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Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil-like materials —

Part 2: Batch test using a liquid to solid ratio of 10 l/kg dry matter

Qualité du sol — Modes opératoires de lixiviation en vue d'essais chimiques et écotoxicologiques ultérieurs des sols et matériaux du sol —

Partie 2: Essai en bâchée avec un rapport liquide/solide de 10 l/kg de matière sèche

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Impact assessment*.

This first edition of ISO 21268-2:2019 cancels and replaces the first edition (ISO/TS 21268-2:2007), which has been technically revised. The main changes compared to the previous edition are as follows:

— the maximum grain size has been changed to <2 mm as usual for soil;

— the demineralized water has been added as possible leachant;

— 7.1 and 7.2 have been renumbered and renamed to read 7.1 "Particle size" and 7.2 "Sample preparation";

— 12.1 "General" and 12.2 "Validation results obtained for DIN 19529" have been added;

— A.3.6 "Special requirements for tests considering semi-volatile substances" has been added;

— a new informative Annex C "Calculation of centrifugation duration depending on centrifugation speed and rotor dimensions" has been added;

— references in Clause 2 and the Bibliography have been updated.

A list of all parts in the ISO 21268 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In various countries, tests have been developed to characterize and assess the substances which can be released from materials. The release of soluble substances upon contact with water is regarded as a main mechanism of release, which results in a potential risk to the environment during the use or disposal of materials. The intent of these tests is to identify the leaching properties of materials. The complexity of the leaching process makes simplifications necessary[1].

Not all of the relevant aspects of leaching behaviour can be addressed in one standard (see description of influencing factors in Annex A).

Tests to characterize the behaviour of materials can generally be divided into three categories addressed in ISO 18772[2] and EN 12920[3]. The relationships between these tests are summarized below.

a) "Basic characterization" tests are used to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials. Liquid/solid ratios (L/S), leachant composition, factors controlling leachability, such as pH, redox potential, complexing capacity, role of dissolved organic carbon (DOC), ageing of material and physical parameters, are addressed in these defined tests.

b) "Compliance" tests are used to determine whether the material complies with a specific behaviour or with specific reference values. These tests focus on key variables and leaching behaviour previously identified by basic characterization tests.

c) "On-site verification" tests are used as a rapid check to confirm that the material is the same as that which has been subjected to the compliance test(s). On-site verification tests are not necessarily leaching tests.

The test procedure described in this method belongs to category b): compliance tests.

This document was originally elaborated on the basis of EN 12457-2:2004[4]. Especially, modifications considering requirements on subsequent ecotoxicological testing and analysis of organic substances have been included. Validation results have been adopted from DIN 19529[5].

Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil-like materials —

Part 2:
Batch test using a liquid to solid ratio of 10 l/kg dry matter

1 Scope

This document specifies a test providing information on leaching of soil and soil materials under the experimental conditions specified hereafter, and particularly at a liquid to solid ratio of 10 l/kg dry matter.

The document has been developed to measure the release of inorganic and organic substances from soil and soil-like material as well as to produce eluates for subsequent ecotoxicological testing. For ecotoxicological testing, see ISO 15799[6] and ISO 17616[7].

NOTE 1 Volatile organic substances include the low-molecular-weight substances in mixtures such as mineral oil.

NOTE 2 It is not always possible to optimize test conditions simultaneously for inorganic and organic substances and optimum test conditions can also vary between different groups of organic substances. Test requirements for organic substances are generally more stringent than those for inorganic substances. The test conditions suitable for measuring the release of organic substances will generally also be applicable to inorganic substances.

NOTE 3 Within the category of organic substances, a significant difference in behaviour exists between the more polar, relatively water-soluble compounds and apolar, hydrophobic organic substances (HOCs). In the latter case, mechanisms of release (e.g. particle-bound or dissolved organic carbon-bound) can be more crucial as well as sorption losses of soluble HOCs on different materials with which they come in contact (e.g. bottles, filters). The test and the results should be used for leaching of organic substances only with thorough consideration of the specific properties of the substances in question and the associated potential problems.

NOTE 4 For ecotoxicological testing, eluates representing the release of both inorganic and organic substances are needed. In this document, ecotoxicological testing is also meant to include genotoxicological testing.

This test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods in accordance with existing standard methods. The test is not suitable for substances that are volatile under ambient conditions.

This procedure is not applicable to materials with a dry-matter-content ratio lower than 33 %.

This test is mainly aimed at being used for routine and control purposes, and it cannot be used alone to describe all leaching properties of a soil. Additional leaching tests are needed for that extended goal. This document does not address issues related to health and safety. It only determines the leaching properties as outlined in Clause 4.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 7027-1, *Water quality — Determination of turbidity — Part 1: Quantitative methods*

ISO 10523, *Water quality — Determination of pH*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
leaching test
test during which a material is put into contact with a *leachant* (3.2) under strictly defined conditions and some substances of the material are extracted

3.2
leachant
liquid used in a *leaching test* (3.1)

Note 1 to entry: For the purpose of this document, the leachant is specified in 5.1.

3.3
eluate
solution recovered from a *leaching test* (3.1)

Note 1 to entry: Eluate is also referred to as leachate.

3.4
liquid to solid ratio
L/S
ratio between the total volume of liquid (L in litres), which in this extraction is in contact with the soil sample, and the dry mass of the sample (S in kg of dry matter)

Note 1 to entry: L/S is expressed in l/kg.

3.5
dry matter content
w_{dm}
ratio, expressed in percent, between the mass of the dry residue, determined in accordance with ISO 11465, and the corresponding raw mass

3.6
water content
$w_{\mathrm{H_2O}}$
ratio, expressed in percent, between the mass of water contained in the material as received and the corresponding dry residue of the material

Note 1 to entry: The basis for the calculation of the water content is the mass of the dry residue in this document, as specified in ISO 11465 (for the determination of the water content of soil).

3.7
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

3.8
test sample
sample, prepared from the *laboratory sample* (3.7), from which *test portions* (3.9) are removed for testing or analysis

3.9
test portion
quantity of material of appropriate size for measurement of the concentration or other properties of interest taken from the *test sample* (3.8)

Note 1 to entry: The test portion can be taken from the *laboratory sample* (3.7) directly if no pre-treatment of the sample is required, but usually it is taken from the test sample.

Note 2 to entry: A unit or increment of proper homogeneity, size and fineness, needing no further preparation, can be a test portion.

3.10
soil-like material
excavated soil, dredged materials, manufactured soils, treated soils and fill materials[7]

4 Principle

The test portion, which originally or after suitable pre-treatment has a particle size less than or equal to 2 mm, is brought into contact with water containing a low concentration (0,001 mol/l) of calcium chloride or demineralized water (5.1) under defined conditions. The standard method is based on the assumption that equilibrium or near-equilibrium is achieved between the liquid and solid phases during the test period. The solid residue is subsequently separated from the liquid. The separation procedure may strongly influence the test results and shall be particularly stringent for organic substances. The properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates, and the eluate may be subjected to subsequent ecotoxicological testing.

