BS EN 16171:2016

BSI Standards Publication

Sludge, treated biowaste and soil — Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)

National foreword

This British Standard is the UK implementation of EN 16171:2016.

BSI, as a member of CEN, is obliged to publish EN 16171 as a British Standard. However, attention is drawn to the fact that during the development of this European Standard, the UK committee voted against its approval as a European Standard.

The UK committee is concerned that some of the conditions attached to the validation trials lack clarity.

It is also the opinion of the UK committee that validation trials for horizontal methods should include conditions for the preparation and extraction of the original sample.

The UK participation in its preparation was entrusted to Technical Committee H/-/4, Environmental Testing Programmes.

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.

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

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English Version

Sludge, treated biowaste and soil - Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)

Boues, bio-déchets traités et sols - Détermination des éléments en traces par spectrométrie de masse avec plasma induit par haute fréquence (ICP-MS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Elementen mittels Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS)

This European Standard was approved by CEN on 19 March 2016.

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European foreword

This document (EN 16171:2016) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

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 supersedes CEN/TS 16171:2012.

The preparation of the previous edition of this analytical method by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

This document contains the following technical changes in comparison with the previous edition:

— 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).

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see Annex A for the results of validation).

Table 1 — Matrices for which this European Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost
Soil	Soil

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

1 Scope

This European Standard specifies a method for the determination of the following elements in *aqua regia* or nitric acid digests 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 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 17294-1:2006, 8.3).

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.

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.

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16173, *Sludge, treated biowaste and soil — Digestion of nitric acid soluble fractions of elements*

EN 16174, *Sludge, treated biowaste and soil — Digestion of aqua regia soluble fractions of elements*

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

EN ISO 17294-1:2006, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines (ISO 17294-1:2004)*

3 Principle

Digests of sludge, treated biowaste or soil with nitric acid or *aqua regia* (see EN 16173 and EN 16174) are analysed by ICP-MS to get a multi-elemental determination of analytes.

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).

NOTE For the determination of tin only *aqua regia* extraction applies (EN 16174).

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 17294-1:2006, Clause 6.

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 may be corrected by taking into account the influence from the interfering element (see EN ISO 17294-1:2006). 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. 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.

More information about the use of correction factors is given in EN 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 significant signal suppression or enhancement. Solids can be deposited on the nebuliser tip of a pneumatic nebuliser and on the cones.

It is recommended to keep the level of total dissolved solids below 0,2 % (2 000 mg/l) to minimise 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 minimise 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) usually eliminates the problem.

5 Reagents

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.

5.1 Water, grade 1 as specified in EN ISO 3696 for all sample preparations and dilutions.

5.2 Nitric acid, HNO_3, $\rho(HNO_3)$ = 1,4g/ml, $c(HNO_3)$ = 15 mol/l, $w(HNO_3)$ = 650 g/kg.

5.3 Hydrochloric acid, HCl, $\rho(HCl)$ = 1,18 g/ml, $c(HCl)$ = 12 mol/l, $w(HCl)$ = 370 g/kg.

5.4 Single-element standard 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, ρ(element) = 1 000 mg/l each.

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

Both single-element standard stock solutions and multi-element standard 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.

5.5 Anion standard stock solutions

Cl^-, PO_4^{3-}, SO_4^{2-}, ρ(anion) = 1 000 mg/l each.

Prepare these solutions from the respective acids. The solutions 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.

5.6 Multi-element standard stock solutions

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

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

Mercury standard stock solutions can be stabilised by adding 1 mg/l Au in nitric acid (5.2) or by adding hydrochloric acid (5.3) up to 0,6 %.

NOTE When Au is to be used as a modifier, it cannot be determined accurately itself in the same analysis run.

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

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

Ag, Al, As, B, Ba, Be, Bi, Cd, Ce, Co, Cr, Cu, Fe, Hg, Li, Mn, Nd, Ni, Pb, Pr, Sc, Se, Si, Sm, Sr, Te, Th, Ti, Tl, U, V, Zn.

Use nitric acid (5.2) for stabilisation of multi-element standard stock solution A.

Other elements of interest may be added to the standard stock solution, provided that the resulting multi-element solution is stable.

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

Mo, Sb, Si, Sn, W, Zr.

