArOH ArNH KO ArN ArO ArF	ArNH	x	
Mo	95	15,9		ArKO BrO			
	98	24,1	Ru	BrO K ₂ O	Ru	x	lowest det. limit
Na	23	100	Ti ²⁺ Ca ²⁺	LiO		x	
Nd	146	17,2		BaO RuO ₃		x	
Ni	58	68,1	Fe	CaO ArO CaN NaCl MgS CaOH Si ₂ ArOH	Fe	x	for low Fe
	60	26,2	Sn ²⁺	CaO CaOH MgCl NaCl	⁴⁴ Ca ¹⁶ O	x	least interference
	61	1,1	Sn ²⁺	CaOH ScO	⁴⁴ Ca ¹⁶ O ¹ H, ⁴⁵ Sc ¹⁶ O		
P	31	100		SiH NO NOH N ₂ H CO COH	NO	x	
Pb	206 ^B)	24,1		PtO		x	Sum of 206, 207 and 208
	207 ^B)	22,1		IrO		x	Sum of 206, 207 and 208
	208 ^B)	52,4		PtO		x	Sum of 206, 207 and 208
Pd	105	22,3		YO ArCu			
	108	26,5	Cd	MoO ZrO	Cd	x	
Pr	141	100				x	
Pt	195	33,8		HfO		x	
Rb	85	72,2	Yb ²⁺ Er ²⁺			x	
Re	185	37,4		TmO ErO		x	least interference
	187	62,6	Os	TmO YbO	Os	x	highest abundance
Rh	103	100	Pb ²⁺	SrO ArCu RbO		x	
Ru	101	17,0		ArNi NiCl			
	102	31,6	Pd	SrO	Pd	x	lowest det. limit, least interference

Element	Isotope	Abundance (%)	Potential interferences		Interference with practical relevance	Best usable isotopes	
			Interelement	Polyatomic ions		Preference	Reason
S	34	4,2		O ₂ SH NOH O ₂ H	O ₂	x	
Sb	121	57,4		PdO		x	least interference
	123	47,6	Te	ZrO	Te		
Sc	45	100	Zr ²⁺	CO ₂ SiO BO ₂ AlO CaH CHO ₂ SiOH N ₂ OH	¹³ C ¹⁶ O ₂ , ²⁹ Si ¹⁶ O	x	
Se	77	7,6	Sm ²⁺ Gd ²⁺	ArCl Ar ₂ H CaCl CFNO ₂	⁴⁰ Ar ³⁷ Cl	x	for low Cl
	78	23,8	Kr Gd ²⁺ Dy ²⁺	Ar ₂ CaCl ArCa	³⁸ Ar ⁴⁰ Ar	x	for high Cl and Br
	82	8,7	Kr Ho ²⁺ Er ²⁺ Dy ²⁺	Ar ₂ H BrH CCl ₂ SO ₃ Ar ₂ H ₂ ArCa	Kr, ⁸¹ Br ¹ H	x	for low Br
Si	29	4,7		CO N ₂ BO SiH AlH COH N ₂ H		x	
Sm	147	15,0		RuO ₃		x	
Sn	118	24,2	U ²⁺	MoO RuO PdO		x	least interference
	120	32,6	Te	RuO PdO	Te		
Sr	86	9,9	Kr Yb ²⁺	RbH			
	88	82,6	Yb ²⁺ Lu ²⁺			x	lowest det. limit, least interference
Tb	159	100		NdO PrO		x	
Te	125	7,1				x	least interference
Te	126	19,0	Xe	PdO	Xe	x	lowest det. limit
Th	232	100				x	
Ti	47	7,3	Zr ²⁺	NO ₂ PO SiO CCl SNH SiOH SN N ₂ NO ₂ H	³¹ P ¹⁶ O	x	least interference
	48	73,8	Ca Zr ²⁺	ArC CCl SO NO ₂ PO SN NN ₂ C4	Ca, ³² S ¹⁶ O		
	49	5,5		SOH	³² S ¹⁶ O1H		
Tl	203	29,5		WO ReO WHO			
	205	70,5				x	lowest det. limit, least interference
Tm	169	100		EuO		x	
U	238	99,3				x	
V	51	99,8		HSO ClO CIN ArNH ArC ArN SN SO	³⁵ Cl ¹⁶ O	x	
W	182	26,3		HoO DyO ErO		x	least interference
	184	30,7	Os	ErO YbO	Os		
Y	89	100				x	
Yb	172	21,9		DyO SmO GdO		x	least interference
	174	31,8	Hf	DyO GdO	Hf		
Zn	64	48,6	Ni	TiO CaO PO ₂ SO ₂ AlCl S ₂ PO ₂ H ArN ₂ ArMg	Ni, ⁴⁸ Ti ¹⁶ O, ³² S ¹⁶ O ₂ , ⁴⁰ Ar ²⁴ Mg		
	66	27,9	Ba ²⁺	TiO VO SO ₂ PCI FeC S ₂ SO ₂ H	⁵⁰ Ti ¹⁶ O, ³⁴ S ¹⁶ O ₂	x	for medium Ti ans S
	68	18,8	Ba ²⁺ Ce ²⁺	VO ClO ₂ SO ₂ TiO ArS FeN PCI FeC S ₂ ArN ₂ ArSi	Ba ²⁺ , ⁴⁰ Ar ²⁸ Si	x	for low Ba and Si
Zr	90	51,5				x	