After the test, the leaching conditions imposed by the material, in terms of pH, electrical conductivity and, optionally, DOC, redox potential or turbidity, shall be recorded.

NOTE 1 These parameters often control the leaching behaviour of soil and soil-like materials and are therefore important for evaluation of the test results. DOC, in particular, is crucial in soil and soil materialsoil-like materials for many inorganic and organic substances.

NOTE 2 The leachant is 0,001 mol/l $CaCl_2$ to minimize the mobilization of DOC caused by an ionic strength of the leachant which is too low.

The procedure described in this document is based on the more stringent test requirements for determining the release of organic substances and for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, less stringent requirements may be adopted for some steps of the procedure.

5 Reagents

5.1 Demineralized water or **deionized water** or **water of equivalent purity** (5 < pH < 7,5) with a conductivity of <0,5 mS/m in accordance with grade 3 specified in ISO 3696 made to **0,001 mol/l $CaCl_2$**.

5.2 Calcium chloride ($CaCl_2 \cdot 2\ H_2O$), analytical grade.

5.3 Sodium azide (NaN_3), analytical grade.

5.4 Nitric acid (HNO_3), analytical grade, made to 0,1 mol/l rinsing solution.

5.5 Organic solvent (acetone, analytical grade) for rinsing and cleaning.

6 Apparatus

6.1 Borosilicate glass, of high purity in accordance with ISO 5667-3, with a nominal volume of 1 l, **glass bottles** having caps of inert material, for example PTFE (polytetrafluoroethylene). Rinsing is compulsory and it should be assured that previously used bottles have no background level of analytes.

NOTE 1 If only inorganic parameters are analysed, alternative materials, such as HDPE/PP bottles, are appropriate, except for unpreserved samples for mercury analysis.

NOTE 2 To prevent organic compounds from degradation by light use a dark room, dark colored glassware or place a layer of aluminium-foil around the leaching equipment.

If boron analyses are necessary, any plastics bottles can be used, e.g. PTFE (polytetrafluoroethylene).

The volume of 1 l is selected in combination with the mass, m_D, of 100 g as specified in 7.4 in order to minimize head-space in the bottle at an L/S of 10 l/kg dry matter. In the case of materials with low density, deviation from this requirement can be necessary while still ensuring minimum headspace. This deviation shall be reported.

NOTE 3 Glass of high quality is considered adequate for both metals and organic substances, particularly, since the pH range usually covered in soil testing does not reach the conditions (pH > 10 and pH < 3) where glass itself can be partially dissolved. For ecotoxicity testing, eluates with both inorganic and organic substances are needed, which emphasizes the need to generate integrated eluates.

NOTE 4 Heat treatment of used glassware at 550 °C can be used to remove traces of analytes. However, this treatment has been shown to increase adsorption of organic substances from the air.

6.2 Glass bottle, of high quality (requirements as in 6.1) with a nominal volume of e.g. 5 l, to be used when samples from replicate tests are recombined after centrifugation for further analysis or testing.

6.3 End-over-end tumbler (5 min^{-1} to 10 min^{-1}) **or roller table,** rotating at about 10 min^{-1}.

Other shaking devices may be used provided that they can be shown to provide equivalent results. These agitation devices are specified because excessive abrasion leading to significant particle size reduction should be avoided.

6.4 Filtration apparatus, either a vacuum filtration device (between 2,5 kPa and 4,0 kPa) or a high-pressure filtration apparatus (<0,5 MPa). Rinsing is compulsory. When semi-volatile substances are to be analysed, vacuum filtration shall not be used.

6.5 0,45 µm membrane filters, pre-rinsed or similarly cleaned [e.g. rinsed with 0,1 mol/l HNO_3 (5.2) and water (5.1)] (only for analysis of inorganic substances).

The filters shall be chosen so as not to adsorb (or release) substances of interest.

NOTE This can be tested in preliminary experiments.

6.6 Glass fibre filters, with a degree of separation of 0,7 µm.

The filters shall be chosen so as not to adsorb (or release) substances of interest.

NOTE This can be tested in preliminary experiments.

6.7 Sieving equipment, with sieves of 2 mm nominal screen size.

NOTE Due to sieving, contamination of the sample can occur to an extent which affects the leaching of some substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment or plasticizers from plastic sieves.

6.8 Centrifuge, operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of PFA (perfluoroalkoxy alkane), fluorinated ethylene propylene (FEP) or tubes of an alternative material which is inert with regard to both inorganic and organic compounds and suitable for high-speed centrifugation[8].

NOTE Potential sorption of hydrophobic organic substances to the centrifuge tubes can be tested in preliminary experiments.

Alternatively, if a high-speed centrifuge is not available, a centrifuge operating at 2 000 *g* to 3 000 *g* using glass bottles may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

6.9 Device for measuring electrical conductivity.

6.10 pH meter, in accordance with ISO 10523 with an accuracy of at least ±0,05 pH units.

6.11 Thermometer, for air temperature measurement.

6.12 Redox potential meter, (optional).

6.13 Balance, with an accuracy of at least 0,1 g.

6.14 Measuring cylinders, for volume determination with 1 % accuracy.

6.15 Sample splitter, for sub-sampling of laboratory samples (optional).

6.16 Turbidity meter, as specified in ISO 7027-1.

6.17 Crushing equipment, a jaw crusher.

NOTE Due to particle size reduction, contamination of the sample can occur to an extent which affects the leaching of some substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment.

7 Sample pretreatment

7.1 Preparation of laboratory sample and specification of particle size

A representative laboratory sample of at least 2 kg (dry matter) is obtained (e.g. as described in ISO 18400-101, ISO 18400-104, ISO 18400-105, ISO 18400-202[10-13] and ISO 23909[14]) and shall be stored in closed packages and at low temperatures (4 °C), in order to avoid unwanted changes in the material (see e.g. ISO 18400-105[12]).

The test shall be carried out on soil or soil-like material sieved to <2 mm (e.g. as described in ISO 11464[9]). Oversized material of natural origin in the sample shall be separated and discarded. The type and amount of all discarded material shall be reported. If oversized material of anthropogenic origin is present and assumed to contain substances of interest, this part can be subject to alternative sample preparation or testing.

If the laboratory sample cannot be homogenized or sieved because of its water content, it is allowed in this case only to dry the laboratory sample (e.g. as described in ISO 11464[9]). The drying temperature shall not exceed 30 °C.

NOTE 1 Sieving and drying at more than 30 °C, as well as crushing, can lead to a loss of semi-volatile substances (inorganic and organic) and can alter the leaching characteristics (refer also to A.3.6).

NOTE 2 Due to sieving, contamination of the sample can occur to an extent that affects the leaching of some substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment or plasticizers from plastic sieves.

