TECHNICAL SPECIFICATION

ISO/TS 21268-1

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

Part 1:
Batch test using a liquid to solid ratio of 2 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 1: Essai en bâchée avec un rapport liquide/solide de 2 l/kg de matière sèche

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Contents

Page

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

ISO/TS 21268-1 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

ISO/TS 21268 consists of the following parts, under the general title *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials*:

— *Part 1: Batch test using a liquid to solid ratio of 2 l/kg dry matter*

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

— *Part 3: Up-flow percolation test*

— *Part 4: Influence of pH on leaching with initial acid/base addition*

Introduction

In various countries, tests have been developed to characterise and assess the constituents which can be released from materials. The release of soluble constituents 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.

Not all of the relevant aspects of leaching behaviour can be addressed in one standard.

Tests to characterise the behaviour of materials can generally be divided into three categories (EN 12920; EN 12457-2) and are addressed in ISO 18772[9]. The relationships between these tests are summarised below.

a) “Basic characterisation” tests are used to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials. Liquid/solid (L/S) ratios, 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 characterisation 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.

NOTE Up to now, the test procedures described in this part of ISO/TS 21268 have not been validated.

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

Part 1:
Batch test using a liquid to solid ratio of 2 l/kg dry matter

1 Scope

This part of ISO/TS 21268 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 2 l/kg dry matter. It applies to soil and soil material with a particle size less than or equal to 4 mm.

This part of ISO/TS 21268 has been developed to measure the release of inorganic and organic constituents from soil and soil material and the ecotoxicological effects of eluates with respect to micro-organisms, fauna and flora. The test is not suitable for constituents that are volatile under ambient conditions. For ecotoxicological testing, see ISO 15799.

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

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

NOTE 3 For ecotoxicological testing, eluates representing the release of both inorganic and organic contaminants are needed. In this document, ecotoxicological testing is also meant to include genotoxicological testing.

The test procedure specified in this part of ISO/TS 21268 produces eluates, which are subsequently characterised by existing physical, chemical and ecotoxicological standard methods.

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 part of ISO/TS 21268 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 referenced documents are indispensable for the application 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: Guidance on the preservation and handling of water samples*

ISO 7027, *Water quality — Determination of turbidity*

ISO 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 10381-3, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 10381-4, *Soil quality — Sampling — Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*

ISO 10381-5, *Soil quality — Sampling — Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*

ISO 10381-6, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil under aerobic conditions for the assessment of microbiological processes, biomass and diversity in the laboratory*

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.

3.1
leaching test
test during which a material is put into contact with a leachant under strictly defined conditions and some constituents of the material are extracted

3.2
leachant
liquid used in a leaching test

NOTE For the purposes of this part of ISO/TS 21268, the leachant is water as specified in 5.1.

3.3
eluate
solution recovered from a leaching test

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 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 The basis for the calculation of the moisture content is the mass of the dry residue in this part of ISO/TS 21268, 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, from which test portions 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

NOTE 1 The test portion can be taken from the laboratory sample directly if no pre-treatment of the sample is required, but usually it is taken from the test sample.

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

3.10
soil material

excavated soil, dredged materials, manufactured soils, treated soils and fill materials

[ISO 15176:2002, definition 3.1.4]

4 Principle

The test portion, which originally or after suitable pre-treatment has a particle size less than or equal to 4 mm, is brought into contact with water containing a low concentration (0,001 M) of calcium chloride 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 constituents. The properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates, and/or the eluate may be subjected to subsequent ecotoxicological testing.

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

NOTE 1 These parameters often control the leaching behaviour of soil materials and are therefore important for evaluation of the test results.

NOTE 2 The leachant is 0,001 M $\mathrm{CaCl_2}$ to minimize the mobilisation of DOC caused by an ionic strength of the leachant which is too low.

The procedure described in this part of ISO/TS 21268 is based on the more stringent test requirements for determining the release of organic constituents and for subsequent ecotoxicological testing. If only the release of inorganic constituents is to be measured, less stringent requirements may be adapted for some steps of the procedure.

5 Reagents

5.1 Demineralised water or **deionised water** or **water of equivalent purity** ($5 < \text{pH} < 7,5$) with a conductivity $< 0,5$ mS/m in accordance with grade 3 specified in ISO 3696, made to 0,001 M $CaCl_2$. For eluates that are not to be used for ecotoxicological testing, sodium azide (NaN_3) shall be added to a resulting concentration of 0,1 % in order to prevent microbial degradation of organic contaminants.

NOTE 1 Microbial degradation of organic contaminants may occur in eluate without NaN_3.

NOTE 2 If only inorganic compounds are measured, the addition of NaN_3 is not required.

5.2 Rinsing solutions: nitric acid 0,1 mol/l (analytical grade) and/or organic solvent (acetone).

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, can be used, except for unpreserved samples for mercury analysis.