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Table 2. Examples of equations for isobaric overlay.

Element	Recommended isotope and inter-element correction
As	$^{75}\text{As} - 3,127(^{77}\text{Se} - 0,815^{82}\text{Se})$ or $^{75}\text{As} - 3,127(^{77}\text{Se} + 0,3220^{78}\text{Se})$
Ba	$^{138}\text{Ba} - 0,0009008^{139}\text{La} - 0,002825^{140}\text{Ce}$
Cd	$^{114}\text{Cd} - 0,02684^{118}\text{Sn}$
Ge	$^{74}\text{Ge} - 0,1385^{82}\text{Se}$
In	$^{115}\text{In} - 0,01486^{118}\text{Sn}$
Mo	$^{98}\text{Mo} - 0,1106^{101}\text{Ru}$
Ni	$^{58}\text{Ni} - 0,04825^{54}\text{Fe}$
Pb	$^{208}\text{Pb} + ^{207}\text{Pb} + ^{206}\text{Pb}$
Se	$^{82}\text{Se} - 1,009^{83}\text{Kr}$
Sn	$^{120}\text{Sn} - 0,01344^{125}\text{Te}$
V	$^{51}\text{V} - 3,127(^{53}\text{Cr} - 0,1134^{52}\text{Cr})$
W	$^{184}\text{W} - 0,001242^{189}\text{Os}$

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Validation

Note: The data in table 1 are recorded with quadrupole mass spectrometer equipped with a reaction cell and the use of Helium [He] and Hydrogen [H₂] gas in reaction mode.

Table 1: Method Limit of detection (informative): (taken from ref 1)