NOTE 3 Due to sieving, contamination of the sample can occur to an extent which affects the leaching of some substances of concern, e.g. chromium, nickel and molybdenum form stainless steel equipment or plasticisers from plastic sieves.

7.2 Preparation of test sample

Use a sample splitter (6.15) or apply coning and quartering to split the laboratory sample and obtain a test sample. The size of test sample required depends on the volume of eluate needed for the specific purpose and the subsequent chemical analysis and/or ecotoxicological tests to be carried out on the eluate.

NOTE 1 If needed for chemical analysis or ecotoxicological testing, larger volumes of eluate can be obtained by combining eluates from replicate tests after centrifugation (or filtration). Alternatively, larger volumes of eluate can also be produced in a single test, provided that the ratios in terms of L/S and minimum headspace are maintained.

NOTE 2 The required amount of the test sample is dependent on the particle size distribution of the soil to be analysed (see ISO 23909[14]). The specified sample amount will generally be adequate. In specific cases, a smaller sample amount can be accepted, for instance, if for specific reasons less material is available, provided that the test can be carried out as specified in 7.2 to 7.4.

7.3 Determination of dry matter content and water content

The whole test sample, complying with the size criterion in 7.1, shall not be further dried. The water content of the test sample shall be determined on a separate test portion at (105 ± 5) °C. If the soil sample is air-dried prior to testing, the dry matter content w_{dm} of the air-dried sample shall be determined as well. This shall be taken into account when adjusting the L/S. The dry mass of the sample shall be determined at (105 ± 5) °C in accordance with ISO 11465 and the dry matter content is calculated in Formula (1).

$$w_{dm} = 100 \times m_D / m_W \quad (1)$$

where

w_{dm} is the dry matter content, expressed in percent (%);

m_D is the mass of the dried sample, expressed in kilograms (kg);

m_W is the mass of the undried sample, expressed in kilograms (kg).

The water content (w_{H_2O} in %) is calculated as following Formula (2):

$$w_{H_2O} = 100 \times (m_W - m_D) / m_D \quad (2)$$

NOTE If volatile or unstable compounds are present in the soil sample, this gravimetric method cannot be used for accurate determination of the water content.

If, for reasons expressed in 7.1, the material was (partly) dried before sample splitting, the overall mass loss shall be taken into account.

7.4 Preparation of the test portion

Prepare, from the test sample, a test portion with a total mass m containing (100 ± 5) g (measured with an accuracy of 0,1 g, 6.13) of dry mass (m_D), following Formula (3).

$$m = 100 \times m_D / w_{dm} \quad (3)$$

Use a sample splitter (6.15) or apply coning and quartering to split the sample.

NOTE Sample splitting or coning and quartering can lead to loss of semi-volatile substances (inorganic and organic).

In view of the minimum requirements of eluate volume for analytical purposes, it may be necessary to use a larger test portion and a correspondingly larger volume of leachant. This deviation from this document shall be specified in the test report.

If the test is performed on an air-dried sample, use $w_{dm,AD}$ instead of w_{dm} to determine the sample mass of the test portion.

8 Procedure

8.1 Temperature

The compliance test for leaching shall be carried out at room temperature: (22 ± 3) °C.

For material that is very sensitive to biological degradation, performance of the test at reduced temperature (e.g. 4 °C) and preventing direct exposure to light will limit biological activity significantly. A reduced temperature may result in slower/lower release of organic substances and hence lower concentrations of these compounds in the leachates. If the test is modified in this way, this deviation shall be reported in the test report.

8.2 Description of the procedure

8.2.1 Preparation of the eluent

Prepare a solution made to 0,001 M $CaCl_2$ by dissolving 0,147 g $CaCl_2$ in water and dilute to 1 000 ml.

In special cases (i.e. measurement of Ca and/or chloride in the eluate are of interest or the sample exhibits an own salt load), water without addition of $CaCl_2$ can also be used. The leachant type used shall be recorded in the test report.

NOTE 1 The application of demineralized water as leachant can induce higher turbidity and lower ionic strength in the eluate for some types of soils (e.g. high content of organic matter) and can cause increased concentrations of analytes adsorbed to colloids.

NOTE 2 For eluates that are not to be used for ecotoxicological testing, sodium azide (NaN_3) can be added to a resulting concentration of 0,1 % in order to reduce microbial degradation of organic substances. However, the addition of NaN_3 is known to only minimize biodegradation if a very high but in turn extremely poisonous concentration in the eluent is applied. Therefore, other appropriate measures can be considered to prevent/reduce biodegradation in the sample or collected eluate (e.g. application of γ-radiation to the sample, dark and air-conditioned room, shorter eluate collection periods, etc). If only inorganic compounds are measured, the addition of NaN_3 is not appropriate.

8.2.2 Leaching step

Place the test portion with the total mass m corresponding to (100 ± 5) g of dry mass m_D in a bottle (6.1).

Depending on the particle size distribution, other test portions may be applied ensuring that a representative portion is used (see 7.1).

Add an amount of leachant (V_L) using a balance (6.13) or measuring cylinder (6.14), to establish a liquid to solid ratio (L/S) of (10 ± 0,2) l/kg during the extraction, following Formula (4). Care shall be taken to obtain good mixing of solid and liquid.

$$V_L = \left[10 - w_{H_2O} / \left(\rho_{H_2O} \times 100\right)\right] \times m_D \quad (4)$$

where

V_L is the volume of leachant used (l);

m_D is the dry mass of the test portion (kg);

ρ_{H_2O} is the density of water (usually taken as 1 kg/l);

w_{H_2O} is the water content for the test portion (%).

Place the capped bottle in an agitation device (6.3). Agitate for (24 ± 0,5) h.

As an alternative, instead of 24 h, 6 h can be adopted when it can be demonstrated that equilibrium or semi-equilibrium is reached or a quick turn-around time is required for quality control purposes. In this case, it shall be recorded that the leaching was carried out for 6 h.

Settling of solids in the bottle during agitation shall be avoided. At the end of the agitation period, the bottle is removed from the agitation device.

8.2.3 Liquid/Solid separation step

Allow the suspended solids to settle for (15 ± 5) min.

Transfer the supernatant to centrifuge tubes (6.8). The centrifugation containers shall be chosen so as not to adsorb (or release) analytes.

There are two options for centrifugation:

a) Centrifuge the eluate for 30 min at 20 000*g* to 30 000*g* using a high-speed centrifuge (6.8, see also Annex C).

b) Centrifuge the eluate for 5 h at 2 000*g* to 3 000*g* in glass bottles using a lower-speed centrifuge (6.8).

Cooling shall be applied to maintain the temperature at (22 ± 3) °C (see 8.1).

NOTE 1 Based on Stoke's law, the results of both centrifugation methods are expected to be comparable. Other alternative combinations of centrifugation acceleration and time can be applied given comparable conditions are calculated related to the specification of the rotor (see guidance in Annex C).