The volume of 1 l is selected in combination with the mass, m, of 350 g as specified in 7.4 in order to minimise head-space at a L/S ratio of 2 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 should be reported.

NOTE 2 Glass of high quality is considered adequate for both metals and organic contaminants, particularly, since the pH range usually covered in soil testing does not reach the conditions ($\text{pH} > 10$ and $\text{pH} < 3$) where glass itself may be partially dissolved.

NOTE 3 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 with a nominal volume of 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 components are to be analysed, vacuum filtration shall not be used.

6.5 0,45 µm membrane filters, pre-rinsed or similarly clean, for filtration [e.g. rinsed with 0,1 mol/l HNO_3 (5.2) and water (5.1)].

The filter should be chosen so as not to adsorb (or release) compounds of interest. This could be tested in preliminary experiments.

6.6 Sieving equipment with sieves of 4 mm nominal screen size.

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

6.7 Centrifuge operating at 20 000*g* to 30 000*g* using centrifuge tubes of 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.

Alternatively, if a high-speed centrifuge is not available, a centrifuge operating at 2 000*g* to 2 500*g* using glass bottles may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

6.8 Device for measuring electrical conductivity.

6.9 pH meter in accordance with ISO 10523.

6.10 Two thermometers for air and leachant temperature measurement.

6.11 Redox potential meter (optional).

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

6.13 Measuring cylinders for volume determination with 1 % accuracy.

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

6.15 Turbidity meter as specified in ISO 7027.

6.16 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 constituents of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment.

7 Sample pre-treatment

7.1 Sample size

Obtain a representative laboratory sample of at least 2 kg (dry matter) of the material. Use a sample splitter (6.14) or apply coning and quartering to split the sample.

Sampling shall be performed in accordance with the guide to the preparation of a sampling plan for soil materials, as specified in ISO 10381-1 to ISO 10381-6, in order to obtain representative laboratory samples.

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).

NOTE 2 Alternatively, larger volumes of eluate may also be produced in a single test, provided that the ratios in terms of L/S and minimum headspace are maintained.

NOTE 3 The volume of eluate required depends on the specific purpose and the subsequent chemical analysis and/or ecotoxicological tests to be carried out on the eluate. Analysis for ingorganic components may typically require from 20 ml to 500 ml of eluate, analysis for organic components from 250 ml to 2 000 ml, depending on the number and type of groups of organic components to be analysed (DOC from 100 ml to 250 ml), and ecotoxicological testing from 100 ml to 2 000 ml.

NOTE 4 The required size of the laboratory sample is dependent on the particle size distribution of the soil to be analysed (see ISO 11277). The specified sample size will generally be adequate. In specific cases, a smaller sample size 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.

Any deviation(s) to accommodate sample size or volume requirements shall be recorded in the test report.

7.2 Particle size reduction

The tests shall be carried out preferably on material as received. However, the test portion to be prepared shall have a grain size less than or equal to 4 mm of at least 95 % (mass fraction). If oversized material is not of natural origin and exceeds 5 % (mass fraction), the entire oversized fraction shall be separated by sieving (see 6.6) and crushed with suitable crushing equipment (6.16). On no account shall the material be finely ground. Oversized material of natural origin (e.g. stones, pebbles, twigs) in the sample shall be separated and discarded. Irrespective of any necessary size reduction, the separate fractions, with the exception of non-crushable and discarded material, shall be mixed to constitute the test sample. If the laboratory sample cannot be crushed or sieved because of its water content, it is allowed, in this case only, to dry the laboratory sample. The drying temperature shall not exceed 25 °C.

NOTE Sieving and drying at 25 °C, as well as crushing, can lead to a loss of semi-volatile components (inorganic and organic). In this case, particle size reduction and drying is best avoided, as it may alter the leaching characteristics.

7.3 Determination of dry matter content and water content

The whole test sample, complying with the size criterion in 7.2, shall not be further dried. The water content of the test sample shall be determined on a separate test portion at (105 ± 5) °C. This shall be taken into account when adjusting the L/S ratio. 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 Equation (1).

$$w_{\mathrm{dm}} = 100 \times m_{\mathrm{D}} / m_{\mathrm{W}} \tag{1}$$

where

w_{dm} is the dry matter content (%);

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

m_{W} is the mass of the undried sample (kg).

The water content ($w_{\mathrm{H_2O}}$ in %) is calculated as follows:

$$w_{\mathrm{H_2O}} = 100 \times \left(m_{\mathrm{W}} - m_{\mathrm{D}} \right) / m_{\mathrm{D}} \tag{2}$$

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 the soil sample is air-dried prior to testing, the dry matter content $w_{\mathrm{dm,AD}}$ of the air-dried sample shall be determined as well.