Method detection limit (in mg/kg dw)*

Element	Blank	ICS1	ICS2	ICS1+2
	3 gram 100 mL	3 gram 100 mL	3 gram 100 mL	3 gram 100 mL
Be / 9 [He]	0.06	0.05	0.05	0.06
V / 51 [He]	0.15	0.26	0.19	0.21
Cr / 52 [He]	0.16	0.13	0.13	0.13
Cr / 53 [He]	0.60	0.65	0.66	0.57
Ni / 58 [He]	0.12	4.82	0.09	3.01
Co / 59 [He]	0.06	0.05	0.04	0.03
Ni / 60 [He]	0.09	0.10	0.06	0.06
Ni / 61 [He]	0.31	0.53	1.15	0.77
Cu / 63 [He]	0.91	1.05	1.01	1.06
Zn / 64 [He]	0.21	0.21	0.14	0.16
Cu / 65 [He]	0.88	0.99	0.98	1.02
Zn / 66 [He]	0.21	0.18	0.18	0.19
Zn / 68 [He]	0.31	0.28	0.19	0.19
As / 75 [He]	0.12	0.15	0.09	0.07
Se / 77 [He]	1.67	1.24	0.83	1.69
Se / 78 [He]	1.27	1.05	1.41	1.44
Se / 82 [He]	0.71	0.67	0.55	0.63
Mo / 95 [He]	0.19	0.54	0.51	0.60
Mo / 98 [He]	0.19	0.56	0.53	0.62
Cd / 111 [He]	0.06	0.05	0.05	0.04
Cd / 114 [He]	0.06	0.03	0.04	0.03
Sn / 118 [He]	0.13	0.10	0.07	0.09
Sn / 120 [He]	0.13	0.10	0.08	0.10
Sb / 121 [He]	0.07	0.09	0.07	0.09
Sb / 123 [He]	0.07	0.09	0.10	0.09
Te / 125 [He]	0.08	0.06	0.10	0.10
Te / 126 [He]	0.05	0.05	0.04	0.04
Ba / 135 [He]	0.12	0.09	0.10	0.06
Ba / 137 [He]	0.10	0.07	0.06	0.05
Hg / 200 [He]	0.01	0.01	0.01	0.01
Hg / 201 [He]	0.01	0.01	0.01	0.01
Hg / 202 [He]	0.01	0.01	0.01	0.01
Tl / 203 [He]	0.14	0.16	0.11	0.10
Tl / 205 [He]	0.16	0.16	0.11	0.09
Pb / 206 [He]	0.07	0.04	0.05	0.05
Pb / 207 [He]	0.08	0.04	0.06	0.06
Pb / 208 [He]	0.07	0.04	0.05	0.04
Se / 77 [H ₂]	0.34	0.56	0.60	0.70
Se / 78 [H ₂]	0.05	0.08	0.07	0.10

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Be / 9 [St]	0.04	0.03	0.02	0.04
V / 51 [St]	0.77	1.68	0.96	1.23
Cr / 52 [St]	0.20	0.13	0.19	0.11
Cr / 53 [St]	2.63	5.55	3.34	4.08
Ni / 58 [St]	0.13	11.48	0.08	5.75
Co / 59 [St]	0.05	0.04	0.03	0.04
Ni / 60 [St]	0.11	0.09	0.13	0.10
Ni / 61 [St]	0.37	2.09	8.83	6.36
Cu / 63 [St]	0.91	1.05	1.18	1.17
Zn / 64 [St]	0.17	0.22	0.37	0.25
Cu / 65 [St]	0.85	1.00	0.97	1.02
Zn / 66 [St]	0.20	0.19	0.20	0.14
Zn / 68 [St]	0.20	0.18	0.14	0.12
As / 75 [St]	0.57	1.81	1.32	1.32
Se / 77 [St]	5.07	11.93	9.32	8.46
Se / 78 [St]	2.05	4.46	5.34	5.19
Se / 82 [St]	0.34	0.21	0.37	0.26
Mo / 95 [St]	0.15	0.28	0.25	0.29
Mo / 98 [St]	0.16	0.27	0.26	0.32
Cd / 111 [St]	0.06	0.03	0.03	0.04
Cd / 114 [St]	0.04	0.02	0.02	0.02
Sn / 118 [St]	0.11	0.11	0.10	0.13
Sn / 120 [St]	0.11	0.10	0.08	0.11
Sb / 121 [St]	0.05	0.09	0.06	0.09
Sb / 123 [St]	0.05	0.10	0.07	0.08
Te / 125 [St]	0.05	0.03	0.03	0.02
Te / 126 [St]	0.03	0.01	0.02	0.02
Ba / 135 [St]	0.10	0.05	0.06	0.04
Ba / 137 [St]	0.09	0.07	0.08	0.06
Hg / 200 [St]	0.02	0.01	0.02	0.01
Hg / 201 [St]	0.02	0.02	0.02	0.02
Hg / 202 [St]	0.02	0.01	0.01	0.01
Tl / 203 [St]	0.14	0.16	0.13	0.11
Tl / 205 [St]	0.14	0.14	0.12	0.11
Pb / 206 [St]	0.08	0.06	0.06	0.03
Pb / 207 [St]	0.07	0.04	0.04	0.03
Pb / 208 [St]	0.06	0.03	0.04	0.03