Gentle braking of the centrifuge shall be applied in order to avoid resuspension. The deceleration time shall not exceed 20 min.

NOTE 2 In case lightweight substances (e.g. coaly particles) are still floating after centrifugation, a glass fibre filtration (6.6) can be applied to remove such particles or to reduce the turbidity.

After centrifugation, the eluate shall be transferred immediately to an appropriate container for measurement of pH and redox potential (see also the next-to-last paragraph of 8.2.2) and stored for subsequent chemical analysis and/or ecotoxicological testing. In general this eluate can be used for both analyses of inorganic and organic substances.

If only inorganic substances are measured, the centrifugation step can be omitted, and the decanted eluate can be filtered directly using the appropriate membrane filters (6.5) and a vacuum or pressure filtration device (6.4), (see Annex B for an example). When this filtration as specified is not possible in less than 1 h with a liquid flow rate of at least 30 ml/cm^2/h, a liquid-solid separation procedure, specific

for the considered case, shall be applied. Report the details in the test report. This specific procedure shall not include the use of additives.

NOTE 3 For inorganic substances, it is often preferable to pre-centrifuge the eluate at 2 000 *g* to 3 000 *g* for 20 min before filtration using glass bottles with a screw cap and polytetrafluoroethylene inlay (or, if possible, using the leaching bottle directly) prior to filtration. Higher speed or longer time can also be applied (see Annex C).

NOTE 4 Such a specific liquid-solid separation procedure can include settling, pre-filtration on a coarser filter, centrifugation, filtration on a large-size membrane filter, filtration at high pressure, filtration at increasing high pressure following a first period without pressure, etc.

NOTE 5 An example of a specific liquid-solid separation procedure is given in Annex B and has been applied to soil samples.

Determine the volume of eluate, V_E or record the volume of the aliquot used.

Measure immediately electrical conductivity (in mS/m) and pH of the eluate. Measurement of turbidity, redox potential (E_h in mV) and DOC is highly recommended.

NOTE 6 Information on DOC concentration in the eluate is is relevant both for release of inorganic substances, as well as for organic substances.

Under certain circumstances, particularly for alkaline eluates, it is recommended to measure the pH of the raw eluate prior to filtration or centrifugation, since these operations may change the pH of the eluate.

8.3 Further preparation of the eluate for analysis

If necessary, divide the eluate into an appropriate number of sub-samples for different chemical analyses and store them in accordance with the requirements in ISO 5667-3.

Since eluates for bio-assays should not contain NaN_3 (see 8.2.1, Note 2), microbial degradation of organic substances may occur during the test and during the period of eluate storage. Therefore, it is highly recommended to perform bio-assays on eluates containing organic substances as soon as possible after completion of the leaching test.

8.4 Blank test for the application of the leaching procedure

Blank tests shall be carried out at regular intervals in order to check, as far as possible, how well the whole procedure is performed. A volume of leachant of 900 ml is submitted to the whole procedure, starting at 8.2.1 and using no soil sample.

The eluate of this blank test shall fulfil the following minimum requirements: in the eluate of the blank test, the concentration of each considered element shall be less than 20 % of the concentration determined in the eluate of the tested material or less than 20 % of the concentration in the eluate of a limit value to which the measurement result is to be compared. The elements to be considered are all the elements which are to be determined in the eluate of the tested material.

If the above requirements are not fulfilled, it is necessary to reduce the contamination. The blank test results shall not be deducted from the results of the material leaching test.

The above provision does not take into account the sieving step, crushing step or the splitting step. In order to minimize the possible contamination during these three steps, it is recommended to process a representative portion of the laboratory sample through the sieving device, the crushing device and through the splitting device and to discard such material thereafter. This provision does not cover the situation described in the note under 6.6.

9 Calculation

The concentrations of substances in the extraction solution are measured by appropriate analytical methods. They give concentrations in mg/l. The final result is a mass fraction, calculated on the basis of the extract solution volume and the mass of the test portion used, in mg/kg dry matter.

Calculate the quantity of a substance leached from the material, based on the dry mass of the original material, from Formula (5):

$$A = \rho_{\text{subst}} \times \left\{ \left(V_{\text{L}} / m_{\text{D}} \right) + \left[w_{\text{H}_2\text{O}} / \left(\rho_{\text{H}_2\text{O}} \times 100 \right) \right] \right\} \tag{5}$$

where

A is the release of a substance at a L/S = 10 (mg/kg of dry matter);

ρ_{subst} is the concentration of a particular substance in the eluate (mg/l);

V_{L} is the volume of leachant used (l);

$w_{\text{H}_2\text{O}}$ is the water content as calculated in Formula (2);

m_{D} is the mass of the dried test portion (kg);

$\rho_{\text{H}_2\text{O}}$ is the density of water (usually taken as 1 kg/l).

10 Test report

The test report shall include the following details:

a) a reference to this document (ISO 21268-2);

b) address of laboratory, name of responsible person;

c) any information necessary for the complete identification of the sample;

d) information on sample pretreatment;

e) water content;

f) type of leachant;

g) centrifugation speed/force, time and type of vessels used, temperature readings;

h) detailed description of the filtration step and results of adsorption tests on the filters applied if hydrophobic organic compounds are reported;

i) the test results including at least pH, electrical conductivity, measured concentrations (mg/l), released quantities (mg/kg dry matter), and limit of detection for each substance;

j) the blank test results;

k) any details that are optional or any deviations from the specifications of this document, and any effects which may have affected the results.

11 Analytical determination

11.1 General

Since the analysis step is not included in the scope of this document, the analytical method applied together with the limit of quantification shall be reported.

11.2 Blank test information

The following shall be included in the test report:

— date of the last blank test performed;

— results of the blank test, including the elements considered for the tested material and the levels above which the results can be considered as valid, when compared with the measured concentrations, in mg/l.

12 Performance characteristics

12.1 General

Data on robustness, repeatability and reproducibility of selected inorganic and organic substances are available from German research studies for the validation of DIN 19529[5] based on soils and soil-like materials (see Table 1 to Table 10). The test conditions are almost the same to this document except the L/S which was 2 l/kg and the eluent which was always demineralized water. It can be assumed that the concentrations at L/S 10 l/kg are mostly smaller in comparison to L/S 2 l/kg which may be then close to detection limits. It has to be noted that particularly for organic substances this may have an effect on the performance parameters of this procedure.

Membrane filtration at 0,45 µm was used for inorganic parameters and glass fibre filtration at 0,7 µm was used for organic compounds subsequently to the centrifugation.

The maximum grain size of soils used in the validation study was either <2 mm or partly <10 mm (no crushing was applied during sample preparation). The results can be adopted to this document taking into account the limitations described.

