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

Part 1:

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

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

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*Partie 1: Essai en bûchée avec un rapport liquide/solide de 2 l/kg de
matière sèche*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 21268-1:2019 cancels and replaces the first edition (ISO/TS 21268-1:2007), 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 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.

This document was originally elaborated on the basis of EN 12457-1:2004^[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].

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

Part 1:

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

1 Scope

This document specifies a test providing information on leaching of soil and soil-like materials under the experimental conditions specified hereafter, and particularly at a liquid to solid ratio of 2 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 optimise 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_{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

4 Principle**iTeh STANDARD PREVIEW**

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 demineralised 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-like materials for many inorganic and organic substances.

NOTE 2 The leachant is 0,001 mol/l 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 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 Demineralised water or deionised water or water of equivalent purity ($5 < \text{pH} < 7,5$) with a conductivity of $< 0,5 \text{ mS/m}$ in accordance with grade 3 specified in ISO 3696 made to **0,001 mol/l CaCl_2** .

5.2 Calcium chloride ($\text{CaCl}_2 \cdot 2 \text{H}_2\text{O}$), analytical grade.

5.3 Sodium azide (NaN_3), analytical grade.

5.4 **Nitric acid (HNO₃)**, 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 350 g as specified in 7.4 in order to minimize headspace in the bottle at an L/S 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 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.

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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 HNO₃ (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 plasticisers from plastic sieves.

6.8 Centrifuge, operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of PFA (perfluoroalkoxy alkane), FEP (fluorinated ethylene propylene) 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 $\pm 0,05$ pH units.

6.11 Thermometer, for air temperature measurement.

6.12 Redox potential meter, (optional).

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

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

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

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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).

$$w_{dm} = 100 \times m_D / m_W \tag{1}$$

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 following Formula (2):

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

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