Use hydrochloric acid (5.3) for stabilisation of multi-element standard stock solution B.

Other elements of interest may be added to the standard stock solution, provided that the resulting multi-element solution is stable.

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

Ca, Mg, Na, K, P, S.

Use nitric acid (5.2) for stabilisation of multi-element standard stock solution C.

5.7 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 (5.2 and/or 5.3) 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 calibration solutions.

5.8 Internal standard solution

Internal standards can either be added to every flask or added online. It is essential that the same concentration of internal standard is added to all measurement solutions. The elements In, Lu, Re, Ge and Rh have been found suitable for this purpose.

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 internal standards elements shall not be analytes and the concentrations of the selected elements should be negligibly low in the digests of samples.

Generally, a suitable final concentration of the internal standard in samples and calibration solutions is 1 µg/l to 50 µg/l (for a high and stable count rate). The use of a collision/reaction cell may require higher concentrations.

5.9 Calibration blank solution

Prepare the calibration blank solution by diluting acids (5.2, 5.3) with water (5.1) to the same concentrations as used in the calibration solutions and test solutions.

5.10 Test blank solution

The test blank solution shall contain all of the reagents in the same concentrations and shall be handled in the same way throughout the procedure as the samples. The test blank solution contains the same acid concentration in the final solution as the test solution after the digestion method is applied.

5.11 Optimisation solution

The optimisation solution is used for mass calibration and for optimisation 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 optimisation solution depends on the elements of interest, instrument and manufacturer's instructions. An optimisation 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 optimisation should allow count rates of more than 10^4 counts per second.

5.12 Interference check solution

The interference check solutions are used to determine the corresponding factors for the correction 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 interferences of practical relevance given in EN ISO 17294-1, at a concentration level at the same range as expected in the samples (see also 10.4).

Leaving out an interfering element according to EN ISO 17294-1 is permitted if it can be demonstrated that its impact is negligible and lasting.

In unusual situations, the other interfering elements according to EN ISO 17294-1 shall also be investigated for relevance.

EXAMPLE An example of the composition of an interference check solution is:

$\rho(\text{Ca})$ = 2 500 mg/l; $\rho(\text{Cl}^-)$ = 2 000 mg/l; $\rho(\text{PO}_4^{3-})$ = 500 mg/l and $\rho(\text{SO}_4^{2-})$ = 500 mg/l

and for digests also

$\rho(\text{C})$ = 1 000 mg/l; $\rho(\text{Fe})$ = 500 mg/l; $\rho(\text{Na})$ = 500 mg/l and $\rho(\text{Al})$ = 500 mg/l.

6 Apparatus

6.1 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 (< 1 µg/kg), glass or polyvinyl chloride (PVC) should not be used. Instead, it is recommended that perfluoroalkoxy alkane (PFA), hexafluoroethene propene (FEP) or quartz containers, cleaned with diluted, high quality nitric acid or hot, concentrated nitric acid in a closed system be used. For the determination of elements in a higher concentration range, containers made from high density polyethylene (HDPE) or polytetrafluoroethene (PTFE) are also suited for the collection of samples. Immediately before use, all containers should be washed thoroughly with diluted nitric acid (e.g. $w(HNO_3) = 10\ \%$), and then rinsed several times with water (5.1).

The limit of detection of most elements is affected by contamination of solutions and this depends predominantly on the cleanliness of laboratory air.

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 plastics vessels shall be tested for impurities.

For more detailed information on the instrumentation see EN ISO 17294-1:2006, Clause 5.

6.2 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, time-of-flight ICP-MS and collision/reaction cell ICP-MS instrumentation are suitable for measurement.

6.3 Mass-flow controller

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

6.4 Nebuliser with variable speed peristaltic pump

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

6.5 Gas supply

6.5.1 Argon, Ar, with high purity grade, i.e. > 99,99 %.

6.5.2 Reaction gas, e.g. Helium (He), Hydrogen (H_2), Oxygen (O_2), ammonia gas (NH_3), or methane (CH_4) with high purity grade, i.e. > 99,99 %.

6.6 Storage bottles for the stock, standard, calibration and sample solutions.

Preferably made from perfluoroalkoxy alkane (PFA) or hexafluoroethene propene (FEP). For the determination of elements in a higher concentration range (> 1 µg/kg), high density polyethylene (HDPE) or polytetrafluoroethene (PTFE) bottles may be suitable.