Within the validation of the batch leaching tests (EN 12457-1 to EN 12457-4) for waste, a contaminated soil was studied additionally (see References [4,17]). The tests comprise very similar test conditions to this document except demineralized water is used as leachant and there is no option for eluate preparation to analyse organic substances. The validation trial was based on interlaboratory comparisons using four different waste types and one contaminated soil among them.

12.2 Validation results obtained for DIN 19529

12.2.1 General

All validation results were obtained in accordance with the principles of ISO 5725-1[15] and ISO 5725-2[16].

Some of the values reported for the reproducibility, repeatability, and the number of outliers is relatively high (particularly at low concentrations) and reflects what can currently be achieved in testing laboratories. There are no specific criteria to determine whether these values are acceptable or not. The values for the reproducibility and repeatability can be used to derive uncertainties associated with testing results.

12.2.2 Results for test material containing inorganic substances

Table 1 — Characteristics of the test material (contaminated soil-like material, <10 mm)

Parameter	Dimension	Value
Humidity	% by weight	0,51
pH-value	—	8,51
Electrical conductivity	µS/cm	242,1
Carbonate content	% by weight	3,77
Loss on ignition	% by weight	1,14
Particle density	g/cm^3	2,652
Grain size distribution (dry sieving, no crushing):		
10 mm – 6,3 mm	% by weight	2,37
6,3 mm – 2 mm	% by weight	9,92
2 mm – 0,63 mm	% by weight	17,88
0,63 mm – 0,2 mm	% by weight	54,65
0,2 mm – 0,063 mm	% by weight	13,81
<0,063 mm	% by weight	1,38

Table 2 — Performance characteristics

	l	n	n_A	n_{AP} %	$\bar{x}$ µg/l	s_R µg/l	CV_R %	s_r µg/l	CV_r %
Cr	13	23	13	36,11	1,54	0,65	42,12	0,23	14,87
Cu	35	69	8	10,39	18,24	9,29	50,94	4,56	24,98
Pb	9	17	2	10,53	2,77	4,15	150,00	3,22	116,39
Zn	24	45	10	18,18	16,32	13,41	82,18	7,49	45,90
Ni	19	37	8	17,78	4,94	3,53	71,38	1,25	25,34

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

12.2.3 Results for test materials containing organic substances

12.2.3.1 Validation trial 1

(Based on 7 different PAH contaminated soils, sieved at 2 mm, no crushing of oversized material, leachant demineralized water.)

(Based on 7 different soils contaminated with polycyclic aromatic hydrocarbons (PAH), sieved at 2 mm, no crushing of oversized material, leachant demineralized water.)

Table 3 — Results for turbidity and conditions of centrifugation for validation trial 1

Parameter	Sample	Participant									
		1	2	3	4	5	6	7	11	13	14
Turbidity (FNU)	Soil 1	6,8	6,1	3,9	2,3	8,3	3,3	4,1	3,1	13	9,2
	Soil 2	5,1	4,8	1,0	2,5	<2,0	1,0	3,0	2,7	3,3	12,5
	Soil 3	3,9	2,7	< 0,3	0,7	<2,0	2,7	3,2	2,2	1,6	4,8
	Soil 4	1,2	—	0,3	0,3	<2,0	1,4	0,9	0,6	0,6	3,7
	Soil 5	1,4	—	1,0	1,5	7,4	1,7	2,2	1,2	4,8	2,0
	Soil 6	0,9	2,6	0,3	2,5	<2,0	0,5	1,3	0,3	1,2	0,3
	Soil 7	1,1	2,3	0,3	0,5	<2,0	0,5	1,2	0,4	2,7	0,3
Centrifugation speed (g)	Soil 1 to 7	20 000	2 000	20 000	17 000	22 000	8 500	20 000	20 000	20 000	2 000
Centrifugation duration (h)	Soil 1 to 7	0,5	5,0	0,5	0,25	0,5	1,0	0,5	0,5	0,5	5,0

Table 4 — Performance characteristics for PAH for validation trial 1

Parameter	Sample	l	n	n_A	n_{AP} %	$\bar{x}$ µg/l	s_R µg/l	CV_R %	S_r µg/l	CV_r µg/l
Σ PAH	Soil 1	13	32	0	0	244	128	52	31,9	13,1
Naphthalene		12	30	0	0	13,2	7,58	58	1,58	12,0
Phenanthrene		12	30	0	0	48,4	24,4	50	8,33	17,2
Pyrene		12	29	3	9	4,24	2,52	59	0,63	14,8
Σ PAH	Soil 2	12	29	3	9	101	50,3	50	10,1	10,0
Naphthalene		12	30	0	0	6,38	3,55	56	0,93	14,6
Phenanthrene		11	27	3	10	20,0	9,94	50	1,47	7,3
Pyrene		12	29	3	9	1,36	0,68	50	0,19	14,3
Σ PAH	Soil 3	13	32	0	0	144	78,9	55	15,2	10,6
Naphthalene		12	30	0	0	10,9	6,09	56	1,23	11,3
Phenanthrene		12	30	0	0	35,2	18,3	52	3,49	9,9
Pyrene		13	32	0	0	1,85	1,02	55	0,18	9,7
Σ PAH	Soil 4	11	26	3	10	86,8	55,4	64	11,0	12,7
Naphthalene		11	27	0	0	5,04	3,29	65	0,62	12,3
Phenanthrene		10	24	0	0	19,6	11,2	57	1,96	10,0
Pyrene		12	29	0	0	1,49	0,92	62	0,18	12,4

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 4 *(continued)*

Parameter	Sample	l	n	n_A	n_{AP} %	$\bar{x}$ µg/l	s_R µg/l	CV_R %	S_r µg/l	CV_r µg/l
Σ PAH	Soil 5	9	17	7	29	2,13	1,90	89	0,11	5,2
Naphthalene		7	14	1	7	0,215	0,17	79	0,11	53,0
Phenanthrene		9	17	7	29	0,643	0,88	137	0,034	5,3
Pyrene		9	18	2	10	0,115	0,07	59	0,019	16,5
Σ PAH	Soil 6	12	28	3	10	412	282	68	32,6	7,9
Naphthalene		11	26	0	0	11,4	7,24	64	1,54	13,5
Phenanthrene		12	29	0	0	83,3	56,0	67	11,4	13,6
Pyrene		13	30	0	0	5,77	3,73	65	0,53	9,2
Σ PAH	Soil 7	13	31	0	0	77,9	53,6	69	10,2	13,1
Naphthalene		10	23	0	0	1,31	0,78	60	0,24	18,5
Phenanthrene		12	27	0	0	24,3	14,0	57	1,45	6,0
Pyrene		13	31	0	0	1,56	0,93	59	0,16	10,0

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

12.2.3.2 Validation trial 2

The validation trial was based on two different reference soils which were mixed with contaminated soil-like material to four test materials.

Demineralized water was applied as leachant.

