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

Part 5:

**Batch test with forced aerobic or
anaerobic conditions**

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

*Partie 5: Essai en bâchée dans des conditions aérobies ou anaérobies
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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 document 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).

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

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.

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

“Basic characterization” tests are used to obtain information on the short-term and long-term leaching behaviour and characteristic properties of materials. Liquid to 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.

“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.

“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 a) “Basic characterization” tests.

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 substances 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 can 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 meant to include genotoxicological testing.

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

Part 5: Batch test with forced aerobic or anaerobic conditions

1 Scope

This document specifies a test with which in situ available concentrations of inorganic substances (such as heavy metals, arsenic and phosphorus) and organic substances in soil and soil-like materials can be simulated under forced aerobic and anaerobic conditions. The toxicity can then be estimated based on these available concentrations.

The test described in this document aims 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 and ISO 17616.

The eluate obtained 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 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 11271, *Soil quality — Determination of redox potential — Field method*

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:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

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 8.2.2 and 8.2.3.

3.3 eluate

leachate
solution recovered from a *leaching test* (3.1)

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 sample, and the dry mass of the sample (S in kg of dry matter)

Note 1 to entry: L/S is expressed in litres per kilogram (l/kg).

3.5 dry matter content

w_{dm}
ratio, expressed in per cent, between the mass of the dry residue and the corresponding raw mass

Note 1 to entry: The mass of the dry residue shall be determined in accordance with ISO 11465.

3.6 water content

w_{H_2O}
ratio, expressed in per cent, 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 subsample(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

3.11

redox potential

electrochemical potential reflecting the oxidation-reduction status of a liquid chemical system

Note 1 to entry: For the purpose of this document, the liquid chemical system is the soil solution.

3.12

aerobic

descriptive of a condition in which molecular oxygen is freely available

3.13

anaerobic

descriptive of a condition in which molecular oxygen is not available

4 Principle

The test portions, which originally or after suitable pre-treatment have a particle size less than or equal to 2 mm, are brought into contact with water containing a low concentration (0,001 mol/l) of calcium chloride. The method is based on the assumption that during the test period equilibrium is approached between the liquid and solid phases at an imposed (aerobic or anaerobic) redox condition. The redox condition in the test is not based on the condition of the material itself, but on a forced aerobic or anaerobic condition, reflecting the application of the material in a different redox environment. Examples are the application of originally anaerobic sediment in an aerobic soil environment, or the utilization or disposal of originally aerobic soil at anaerobic aquatic conditions (e.g. in former sand pits). After the test period the solid residue is separated from the liquid. The separation procedure may strongly influence the test results and shall be particularly stringent for organic substances. Further characteristics of the eluate can be carried out with methods that are suitable for monitoring water. The eluate obtained is also suitable for carrying out ecotoxicological tests.

The conditions under which leaching has taken place such as pH, conductivity, DOC and redox potential are recorded in the test report. If the material causes turbidity in the eluate this shall also be recorded in the test report.

NOTE 1 These parameters often control the leaching behaviour of soil and soil-like materials and are therefore important for the 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 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

Reagents used shall be of analytical grade purity.

5.1 Demineralized water or deionized 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.

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

5.5 **Compressed air.**

5.6 **Nitrogen gas N_2 .**

5.7 **Na-L-Lactate** ($\text{C}_3\text{H}_5\text{NaO}_3$), analytical grade.

5.8 **Organic solvent** (acetone, analytical grade) **for rinsing and cleaning**, suitable for the material used for the bottle used and the type of analyte.

6 Apparatus

6.1 **Borosilicate glass bottle**, of high purity in accordance with ISO 5667-3, with a nominal volume of 1 l, having a cap of inert material, for example PTFE (polytetrafluoroethylene). Rinsing is compulsory; it shall be ensured 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 ($\text{pH} > 10$ and $\text{pH} < 3$) 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 **Measuring cylinders** for determining volumes with an accuracy of 1 %.

6.3 **Mechanical top stirrer.**

6.4 **PTFE stirring rod with two movable blades.**

The blades of the stirring rod shall preferably be folded in so that they can be passed through the neck of the bottle (6.1). During stirring the blades shall be unfolded.

6.5 **Gas washing bottle**, with a nominal volume of 1 l of inert material.

NOTE Inert material such as glass, PTFE or PFA (perfluoroalkoxy alkane).