7 Procedure

7.1 Test sample solution

The test sample solution is a particle-free digest or extraction solution prepared according to EN 16173 or EN 16174.

7.2 Test solution

The test solution is an aliquot of the test sample solution and may be directly obtained from the test sample solution or may be diluted to accommodate the measurement range or to dilute the matrix.

The acidity of calibration solutions shall match the acid concentration in test solutions.

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

7.3 Instrument set-up

Adjust the instrumental parameters of the ICP-MS system in accordance with the manufacturer's instructions. A guideline for method and instrument set up is given in EN ISO 17294-1:2006.

Define the isotopes and the need for corresponding corrections. See EN ISO 17294-1:2006, 6.3.2 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 mandatory. Add the internal standard solution (5.8) to the interference check solution (5.12), to all multi-element calibration solutions (5.7), to the calibration blank solutions (5.9), and to all measuring solutions.

NOTE 1 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 cases, the calibration solutions are diluted the same way as the sample solutions.

The mass concentration of the internal standard elements shall be the same in all solutions. Generally, a suitable concentration of the internal standard element in sample and calibration solutions is 1 µg/l to 50 µg/l (for a high and stable count rate).

Adjust the instrument to working condition. This takes usually 30 min.

Before each series of measurements check the sensitivity and the stability of the system and minimise interferences, e.g. by using the optimisation solution (5.11).

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

NOTE 2 ICP-MS has excellent multi-element capability. Nevertheless it does not mean that all elements can be analysed during one measurement run. The sensitivity of determination depends on numerous parameters (nebuliser flow, radio-frequency power, lens voltage, lens voltage mode etc.). The optimal instrument settings cannot be reached for all elements at once.

7.4 Calibration

7.4.1 Linear calibration function

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

NOTE 1 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. Consequently, 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.

NOTE 2 An alternative, but less efficient, approach is ordinary linear regression where the line is forced through the blank value or through zero.

A two point calibration is allowed if the calibration function is linear, which is usually the case. Check regularly for linearity with a calibration solution of known dilution.

Instead of one measurement per level, more measurements can be performed to reduce the uncertainty of the calibration line.

7.4.2 Standard addition calibration

Add a known amount of standard solution of the analyte and an equal amount of blank solution 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 times 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.

7.4.3 Determination of correction factors

The need for the use of correction factors is determined during method development. Correction factors should be evaluated and updated, for example by measuring interference check solutions (5.12) at regular intervals within a sequence.

NOTE For example the interference correction factor for $^{40}Ar^{35}Cl^+$ on ^{75}As is determined by recording the signal at mass 75 and 35 of a Cl^- solution; the ratio of the net signal at mass 75 and net signal at mass 35 is the correction factor. For the isobaric molecular interference of $^{98}Mo^{16}O^+$ the correction factor is determined by the recording of the signal at mass 114 and 98 of a Mo solution.

7.4.4 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).

7.5 Sample measurement

The following steps are an example of an appropriate measurement procedure.

Run at least one measurement using multi-element calibration solutions (5.7) and a calibration blank solution (5.9).

Run the interference check solution(s) (5.12) to establish interference correction or to check for presence of interference.

Every 25 samples or less and at the beginning and end, run a calibration blank solution (5.9) and a multi-element calibration solution (5.7) of an independent source.

Every (e.g.) 50 samples and at the end of a run, analyse an interference check solution (5.12).

Run all samples including one or more test blank solution (5.10).

Run at least one post digestion spiked sample from the series to check recovery. If standard addition calibration is applied to all samples, leave out recovery check.

Some elements (e.g. 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 sample with unknown matrix composition is encountered, check:

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

8 Calculation

Calculate the element concentration in the digested solid sample using Formula (1):

$$w_i = \frac{\left(\rho_1 - \rho_0\right) \cdot f \cdot V \cdot C}{m} \tag{1}$$

with

$$C = 100 / w_{\mathrm{dm}} \tag{2}$$

where

w_i	is the mass fraction of the element in the solid sample *i*, expressed in milligrams per kilogram (mg/kg) corrected to dry matter at 105 °C;
ρ_1	is the concentration of the element in the test solution (7.2), expressed in milligrams per liter (mg/l);
ρ_0	is the concentration of the element in the test blank solution (5.10), expressed in milligrams per liter (mg/l);
f	is the dilution factor of the test solution (7.2);
V	is the volume of the digest solution prepared according to EN 16173 or EN 16174, expressed in liters (l);
m	is the mass of the digested test portion, expressed in kilograms (kg);
C	is the correction factor for dry mass;
w_{dm}	is the dry matter fraction of the sample, determined according to EN 15934, in per cent (%).