*: Horizontal phase II: ICP-MS Ruggedness test table 8

Legenda:

Component	ICS 1	ICS 2
Aluminium	1000 mg/L	-
Iron	1000 mg/L	-
Potassium	2000 mg/L	-
Sodium	1000 mg/L	-
Phosphorus	-	1000 mg/L
Calcium	1000 mg/L	4000 mg/L
Magnesium	1000 mg/L	-
Sulphur	-	1000 mg/L
Carbon	-	2000 mg/L
Chlorine	2000 mg/L	2000 mg/L

Cl: can interfere on V, Cr 53, As, Se77

Precision

Table 2: Measurement of precision (in VC_w %):

Element	Rep. limit Limit	CRM145R	FeNeLab	LKSD-1	STSD	CRM7001	CRM144R	CRM141R	AMBWB	AMBSZ	AMB COMP	Average
Be / 9 [He]	0.1	30.1	4.9	25.1	2.1	4.9	54.3	3.5	7.7	<r.l.	<r.l.	16.6
V / 51 [He]	1	6.2	7.9	5.3	7.4	4.7	6.4	4.1	5.1	10.7	10.9	6.9
Cr / 52 [He]	15	5.6	5.2	5.5	4.3	6.4	5.0	4.7	5.6	<r.l.	6.9	5.5
Cr / 53 [He]	15	5.9	5.0	9.0	4.3	5.7	5.3	4.6	5.8	<r.l.	8.4	6.0
Ni / 58 [He]	3	5.5	4.7	4.4	2.9	4.2	4.2	4.4	4.8	<r.l.	7.5	4.7
Co / 59 [He]	1	5.0	5.0	4.5	3.8	5.0	4.7	4.6	5.3	<r.l.	8.2	5.1
Ni / 60 [He]	3	5.4	5.3	5.0	3.9	5.4	4.6	4.5	5.4	<r.l.	7.2	5.2
Ni / 61 [He]	3	4.8	5.6	7.7	4.1	5.4	6.8	5.5	5.9	<r.l.	17.0	7.0
Cu / 63 [He]	5	4.4	4.5	4.6	3.7	5.1	3.9	4.3	5.2	<r.l.	6.4	4.7
Zn / 64 [He]	17	3.7	3.7	3.4	2.7	4.3	3.1	2.7	4.1	<r.l.	5.0	3.6
Cu / 65 [He]	5	4.5	4.8	4.7	4.1	5.5	4.0	4.6	5.5	<r.l.	7.0	5.0
Zn / 66 [He]	1	3.6	3.9	3.6	3.1	3.9	3.1	3.7	4.2	4.0	5.0	3.8
Zn / 68 [He]	17	3.5	3.9	3.4	3.0	4.0	3.3	3.4	4.2	<r.l.	4.4	3.7
As / 75 [He]	4	5.1	3.2	3.0	2.5	3.2	<r.l.	2.8	3.8	<r.l.	<r.l.	3.4
Se / 77 [He]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Se / 78 [He]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Se / 82 [He]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Mo / 95 [He]	1.5	23.7	<r.l.	7.0	4.1	<r.l.	18.3	<r.l.	<r.l.	<r.l.	<r.l.	13.2
Mo / 98 [He]	1.5	22.8	<r.l.	7.5	4.6	<r.l.	18.6	<r.l.	<r.l.	<r.l.	<r.l.	13.4
Cd / 111 [He]	0.17	2.6	3.6	4.6	3.9	7.4	5.2	2.7	<r.l.	<r.l.	8.3	4.8
Cd / 114 [He]	0.17	5.0	4.0	3.8	5.0	8.1	4.9	3.6	<r.l.	<r.l.	8.2	5.3
Sn / 118 [He]	6	4.1	5.4	<r.l.	<r.l.	11<r.l.	5.2	<r.l.	<r.l.	<r.l.	<r.l.	4.9
Sn / 120 [He]	6	4.1	5.3	<r.l.	<r.l.	<r.l.	5.3	<r.l.	<r.l.	<r.l.	<r.l.	4.9
Sb / 121 [He]	1	4.6	9.4	<r.l.	12.7	<r.l.	8.0	<r.l.	<r.l.	<r.l.	<r.l.	8.7