7.4 Preparation of test portion

Prepare, from the test sample, a test portion with a total mass m containing (350 ± 5) g (measured with an accuracy of 0,1 g) of dry mass (m_{D}). Use a sample splitter (6.14) or apply coning and quartering to split the sample.

$$m = 100 \times m_{\mathrm{D}} / w_{\mathrm{dm}} \tag{3}$$

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

In view of the minimum requirements of sample volume for analytical purposes, it may be necessary to use a larger test portion size and a correspondingly larger volume of leachant. This deviation from this part of ISO/TS 21268 shall be specified in the test report.

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

8 Procedure

8.1 Testing conditions

The compliance test for leaching shall be carried out at room temperature: (20 ± 5) °C.

8.2 Description of the procedure

8.2.1 Leaching step

Place the test portion with the total mass m corresponding to (350 ± 5) g of dry mass m_{D} in a bottle (6.1).

Using a measuring cylinder (6.13), add an amount of leachant (V_{L}) to establish a liquid to solid (L/S) ratio of (2 ± 0,04) l/kg during the extraction. Care shall be taken to obtain good mixing of solid and liquid.

$$V_{\mathrm{L}} = \left[10 - w_{\mathrm{H_2O}} \Big/ \left(\rho_{\mathrm{H_2O}} \times 100\right)\right] \times m_{\mathrm{D}} \tag{4}$$

where

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

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

$\rho_{\mathrm{H_2O}}$ is the density of water (usually taken as 1 kg/l);

$w_{\mathrm{H_2O}}$ 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, for quality control purposes, a quick turn-around time is required. In this case, it should 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.

8.2.2 Liquid-solid separation step

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

NOTE At this point, it is considered necessary to carry out the centrifugation step to ensure repeatable and reproducible results. When the validation studies show that the centrifugation is less critical, then the procedure can be relaxed, for instance, by including a turbidity measurement, based on which it may be decided to continue with centrifugation or that this step can be omitted.

Transfer the supernatant to centrifuge tubes (6.7).

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 (6.7). The centrifugation container should be chosen so as not to adsorb (or release) analytes.

b) Centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower-speed centrifuge (6.7).

Cooling shall be applied to maintain the temperature at (20 ± 5) °C (see 8.1).

NOTE 1 Based on Stoke's law, the results of both centrifugation methods are expected to be comparable.

Gentle breaking shall be applied in order to avoid resuspension. The deceleration time shall not exceed 20 min.

After centrifugation, the eluate shall be transferred immediately to an appropriate container for measurement of pH, redox potential (see also the next-to-last paragraph of 8.2.2) and stored for subsequent chemical analysis and/or ecotoxicological testing.

If only inorganic constituents are measured, the eluate can be filtered using the appropriate membrane filters (6.5) and a vacuum or pressure filtration device (6.4). When 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 2 For inorganic constituents, it is often preferable to pre-centrifuge the eluate at 2 000 g to 3 000 g for 20 min using glass bottles with a screw cap and polytetrafluoroethylene inlay (or, if possible, using the leaching bottle directly) prior to filtration.

NOTE 3 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 4 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.

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

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.

Analysis of DOC in the eluate is highly recommended, as this property is relevant both for release of inorganic substances, as well as for organic substances.

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. If the eluates are to be used for bio-assays, they shall be processed as soon as possible, but at least within 7 d storage at a maximum of 4 °C.

Since eluate for bio-assays should not contain NaN_3 (see 5.1), microbial degradation of organic contaminants may occur during the test and during the period of eluate storage. Therefore, it is highly recommended to perform bio-assays on eluate containing organic contaminants 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 or the splitting step. In order to minimise the possible contamination during these three steps, it is recommended to process a representative portion of the laboratory sample through the sieving device, the fragmentation 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 constituents 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 constituent leached from the material, based on the dry mass of the original material, from Equation (5):

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

where

A is the release of a constituent at a L/S = 2 (mg/kg of dry matter);

ρ_{consti} is the concentration of a particular constituent 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 Equation (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 refer to this part of ISO/TS 21268 and include the following details:

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

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

c) the test results (e.g. chemical analysis);

d) any details that are optional or any deviations from the specifications of this part of ISO/TS 21268, 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 part of ISO/TS 21268, the analytical method specified in the part of ISO/TS 21268 dedicated to such analysis shall be incorporated, including the date of analysis, the storage conditions until the analysis is performed, the quantification limit of the analytical method being used, the address of the laboratory and name(s) of operator(s).

11.2 Calculations and blank test information

The following shall be included:

- results of the calculation of the leached quantities, in mg/kg of dry mass, with references to the relevant subclauses of this part of ISO/TS 21268;
- 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

When this part of ISO/TS 21268 was adopted by ISO, the test specified in this part of ISO/TS 21268 was not validated and no data were available on robustness, repeatability and reproducibility.