9 Expression of results

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

10 Performance characteristics

10.1 Blank

The 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.

10.2 Calibration check

For demonstration of calibration traceability, a calibration verification solution with certified concentration and known measurement uncertainty shall be used. Additionally, this solution or a calibration solution may be used for drift control during the measurement cycle. The accepted deviation shall be in the limit of the laboratory quality control policy.

10.3 Internal standard response

The relative response of the internal standard in all solutions shall be between 70 % and 130 % of the internal standard in the preceding blank or calibration check solution.

If the internal standard response deviates for specific sample measurement solutions then either an error in internal standard addition to the affected solution has been made or an intolerable physical interference has occurred (4.2.3). Prepare new solutions ensuring that internal standard solution is added correctly and analyse. Sample measurement solutions may need to be additionally diluted five-fold (10.5) if physical interference has occurred. If the internal standard response deviates for all solutions, clean the cones and recalibrate.

10.4 Interference

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

EXAMPLE 1 Assume that the concentration for calcium in an interference check solution 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 test sample is lower than 200 mg/l) the concentration of the interfered isotope (Ni60) can be reported.

EXAMPLE 2 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 times the instrumental detection limit or 0,5 times the lowest concentration to be reported) then the maximum concentration of the interfering element (Ca) in the test sample on the interfered isotope (Ni60) is 400 mg/l (= 1/0,5 · 200 mg/l assuming a linear relation). Otherwise Ni60 cannot be reported prior to further investigation of the interference.

These examples refer to the following assumptions: the contribution of corrected and uncorrected isobaric molecular and doubly-charged ions should neither be higher than 3 times the instrumental detection limit in trace analysis nor be higher than half the lowest concentration to be reported.

10.5 Recovery

Spike recovery shall be between 75 % and 125 %. The variation between the results of the original sample and the diluted sample shall be less than 20 % if the concentration of the five-fold dilution is > 3 times of the instrumental detection limit or > 2 times of the lowest concentration to be reported. Spike concentration should be within 0,4 times and 2,5 times the analyte concentration.

10.6 Performance data

The performance characteristics have been determined in an international validation study. The results of this study are given in Annex A.

11 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 16171);

b) all information necessary for identification of the sample;

c) information about the pretreatment and method of digestion of the sample;

d) results of the determination as indicated in Clause 9;

e) any details not specified in this European Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Repeatability and reproducibility data

Table A.1 — Repeatability and reproducibility data for sludge

Element	**Extraction method**	l	n_o	N_ev	n	$\bar{\bar{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
Ag	EN 16173	16	1	15	45	4,968	0,391	7,9	1,940	39,1
	EN 16174, Method A	13	0	13	39	6,137	0,147	2,4	0,612	10,0
	EN 16174, Method B	16	1	15	45	6,256	0,426	6,8	0,888	14,2
As	EN 16173	16	0	16	48	3,778	0,129	3,4	0,842	22,3
	EN 16174, Method A	13	1	12	36	3,397	0,123	3,6	0,503	14,8
	EN 16174, Method B	16	0	16	48	3,901	0,169	4,3	0,997	25,6
Cd	EN 16173	16	1	15	45	0,972	0,045	4,6	0,160	16,4
	EN 16174, Method A	13	0	13	38	0,991	0,04	4,0	0,223	22,5
	EN 16174, Method B	16	0	16	48	1,007	0,054	5,4	0,175	17,4
Co	EN 16173	16	1	15	45	2,681	0,095	3,6	0,295	11,0
	EN 16174, Method A	13	0	13	39	2,337	0,065	2,8	0,294	12,6
	EN 16174, Method B	16	0	16	48	2,820	0,207	7,3	0,382	13,6
Cr	EN 16173	16	2	14	42	18,38	0,69	3,7	2,37	12,9
	EN 16174, Method A	13	1	12	36	18,86	0,75	4,0	1,68	8,9
	EN 16174, Method B	16	1	15	45	19,51	1,19	6,1	2,30	11,8
Cu	EN 16173	15	1	14	42	944,4	22,4	2,4	107,7	11,4