Table 5 — Characteristics of the reference soils used for preparation of test materials

Test material	Soil type	pH —	C_{org} weight%	CEC_{eff} $mmol_c/kg$
Soil TL	clayey loam	4,97	3,52	118
Soil MS	medium sand	8,48	0,64	8,3

both soils were sieved at 2 mm, no crushing of oversized material

Sample codes as listed in Tables 6 to 10:

— TL-PAH/PCB soil (clayey loam) contaminated with PAH and PCB, <2 mm;

— TL-PH/TPH/PAH soil (clayey loam) contaminated with phenols, TPH and PAH, <2 mm;

— MS-PAH/PCB soil (medium sand) contaminated with PAH and PCB, <2 mm;

— MS-PH/TPH/PAH soil (medium sand) contaminated with phenols, TPH and PAH, <2 mm.

Table 6 — Performance characteristics of basic parameters for validation trial 2

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
Turbidity	TL-PAH/PCB	FNU	12	22	2	8,3	3,9	2,9	73,4	0,3	8,0
	TL-PH/TPH/PAH	FNU	9	19		0	9,9	6,1	61,5	3,9	39,5
	MS-PAH/PCB	FNU	11	21	2	8,7	4,9	4,6	93,9	0,2	4,8
	MS-PH/TPH/PAH	FNU	8	17	3	15,0	2,0	1,9	93,6	0,8	41,8
DOC	TL-PAH/PCB	mg/l	11	22		0	52,7	21,9	41,5	2,0	3,9
	TL-PH/TPH/PAH	mg/l	7	15	2	11,8	451,5	48,1	10,6	14,7	3,3
	MS-PAH/PCB	mg/l	10	20	2	9,1	19,4	5,6	28,7	0,9	4,6
	MS-PH/TPH/PAK	mg/l	8	17		0	440,8	25,2	5,7	25,2	5,7
pH	TL-PAH/PCB	—	11	21		0	7,61	0,29	3,8	0,08	1,0
	TL-PH/TPH/PAH	—	9	19		0	7,09	0,24	3,4	0,14	2,0
	MS-PAH/PCB	—	10	21		0	8,09	0,35	4,3	0,17	2,1
	MS-PH/TPH/PAH	—	8	16	3	15,8	7,87	0,53	6,7	0,17	2,1

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 7 — Performance characteristics of PAH for validation trial 2

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
Naphthalene	TL-PAH/PCB	µg/l	12	22	3	12,0	3,7	2,6	70,7	0,4	10,0
	TL-PH/TPH/PAH	µg/l	12	24		0	271,6	126,2	46,5	24,0	8,8
	MS-PAH/PCB	µg/l	15	28		0	12,6	8,9	70,3	3,3	26,6
	MS-PH/TPH/PAH	µg/l	11	21	3	12,5	339,2	140,1	41,3	23,5	6,9
Acenaphthene	TL-PAH/PCB	µg/l	14	26	2	7,1	4,0	2,7	65,7	0,3	6,2
	TL-PH/TPH/PAH	µg/l	11	23	1	4,2	5,6	3,0	54,1	0,8	14,7
	MS-PAH/PCB	µg/l	16	29		0	27,2	16,8	61,7	4,2	15,5
	MS-PH/TPH/PAH	µg/l	11	22	2	8,3	10,3	5,8	56,4	0,6	6,2

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 7 *(continued)*

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
Fluorene	TL-PAH/PCB	µg/l	13	24	4	14,3	1,8	1,4	76,3	0,2	9,6
	TL-PH/TPH/PAH	µg/l	12	24		0	19,0	11,1	58,2	2,3	12,3
	MS-PAH/PCB	µg/l	14	26	2	7,1	11,8	9,9	83,8	0,9	7,3
	MS-PH/TPH/PAH	µg/l	12	24		0	15,3	16,5	107,9	2,5	16,6
Phenanthrene	TL-PAH/PCB	µg/l	11	19	5	20,8	1,3	1,4	106,4	0,1	9,0
	TL-PH/TPH/PAH	µg/l	12	23		0	25,5	13,1	51,3	2,5	10,0
	MS-PAH/PCB	µg/l	14	26	2	7,1	12,1	11,6	95,9	1,4	11,9
	MS-PH/TPH/PAH	µg/l	12	24		0	38,0	15,0	39,6	3,2	8,5
Anthracene	TL-PAH/PCB	µg/l	13	25	3	10,7	1,5	1,0	64,4	0,5	31,3
	TL-PH/TPH/PAH	µg/l	11	22	2	8,3	4,3	2,3	53,7	0,2	5,0
	MS-PAH/PCB	µg/l	11	21	6	22,2	12,3	9,8	80,1	0,5	3,9
	MS-PH/TPH/PAH	µg/l	12	24		0	10,1	4,4	43,1	0,9	8,5
Fluoranthene	TL-PAH/PCB	µg/l	14	26	2	7,1	0,7	0,3	48,6	0,1	8,3
	TL-PH/TPH/PAH	µg/l	11	23		0	4,0	3,3	81,9	2,9	71,5
	MS-PAH/PCB	µg/l	16	29		0	4,7	2,5	54,3	0,7	14,1
	MS-PH/TPH/PAH	µg/l	10	21	2	8,7	4,8	2,6	53,6	0,8	17,4
Pyrene	TL-PAH/PCB	µg/l	13	23	5	17,9	0,3	0,2	68,9	0,01	4,7
	TL-PH/TPH/PAH	µg/l	10	21	2	8,7	1,9	0,8	44,9	0,2	8,2
	MS-PAH/PCB	µg/l	14	27	1	3,6	2,2	1,0	46,0	0,4	19,4
	MS-PH/TPH/PAH	µg/l	10	20	2	9,1	2,9	1,2	43,2	0,3	11,6
Benzo(a) anthracene	TL-PAH/PCB	µg/l	5	8	5	38,5	0,02	0,01	39,1		
	TL-PH/TPH/PAH	µg/l	9	18	1	5,3	0,23	0,09	40,0	0,03	12,2
	MS-PAH/PCB	µg/l	12	22	2	8,3	0,21	0,27	131,6	0,02	11,2
	MS-PH/TPH/PAH	µg/l	8	17	3	15,0	0,30	0,10	34,2	0,06	21,5
Chrysene	TL-PAH/PCB	µg/l	6	11	3	21,4	0,02	0,01	50,0	0,01	25,0
	TL-PH/TPH/PAH	µg/l	10	19		0	0,19	0,08	41,4	0,03	18,3
	MS-PAH/PCB	µg/l	10	20	3	13,0	0,01	0,04	42,9	0,02	21,4
	MS-PH/TPH/PAH	µg/l	9	18	3	14,3	0,23	0,08	36,0	0,06	24,9
Benzo(b) fluoranthene	TL-PAH/PCB	µg/l	2	4	5	55,6	0,01				
	TL-PH/TPH/PAH	µg/l	7	14	3	17,6	0,07	0,02	21,7	0,01	10,1
	MS-PAH/PCB	µg/l	7	14	3	17,6	0,04	0,01	35,1	0,01	24,3
	MS-PH/TPH/PAH	µg/l	5	11	3	21,4	0,08	0,04	44,9	0,03	37,2