Sb / 123 [He]	1	4.7	8.5	<r.l.	13.4	<r.l.	7.3	<r.l.	<r.l.	<r.l.	<r.l.	8.5
Te / 125 [He]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Te / 126 [He]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Ba / 135 [He]	15	11.4	4.1	2.9	3.2	4.3	3.7	3.3	13.8	<r.l.	3.9	5.6
Ba / 137 [He]	15	13.0	4.8	3.8	4.3	5.8	4.5	4.2	15.9	<r.l.	4.9	6.8
Hg / 200 [He]	0.05	1.4	2.0	8.1	8.2	5.4	3.0	3.2	3.2	<r.l.	<r.l.	4.3
Hg / 201 [He]	0.05	3.0	1.9	10.1	<r.l.	9.8	2.9	4.7	3.4	<r.l.	<r.l.	5.1
Hg / 202 [He]	0.05	2.7	1.8	5.2	7.4	3.7	1.8	2.8	2.9	<r.l.	<r.l.	3.5
Tl / 203 [He]	3	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Tl / 205 [He]	3	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Pb / 206 [He]	13	2.5	2.3	1.5	2.7	2.3	2.8	1.8	<r.l.	<r.l.	6.7	2.8
Pb / 207 [He]	13	2.4	2.4	1.7	2.4	2.7	2.7	1.7	<r.l.	<r.l.	6.3	2.8
Pb / 208 [He]	13	3.6	2.2	1.5	2.6	2.5	2.6	1.7	<r.l.	<r.l.	6.1	2.9
Se / 77 [H2]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Se / 78 [H2]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Be / 9 [St]	0.1	21.5	2.9	15.6	2.2	3.9	25.8	3.3	4.1	<r.l.	<r.l.	9.9
V / 51 [St]	1	15.5	7.5	12.2	6.3	6.3	24.5	6.5	6.9	76.7	61.8	22.4
Cr / 52 [St]	15	5.0	6.6	6.2	5.0	4.5	7.6	5.4	5.4	<r.l.	6.7	5.8
Cr / 53 [St]	15	8.0	8.0	<r.l.	9.6	7.8	13.6	6.7	9.5	<r.l.	24.1	10.9
Ni / 58 [St]	3	5.2	6.2	5.1	5.1	5.4	4.9	4.9	4.7	<r.l.	7.3	5.4
Co / 59 [St]	1	5.6	6.3	5.5	4.7	5.1	4.7	4.6	5.0	<r.l.	6.0	5.3
Ni / 60 [St]	3	5.4	6.5	5.8	4.7	4.8	4.8	4.8	5.0	<r.l.	8.0	5.5
Ni / 61 [St]	3	5.9	7.0	12.0	4.9	4.8	10.8	6.2	5.7	<r.l.	18.6	8.4
Cu / 63 [St]	5	4.5	5.4	5.0	4.5	5.1	4.3	4.7	5.0	<r.l.	6.3	5.0
Zn / 64 [St]	17	2.1	3.5	2.7	2.6	5.7	2.7	2.7	3.2	<r.l.	3.5	3.2
Cu / 65 [St]	5	4.3	5.2	4.8	4.6	4.9	4.6	4.7	4.6	<r.l.	6.0	4.9
Zn / 66 [St]	1	2.1	3.2	3.4	2.4	2.8	2.9	2.4	2.5	3.3	3.3	2.8
Zn / 68 [St]	17	2.1	3.1	2.3	2.7	3.1	2.7	2.4	2.9	<r.l.	3.0	2.7
As / 75 [St]	4	58.6	5.1	6.1	5.0	9.9	<r.l.	16.0	7.8	<r.l.	<r.l.	15.5
Se / 77 [St]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Se / 78 [St]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Se / 82 [St]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.

