TECHNICAL SPECIFICATION

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Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil 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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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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.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 21268-2 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 2: Batch test using a liquid to solid ratio of 10 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 10 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 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 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 microbialogical 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 purpose 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_{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 $CaCl_2$ 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** ($5 < pH < 7{,}5$) 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 eluates 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 90 g as specified in 7.4 in order to minimise head-space at a L/S ratio 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 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 ($pH > 10$ and $pH < 3$) 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 of less than or equal to 4 mm 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