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
	EN 16174, Method A	12	1	11	33	953,5	22,0	2,3	79,3	8,3
	EN 16174, Method B	15	1	14	42	952,5	36,3	3,8	103,7	10,9
Hg	EN 16173	12	1	11	33	0,582	0,068	11,7	0,143 2	24,6
	EN 16174, Method A	10	0	10	30	0,605	0,055	9,2	0,150	24,8
	EN 16174, Method B	12	0	12	36	0,633	0,077	12,1	0,184	29,0
Ni	EN 16173	16	1	15	45	18,66	0,68	3,7	1,65	8,9
	EN 16174, Method A	13	1	12	36	18,94	0,7	3,7	1,19	6,3
	EN 16174, Method B	16	0	16	48	19,09	0,84	4,4	2,12	11,1
Pb	EN 16173	16	1	15	45	27,99	0,96	3,4	3,65	13,0
	EN 16174, Method A	13	0	13	39	28,26	0,70	2,5	2,79	9,9
	EN 16174, Method B	16	0	16	48	28,04	1,14	4,1	3,21	11,4
Se	EN 16173	13	1	12	36	1,883	0,149	7,9	0,345	18,3
	EN 16174, Method A	11	2	9	27	1,811	0,033	1,8	0,196	10,8
	EN 16174, Method B	13	1	12	36	1,861	0,177	9,5	0,292	15,7
Sn	EN 16173	16	1	15	45	12,20	2,28	18,7	8,99	73,7
	EN 16174, Method A	13	1	12	36	34,75	1,01	2,9	3,30	9,5
	EN 16174, Method B	16	0	16	48	36,69	2,53	6,9	5,79	15,8
V	EN 16173	13	0	13	39	5,642	0,175	3,1	0,705	12,5
	EN 16174, Method A	11	0	11	33	5,440	0,196	3,6	0,598	11,0
	EN 16174,	12	1	11	33	6,132	0,141	2,3	0,785	12,8

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
	Method B									
Zn	EN 16173	15	0	15	45	1025	26	2,6	134	13,1
	EN 16174, Method A	13	1	12	36	1052	21	2,0	99	9,4
	EN 16174, Method B	15	3	12	36	1045	22	2,1	117	11,2

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
N_ev	Number of evaluated datasets
n_o	number of outlier laboratories
$\overline{\overline{x}}$	total mean of results (without outliers) in milligram per kilogram (mg/kg)
s_R	reproducibility standard deviation in milligram per kilogram (mg/kg)
$C_{V,R}$	coefficient of variation of reproducibility in percent (%)
s_r	repeatability standard deviation in milligram per kilogram (mg/kg)
$C_{V,r}$	coefficient of variation of repeatability in percent (%)

Table A.2 — Repeatability and reproducibility data for compost

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
Ag	EN 16173	12	0	12	36	0,180	0,023	12,8	0,075	41,4
	EN 16174, Method A	10	1	9	26	0,212	0,019	8,8	0,058	27,3
	EN 16174, Method B	12	0	12	36	0,238	0,036	15,1	0,095	40,0
As	EN 16173	16	2	14	42	5,041	0,162	3,2	0,699	13,9
	EN 16174, Method A	13	1	12	36	5,101	0,172	3,4	0,727	14,3
	EN 16174, Method B	16	1	15	45	5,483	0,238	4,4	1,069	19,5