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 7 *(continued)*

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
Fluoranthene	TL-PAH/PCB	µg/l	14	26	2	7,1	0,7	0,3	48,6	0,1	8,3
	TL-PH/TPH/PAH	µg/l	11	23		0	4,0	3,3	81,9	2,9	71,5
	MS-PAH/PCB	µg/l	16	29		0	4,7	2,5	54,3	0,7	14,1
	MS-PH/TPH/PAH	µg/l	10	21	2	8,7	4,8	2,6	53,6	0,8	17,4
Benzo(k) fluoranthene	TL-PAH/PCB	µg/l	3	5	4	44,4	0,01	0,02	128,6		
	TL-PH/TPH/PAH	µg/l	8	16		0	0,06	0,05	85,2	0,02	32,8
	MS-PAH/PCB	µg/l	7	14	3	17,6	0,02	0,01	36,8	0,004	21,1
	MS-PH/TPH/PAH	µg/l	6	13		0	0,05	0,04	73,1	0,04	67,3
Benzo(a) pyrene	TL-PAH/PCB	µg/l	4	7	9	56,3	0,03	0,04	116,7	0,01	20,0
	TL-PH/TPH/PAH	µg/l	8	14	2	12,5	0,09	0,06	64,7	0,01	11,8
	MS-PAH/PCB	µg/l	7	14	3	17,6	0,03	0,02	57,1	0,01	21,4
	MS-PH/TPH/PAH	µg/l	7	14		0	0,11	0,11	101,8	0,06	56,9
Benzo(g,h,i) perylene	TL-PAH/PCB	µg/l	5	9		0	0,02	0,03	137,5	0,01	45,8
	TL-PH/TPH/PAH	µg/l	5	9	2	18,2	0,03	0,02	48,4	0,01	16,1
	MS-PAH/PCB	µg/l	6	11	3	21,4	0,02	0,01	41,2	0,004	23,5
	MS-PH/TPH/PAH	µg/l	4	8		0	0,10	0,12	117,3	0,07	62,5
Dibenzo(a,h) anthracene	TL-PAH/PCB	µg/l	2	4	5	55,6					
	TL-PH/TPH/PAH	µg/l	4	6		0	0,02	0,01	55,6	0,01	55,6
	MS-PAH/PCB	µg/l	2	4	5	55,6					
	MS-PH/TPH/PAH	µg/l	4	6		0	0,04	0,04	105,4	0,02	54,1
Inde-no(1,2,3-cd) pyrene	TL-PAH/PCB	µg/l	3	5	4	44,4	0,01	0,02	164,3		
	TL-PH/TPH/PAH	µg/l	6	10	3	23,1	0,05	0,01	29,8	0,01	10,6
	MS-PAH/PCB	µg/l	3	5	6	54,5	0,04	0,08	192,9		
	MS-PH/TPH/PAH	µg/l	7	12		0	0,15	0,13	91,2	0,06	40,8
sum 15 PAH	TL-PAH/PCB	µg/l	13	25	2	7,4	13,5	8,3	61,8	2,6	19,4
	TL-PH/TPH/PAH	µg/l	11	22		0	385,1	227,2	59,0	27,3	7,1
	MS-PAH/PCB	µg/l	13	25	2	7,4	88,3	50,9	57,6	5,2	5,8
	MS-PH/TPH/PAH	µg/l	10	19	3	13,6	468,5	199,3	42,5	11,7	2,5

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 8 — Performance characteristics of PCB for validation trial 2

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
PCB 28 + 31	TL-PAH/PCB	µg/l	13	26	1	3,7	0,025	0,014	56,0	0,005	20,0
	MS-PAH/PCB	µg/l	14	26		0	0,097	0,074	76,3	0,013	13,4
PCB 52	TL-PAH/PCB	µg/l	13	25	2	7,4	0,206	0,107	51,9	0,022	10,7
	MS-PAH/PCB	µg/l	14	26		0	0,764	0,396	51,8	0,105	13,7
PCB 101	TL-PAH/PCB	µg/l	12	24	3	11,1	0,118	0,040	33,9	0,021	17,8
	MS-PAH/PCB	µg/l	13	25	1	3,8	0,316	0,131	41,5	0,060	19,0
PCB 153	TL-PAH/PCB	µg/l	11	22	5	18,5	0,045	0,015	33,3	0,004	8,9
	MS-PAH/PCB	µg/l	12	23	3	11,5	0,095	0,051	53,7	0,018	18,9
PCB 138	TL-PAH/PCB	µg/l	11	22	5	18,5	0,058	0,022	37,9	0,006	10,3
	MS-PAH/PCB	µg/l	12	23	3	11,5	0,122	0,064	52,5	0,017	13,9
PCB 180	TL-PAH/PCB	µg/l	12	23	2	8,0	0,150	0,006	4,0	0,003	2,0
	MS-PAH/PCB	µg/l	12	21	3	12,5	0,027	0,013	48,1	0,004	14,8
sum PCB	TL-PAH/PCB	µg/l	12	24	2	7,7	0,460	0,158	34,3	0,052	11,3
	MS-PAH/PCB	µg/l	12	23	2	8,0	1,569	0,770	49,1	0,108	6,9

l = number of labs after outlier elimination;
n = number of single results after outlier elimination;
n_A = number of outliers;
n_{AP} = percentage of outliers;
$\bar{x}$ = mean value;
s_R = reproducibility standard deviation;
CV_R = reproducibility coefficient [%];
s_r = repeatability standard deviation;
CV_r = repeatability coefficient [%].

Table 9 — Performance characteristics of PCB and TPH for validation trial 2

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
TPH C_{10}-C_{22}	TL-PH/TPH/PAH	µg/l	8	16	2	11,1	411,9	121,2	29,4	77,0	18,7
	MS-PH/TPH/PAH	µg/l	10	21	0	0	589,8	353,5	59,9	121,5	20,6
TPH C_{10}-C_{40}	TL-PH/TPH/PAH	µg/l	10	19	0	0	602,2	338,6	56,2	64,7	10,7
	MS-PH/TPH/PAH	µg/l	10	21	0	0	704,9	454,1	64,4	118,9	16,9

l = number of labs after outlier elimination;
n = number of single results after outlier elimination;
n_A = number of outliers;
n_{AP} = percentage of outliers;
$\bar{x}$ = mean value;
s_R = reproducibility standard deviation;
CV_R = reproducibility coefficient [%];
s_r = repeatability standard deviation;
CV_r = repeatability coefficient [%].