In the validation of the batch leaching test (EN 12457-1 to EN 12457-4) for waste, contaminated soil was studied ([2] in the Bibliography).

Annex A
(informative)

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

A.1 Introduction

Since this part of ISO/TS 21268 has not yet been validated, in particular with respect to its robustness, the sensitivity to variations in the parameters specified in this procedure and to external factors cannot be quantified. In this annex, information is provided on the possible sources of variability. It mostly addresses leaching of inorganic constituents, but some specific problems associated with the application of this part of ISO/TS 21268 to the leaching of organic constituents are briefly addressed in A.3.5.

A.2 General aspects

The leaching of contaminants from soil 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 contaminant leaching 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 characterisation tests in order that the leaching behaviour of the material is better understood. In EN 12920, the steps required to achieve such a determination are specified for waste. A similar approach can be followed for soil 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 in equilibrium or semi-equilibrium depends on the combination of a soil type and the species 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 constituents stable (equilibrium) conditions are reached, whereas for others this condition is not met. In 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 constituents, 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, 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 species 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 a 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 Influence of the leaching of organic contaminants

The leaching of organic contaminants from material is an area that is still not well addressed. Traditionally, leaching tests have been focused primarily on inorganic constituents and have been applied to leaching of organic contaminants without further evaluation of the suitability of the methods. The leachability of organic contaminants is governed by processes that differ considerably from those for inorganic contaminants. In addition, the properties of organic contaminants in relation to sorption on different materials with which they come in contact (e.g. bottles, filters) are different for organic contaminants and for inorganic contaminants.

Within the category of organic contaminants, a significant difference in behaviour exists between the more polar, relatively water-soluble compounds and apolar, hydrophobic organic contaminants. 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 constituents only with thorough consideration of the specific properties of the constituents in question and the associated potential problems.

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).

A.5 Evaluation of test results

The test results obtained with the compliance tests specified in this part of ISO/TS 21268 only allow a direct comparison with regulatory limits on a pass/fail basis. A comprehensive evaluation of the leaching behaviour requires a basis or framework of reference such as that provided by ISO 18772.

Annex B
(informative)

Example of a specific liquid-solid separation procedure for soil samples (applying only to the leaching of inorganic constituents)

B.1 Introduction

The original scope of leaching tests covers, in particular, solid substances containing larger amounts of dissolved salts. The general feasibility of these methods has limitations when the solubility of substances has to be determined in soil samples, in particular when, for example, oxidised, adsorbed or organically bound heavy metals have to be rather insoluble in those materials. Despite their low solubility, heavy metals are important from an environmental point of view. The lower the “pure” solubility of heavy metals in a contaminated soil sample, the bigger is the relative influence of colloidal particle portions in eluate on the end result.

Especially in the case of fine-textured soil samples that are rich in humus but poor in electrolytes, the filter cake produced during filtration exhibits very fine pores and less colloids pass through the membrane filter. Thus, the production of filter cake largely affects the “solubility” of heavy metals, a fact which is identified by this method. To obtain comparable results, it is necessary to stipulate the factors determining the height of the filter cake. In addition to sample-specific properties, the thickness of the filter cake is determined predominantly by the filter diameter and the volume of the eluate to be filtered. Absorption by the filter cake can be reduced when part of the extract solution is filtered.

B.2 Procedure

B.2.1 Equipment

B.2.1.1 **Pressure filtration unit for membrane filter** (142 mm diameter).

B.2.1.2 **Membrane filter** of pore size 0,45 µm.

If another filter size is used, the volume to be filtered is modified according to the filter surface; an essential precondition is that the relationship between the volume to be filtered and the filter surface is complied with [relationship: about 1 l volume to 158 cm^2 filter surface (diameter 142 mm)].

B.2.2 Material in contact with extracts

Use a media-guiding apparatus in polytetrafluoroethylene.

B.2.3 Procedural steps

For sedimentation of the larger particles, allow the suspension to stand for 15 min after shaking.

Decant almost completely the supernatant liquid into a centrifuge tube or bottle device.

Apply centrifugation (30 min at 2 000g).

Almost completely decant the supernatant liquid into the membrane pressure filter apparatus (B.2.1.1).

Apply, after 5 min of filtration without pressure, a pressure of 1 kPa to accelerate filtration. If after 15 min less than two-thirds of the eluate have passed through the filter, the pressure is increased to 2 kPa. If necessary, the pressure is increased to a maximum of 3,5 kPa after 30 min. Filtration proceeds until all the supernatant of centrifugation has passed through the filter. If the filtration is still incomplete after 2 h, it is stopped and worked on with incomplete filtrate.

By using this procedure, a significant reduction in the possible errors resulting from the proportions of colloidal, dissolved heavy metals in filtrate can be ensured. Subsequently, it is not allowed to decant the first part of the filtrate and to put it again on the filter, a method that is quite common in several laboratories.

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