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
Cd	EN 16173	16	3	13	39	0,554	0,024	4,3	0,051	9,2
	EN 16174, Method A	13	1	12	36	0,572	0,023	4,1	0,042	7,3
	EN 16174, Method B	16	1	15	45	0,583	0,034	5,9	0,065	11,2
Co	EN 16173	16	1	15	45	6,934	0,217	3,1	0,614	8,9
	EN 16174, Method A	13	0	13	39	6,898	0,188	2,7	0,630	9,1
	EN 16174, Method B	16	0	16	48	7,041	0,191	2,7	0,894	12,7
Cr	EN 16173	16	0	16	48	29,46	1,85	6,3	6,30	21,4
	EN 16174, Method A	13	0	13	39	29,73	1,12	3,8	3,04	10,2
	EN 16174, Method B	16	0	16	48	34,35	2,42	7,1	7,33	21,3
Cu	EN 16173	15	0	15	45	43,68	1,17	2,7	5,23	12,0
	EN 16174, Method A	12	2	10	30	43,86	1,31	3,0	4,44	10,1
	EN 16174, Method B	15	3	12	36	44,34	1,25	2,8	4,34	9,8
Hg	EN 16173	10	1	9	27	0,0911	0,0115	12,6	0,0260	28,5
	EN 16174, Method A	8	0	8	24	0,0867	0,0203	23,4	0,0225	26,0
	EN 16174, Method B	11	0	11	31	0,0891	0,0234	26,3	0,0254	28,5
Ni	EN 16173	16	0	16	48	23,56	0,98	4,2	3,41	14,5
	EN 16174, Method A	13	1	12	36	23,31	0,43	1,8	3,08	13,2
	EN 16174, Method B	16	0	16	47	24,64	1,26	5,1	3,99	16,2
Pb	EN 16173	16	1	15	44	34,42	2,01	5,8	2,83	8,2
	EN 16174, Method A	13	1	12	36	34,06	1,29	3,8	2,96	8,7

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
	EN 16174, Method B	16	1	15	45	33,51	1,42	4,2	3,86	11,5
Se	EN 16173	11	1	10	30	0,458	0,053	11,5	0,324	70,8
	EN 16174, Method A	11	1	10	28	0,364	0,047	12,9	0,176	48,3
	EN 16174, Method B	12	1	11	32	0,495	0,076	15,3	0,375	75,7
Sn	EN 16173	15	0	15	45	1,339	0,280	20,9	0,849	63,4
	EN 16174, Method A	13	1	12	36	2,713	0,149	5,5	0,393	14,5
	EN 16174, Method B	16	0	16	48	3,019	0,402	13,3	0,640	21,2
V	EN 16173	13	1	12	36	23,86	1,11	4,7	6,56	27,5
	EN 16174, Method A	12	0	12	36	25,82	0,56	2,2	2,90	11,2
	EN 16174, Method B	13	0	13	39	30,95	2,13	6,9	7,52	24,3
Zn	EN 16173	15	1	14	42	168,4	5,5	3,2	20,2	12,0
	EN 16174, Method A	13	1	12	36	166,6	3,1	1,9	18,0	10,8
	EN 16174, Method B	15	1	14	42	167,7	4,7	2,8	19,5	11,6
Explanation of symbols see Table A.1										

Table A.3 — Repeatability and reproducibility data for soil

Element	**Extraction method**	l	n_o	N_ev	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
Ag	EN 16173	15	1	14	42	0,620	0,043	6,9	0,199	32,2
	EN 16174, Method A	13	3	10	30	0,614	0,026	4,2	0,036	5,8
	EN 16174, Method B	15	0	15	43	0,688	0,062	9,0	0,185	26,9
As	EN 16173	16	0	16	48	44,95	1,14	2,5	4,34	9,7

Element	**Extraction method**	l	n_{o}	N_{ev}	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
	EN 16174, Method A	13	2	11	33	46,01	1,19	2,6	2,86	6,2
	EN 16174, Method B	16	0	16	48	45,25	1,27	2,8	3,50	7,7
Cd	EN 16173	16	0	16	48	13,06	0,51	3,9	1,63	12,5
	EN 16174, Method A	13	1	12	36	13,44	0,56	4,2	0,84	6,2
	EN 16174, Method B	16	1	15	45	13,40	0,50	3,7	0,92	6,9
Co	EN 16173	16	1	15	45	3,881	0,102	2,6	0,247	6,4
	EN 16174, Method A	13	2	11	32	3,792	0,089	2,3	0,324	8,5
	EN 16174, Method B	16	0	16	48	3,908	0,128	3,3	0,281	7,2
Cr	EN 16173	16	0	16	48	21,18	0,81	3,8	1,94	9,2
	EN 16174, Method A	13	1	12	36	21,09	0,73	3,5	1,83	8,7
	EN 16174, Method B	16	0	16	48	21,78	1,08	5,0	1,89	8,7
Cu	EN 16173	15	0	15	45	34,37	1,36	4,0	3,60	10,5
	EN 16174, Method A	12	0	12	36	34,35	1,47	4,3	3,47	10,1
	EN 16174, Method B	15	1	14	42	35,54	1,50	4,2	2,25	6,3
Hg	EN 16173	12	0	12	36	0,771	0,055	7,1	0,166	21,5
	EN 16174, Method A	10	0	10	30	0,851	0,055	6,5	0,105	12,3
	EN 16174, Method B	12	1	11	33	0,876	0,030	3,4	0,106	12,1
Ni	EN 16173	16	0	16	48	6,767	0,460	6,8	0,805	11,9
	EN 16174, Method A	13	1	12	36	7,004	0,343	4,9	0,658	9,4
	EN 16174, Method B	16	2	14	42	7,094	0,369	5,2	0,539	7,6