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Mo / 95 [St]	1.5	14.3	<r.l.	4.6	3.3	<r.l.	10.9	12.7	14.2	<r.l.	<r.l.	10.0
Mo / 98 [St]	1.5	13.5	<r.l.	4.5	3.4	<r.l.	10.3	12.4	13.7	<r.l.	<r.l.	9.6
Cd / 111 [St]	0.17	4.5	1.7	3.5	3.0	6.3	4.1	1.6	<r.l.	<r.l.	3.5	3.5
Cd / 114 [St]	0.17	2.0	1.5	1.9	2.4	4.2	4.3	1.7	<r.l.	<r.l.	3.2	2.7
Sn / 118 [St]	6	2.5	1.5	<r.l.	<r.l.	<r.l.	3.2	<r.l.	<r.l.	<r.l.	<r.l.	2.4
Sn / 120 [St]	6	2.1	1.3	<r.l.	<r.l.	<r.l.	2.5	<r.l.	<r.l.	<r.l.	<r.l.	2.0
Sb / 121 [St]	1	3.0	5.4	<r.l.	11.4	<r.l.	4.8	<r.l.	<r.l.	<r.l.	<r.l.	6.1
Sb / 123 [St]	1	3.2	5.8	<r.l.	11.0	<r.l.	5.1	<r.l.	<r.l.	<r.l.	<r.l.	6.3
Te / 125 [St]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Te / 126 [St]	10	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Ba / 135 [St]	15	9.5	1.8	1.5	2.4	2.8	2.5	2.2	13.1	<r.l.	2.2	4.2
Ba / 137 [St]	15	8.8	1.5	1.7	1.8	2.9	2.3	2.5	12.9	<r.l.	1.8	4.0
Hg / 200 [St]	0.05	2.5	3.7	14.2	20.1	7.2	4.8	4.2	4.7	<r.l.	<r.l.	7.7
Hg / 201 [St]	0.05	2.5	2.9	8.7	<r.l.	10.0	2.2	6.3	4.0	<r.l.	<r.l.	5.2
Hg / 202 [St]	0.05	4.1	4.5	7.8	11.3	7.5	4.2	7.9	4.6	<r.l.	<r.l.	6.5
Tl / 203 [St]	3	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Tl / 205 [St]	3	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.	<r.l.
Pb / 206 [St]	13	2.2	3.7	1.9	3.5	2.1	3.0	2.5	<r.l.	<r.l.	2.4	2.7
Pb / 207 [St]	13	2.0	3.4	1.8	3.2	2.0	2.8	2.3	<r.l.	<r.l.	2.2	2.5
Pb / 208 [St]	13	2.0	3.6	2.5	3.7	2.1	2.9	1.5	<r.l.	<r.l.	2.2	2.6
Average:												6.1

Legenda

1. CRM 145R: sewage sludge.
2. FeNeLab: internal reference material, river clay.
3. Lksd-1: lake sediment.
4. Stsd-2: stream sediment.
5. CRM 7001: light sandy soil.
6. CRM 144R: sewage sludge from domestic origin.
7. CRM 141R: Calcareous loam soil.
8. AMB WB: internal reference material: lake sediment.
9. AMB SZ: internal reference material: 'playground sand'.
10. AMB Compost: internal reference material; biowaste/garden waste.
11. Average: averaged precision of the 10 testsamples.

**Annex B
Informative**

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