Table 10 — Performance characteristics of phenols for validation trial 2

Parameter	Soil	Unit	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
phenol	TL-PH/TPH/PAH	µg/l	7	14	0	0	709	323	45,6	170	24,0
	MS-PH/TPH/PAH	µg/l	7	15	0	0	995	982	98,7	371	37,3
2-methyl phenol (o-cresol)	TL-PH/TPH/PAH	µg/l	7	14	0	0	14,5	7,4	51,4	3,8	26,1
	MS-PH/TPH/PAH	µg/l	7	15	0	0	1283	1052	82,0	497	38,7
3-methyl phenol (m-cresol)	TL-PH/TPH/PAH	µg/l	6	11	3	21,4	933	330	35,4	80,9	8,7
	MS-PH/TPH/PAH	µg/l	7	15	0	0	1876	1275	68,0	666	35,5
4-methyl phenol (p-cresol)	TL-PH/TPH/PAH	µg/l	5	9	2	18,2	6,6	1,9	29,2	1,5	22,7
	MS-PH/TPH/PAH	µg/l	6	13	0	0	1366	705	51,6	487	35,6
2,6-dimethyl phenol	TL-PH/TPH/PAH	µg/l	5	9	0	0	1,7	1,5	90,3	1,2	72,6
	MS-PH/TPH/PAH	µg/l	3	6	7	53,8	4,5	1,8	41,0	0,7	15,5
3,4-dimethyl phenol	TL-PH/TPH/PAH	µg/l	4	7	3	30,0	4,4	1,6	35,2	0,4	8,0
	MS-PH/TPH/PAH	µg/l	6	13	0	0	507	209	41,2	140	27,7
sum phenols	TL-PH/TPH/PAH	µg/l	6	12	0	0	2055	1148	55,9	949	46,2
	MS-PH/TPH/PAH	µg/l	7	15	0	0	5966	4216	70,7	2221	37,2

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Annex A
(informative)

Information on the influence on the test results of the parameters that affect leaching

A.1 Overview

In this annex, information is provided on the possible sources of variability. It mostly addresses leaching of inorganic substances, but some specific problems associated with the application of this document to the leaching of organic substances are briefly addressed in A.3.5.

A.2 General aspects

The leaching of substances from soil and soil-like material is controlled by several parameters and external factors. These factors include the chemical nature of the material, especially in terms of pH, reducing properties and degradable-organic-matter content, the nature of the leachant, the contact time of the leachant with the material and whether leaching of substances is controlled by solubility or by diffusion. Furthermore, the chemical, physical and geotechnical nature of the environment to which the material is exposed are important. The influence and importance of these factors should be examined in the basic characterization tests in order that the leaching behaviour of the material is better understood. In EN 12920[4], the steps required to achieve such a determination are specified for waste. A similar approach can be followed for soil and soil-like material. This generally requires several tests to be performed, the use or establishment of a behavioural model and the validation of the model.

It is to be noted that, in this compliance test, the final conditions of the test are imposed by the material itself. The key factors in this test are briefly addressed in A.3.

A.3 Factors influencing leaching

A.3.1 Influence of contact time

The compliance test is based on the assumption that equilibrium or semi-equilibrium is reached under test conditions. The contact time required to reach the state of equilibrium or semi-equilibrium depends on the combination of the soil type and the substances to be investigated. There are several factors that can affect the leaching amount, as reactions such as dissolution-precipitation, adsorption-desorption, cation exchange, microbial activity, etc. can be active simultaneously during the leaching process. The particle size of soil and soil type, such as marine soil, volcanic soil, organic soil, etc., are important factors that determine how fast equilibrium or semi-equilibrium is reached.

In spite of experimental work to determine the appropriate length of agitation time required to reach equilibrium or semi-equilibrium conditions, the information has not been conclusive, as for some substances stable (equilibrium) conditions are reached, whereas for others this condition is not met. Within 24 h, a stable condition is considered to be sufficiently approached for many parameters from a variety of materials.

For soils which have been exposed to leaching in the field, equilibrium conditions can often be expected, which explains why, for some substances, 80 % to 100 % of concentrations obtained after 24 h agitation were already obtained after 6 h of agitation.

This implies that, in specific cases, 6 h can be sufficient (to be demonstrated). For quality control purposes, a short contact time can be necessary in view of the turn-around time, and results for the shorter contact time can be sufficiently close to justify its use.

A.3.2 Influence of the liquid to solid ratio (L/S)

In the two standards worked out in parallel (ISO 21268-1 and -2), different L/S are specified (10 and 2), leading generally to different test results. This is caused, on the one hand, by different quantities of leachant being put into contact with the same quantity of material and, on the other hand, by different leaching conditions dictated by the material itself (as a result of the compounds of the material dissolved into the leachate). It is to be noted that there is no relation available that could be applied to the results obtained with a given L/S to determine the results, which would have been obtained if the test had been performed at another L/S.

At lower L/S, some substances are present in the leachate at a higher concentration, as a result of the lower quantity of the available leachant.

At L/S = 2, the test is not applicable to different categories of material which have an inherent water content before the test (such as sediments). At L/S = 10, such limitations appear only in a few cases.

A.3.3 Influence of pH

In this compliance test, the final conditions of the test are imposed by the material itself. This is generally the case for pH. The sensitivity of leaching to relatively small changes in pH can be significant. Such sensitivity can induce varying results. Also, exposure to atmospheric CO_2 or O_2, increased CO_2 levels in the laboratory during sample storage, handling, performance of the leaching test and analysis can affect the test results, as they can lead to pH/redox changes in the eluate.

A.3.4 Influence of reducing properties

Materials to be tested can exhibit reducing properties, which is evident from a low redox potential in the leachate. For the proper evaluation of material, it is important to be aware of this aspect as different degrees of oxidation in sample handling and storage can induce varying results.

A.3.5 Factors influencing the leaching of organic substances

The leachability of organic substances is governed by processes that differ considerably from those for inorganic substances. In addition, the properties in relation to sorption on different materials with which they come in contact (e.g. bottles, filters) are different for organic substances and for inorganic substances.

Within the category of organic substances, a significant difference in behaviour exists between the more polar, relatively water-soluble compounds and apolar, hydrophobic organic substances. In the latter case, mechanisms of release (e.g. particle-bound or dissolved organic carbon-bound) can be more crucial. The test and the results should be used for leaching of organic substances only with thorough consideration of the specific properties of the substances in question and the associated potential problems.

A.3.6 Special requirements for tests considering semi-volatile substances

For the preparation of samples containing semi-volatile substances, size reduction has to be avoided (see 7.2). Preferably, cooling measures should be taken into account if particle size reduction is necessary in that case e.g. due to the presence of anthropogenic material in the sample.

A.4 Analytical versus leaching test errors

Since poor repeatability can be attributed to measurements close to the detection limit, it is recommended to apply analytical methods with sufficient sensitivity. In some cases, poor repeatability can be attributed to an extreme sensitivity to relatively small changes in the final pH in the extract (see A.3.3).