Element	**Extraction method**	l	n_o	N_{ev}	n	$\overline{\overline{x}}$	s_r	$C_{V,r}$	s_R	$C_{V,R}$
Pb	EN 16173	16	1	15	45	75,25	3,58	4,8	5,60	7,5
	EN 16174, Method A	13	0	13	39	74,74	3,04	4,1	6,84	9,2
	EN 16174, Method B	16	2	14	42	74,60	4,03	5,4	5,36	7,2
Se	EN 16173	12	1	11	32	0,484	0,067	13,9	0,213	44,0
	EN 16174, Method A	12	2	10	30	0,531	0,080	15,0	0,110	20,7
	EN 16174, Method B	12	1	11	33	0,577	0,067	11,6	0,238	41,2
Sn	EN 16173	16	0	16	46	2,538	0,825	32,5	1,414	55,7
	EN 16174, Method A	13	1	12	36	10,58	0,84	7,9	1,33	12,6
	EN 16174, Method B	16	0	16	48	11,38	2,38	20,9	2,59	22,8
V	EN 16173	13	0	13	39	6,021	0,271	4,5	0,837	13,9
	EN 16174, Method A	12	0	12	36	6,467	0,265	4,1	0,724	11,2
	EN 16174, Method B	13	0	13	39	7,443	0,461	6,2	1,221	16,4
Zn	EN 16173	15	3	12	36	170,3	2,4	1,4	19,5	11,4
	EN 16174, Method A	13	0	13	39	173,45	5,4	3,3	15,8	9,1
	EN 16174, Method B	15	2	13	39	174,1	2,8	1,6	16,5	9,5
Explanation of symbols see Table A.1										

Annex B
(informative)

Selected isotopes and spectral interferences for quadrupole ICP-MS instruments

For selected isotopes and spectral interferences for quadrupole ICP-MS instruments see EN ISO 17294-1.

Table B.1 — Examples of equations for isobaric overlay

Element	Recommended isotope and inter-element correction[a]
As	^{75}As - 3,127(^{77}Se - 0,815 ^{82}Se) or ^{75}As - 3,127(^{77}Se - 0,3220 ^{78}Se)
Ba	^{138}Ba - 0,000 900 8 ^{139}La - 0,002 825 ^{140}Ce
Cd	^{114}Cd - 0,026 84 ^{118}Sn
Ge	^{74}Ge - 0,138 5 ^{82}Se
In	^{115}In - 0,014 86 ^{118}Sn
Mo	^{98}Mo - 0,110 6 ^{101}Ru
Ni	^{58}Ni - 0,048 25 ^{54}Fe
Pb	^{208}Pb + ^{207}Pb + ^{206}Pb
Se	^{82}Se - 1,009 ^{83}Kr
Sn	^{120}Sn - 0,013 44 ^{125}Te
V	^{51}V - 3,127(^{53}Cr - 0,1134 ^{52}Cr)
W	^{184}W - 0,001 242 ^{189}Os

[a] Note that the coefficients shown are based on tabulated natural abundances and should be corrected for instrumental mass discrimination on the ICP-MS equipment used.

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[1] Umweltbundesamt, Texte 42/2015: Validierung von Analyseverfahren, die im Rahmen des Projektes HORIZONTAL entwickelt, jedoch nicht validiert wurden.
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