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Soil quality — Guidance on leaching procedures for subsequent chemical and ecotoxicological testing of soils and soil materials

Qualité du sol — Lignes directrices relatives aux modes opératoires de lixiviation en vue d'essais chimiques et écotoxicologiques ultérieurs des sols et matériaux du sol

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ISO 18772:2008(E)

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Contents

Page

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.

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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.

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ISO 18772 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

Introduction

Current soil and soil-materials management (risk assessment practices or regulations) is often based only on the total amount of contaminants in soil. However, total composition is inadequate for the assessment of several types of impacts such as impacts on soil, groundwater and surface water due to leaching and subsequent transport of contaminants (inorganic, organic and natural radionuclides) with water. Indeed, for many constituents, a significant fraction of the total content is essentially non-leachable, that is to say non-removable when it comes into contact with a liquid.

Thus, a key aspect to assess the possible management solutions for soil and soil materials in relation to the presence of contaminant is the release-to-the-water phase. This can be addressed with leaching tests which can be used to characterise the source term when performing impact assessment and also for the determination of a leached amount of contaminants when checking compliance with respect to existing limits or for comparison purposes (e.g. quality control, treatment efficiency).

These statements are relevant for natural, contaminated and agricultural soils and also for soil materials.

Leaching tests, particularly those developed for soil and soil materials, are suitable for the following applications:

a) Application of leaching tests to determine the leaching behaviour in the framework of impact assessment

Generally, impact assessment is based on the source/pathway/receptor framework.

- Source: assess the release, identify speciation of constituents and retention mechanisms.
- Receptor: determine the potential targets.
- Pathway: estimate the transfer of the source towards the target (e.g. underground water, surface water, plants, soil organisms, ecosystems).

In this process, leaching tests are used to characterise the source term (so-called characterisation tests) in accordance with a given scenario (e.g. contamination of the groundwater due to a contaminated site or a soil amended with sludges), which can either be generic or site-specific.

Leaching tests may also be used as a tool to assess bioavailability (see ISO 17402).

b) Application of leaching tests for compliance and comparison

Based on the background information on the soil and soil materials sampled (e.g. origin, nature of constituents and contaminants, existing documented information, leaching behaviour), relatively simple and quick leaching tests can be performed for compliance and comparison purposes. In contrast to characterisation tests, this type of test is not designed to provide information on leaching mechanisms and controlling factors. However, it should be possible to link the information obtained with compliance tests to the more elaborate characterisation tests.

INTERNATIONAL STANDARD **ISO 18772:2008(E)**

Soil quality — Guidance on leaching procedures for subsequent chemical and ecotoxicological testing of soils and soil materials

1 Scope

This International Standard provides guidance on the appropriate use of leaching tests on soil and soil materials, in order to determine the leaching behaviour in the framework of impact assessment, or for compliance and comparison purposes, including information on the following:

- the choice of leaching tests, depending on the nature of the problem to be solved and the specific features of the different tests;
- the interpretation of the test results;
- the limitations of the tests.

In this respect, it is important to keep in mind that leaching tests do not aim to simulate real field conditions, but are designed to address the contact between a solid and a liquid phase for different purposes that are described in this International Standard.

This International Standard only concerns natural, contaminated and agricultural soils and soil materials. Questions relating to the leaching of wastes are not covered by this International Standard. It also does not cover the subject of bioavailability of contaminants to living organisms, which is covered by ISO 17402.

Leaching tests are designed and used for characterisation of the source term. It may be possible to address transport aspects with leaching tests if some basic requirements are known (e.g. hydrodynamic), thus allowing the determination of key transport parameters (e.g. retardation factors, particle-facilitated transport, attenuation processes).

In this International Standard, when the term “soil” is only quoted to simplify the writing, the broader term “soil and soil materials” shall be considered.

2 Normative references

The following referenced documents are indispensable for the application of this International Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 21268-1, *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials — Part 1: Bach test using a liquid to solid ratio of 2 l/kg dry matter*

ISO/TS 21268-2, *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials — Part 2: Bach test using a liquid to solid ratio of 10 l/kg dry matter*

ISO/TS 21268-3:2007, *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials — Part 3: Up-flow percolation test*

ISO/TS 21268-4, *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials — Part 4: Influence of pH on leaching with initial acid/base addition*

EN 12920, *Characterization of waste — Methodology for the determination of the leaching behaviour of waste under specificied conditions*

3 Terms and definitions

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

3.1
contaminants
substance or agent present in the soil as a result of human activity

NOTE There is no assumption in this definition that harm results from the presence of the contaminant.

[ISO 11074:2005]

3.2
eluate
solution obtained after the laboratory leaching procedure of a soil in contact with a leachant

3.3
leachant
liquid used in a leaching test

3.4
leachate
liquid that has percolated through soil under field conditions

3.5
leaching
dissolution and movement of dissolved substances caused by the movement and quality (e.g. pH, ionic strength) of water or other liquids in the soil

NOTE 1 In pedology, leaching is defined as the movement of dissolved substances caused by the movement of water or other liquids in the soil.

NOTE 2 Adapted from ISO 11074:2005.

3.6
leaching behaviour
release and time change in release from the soil upon contact with a leachant as affected by the conditions specified in the scenario, especially within the specified time frame

3.7
liquid to solid ratio
L/S
ratio between the total amount 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 kilograms of dry matter)

NOTE L/S is expressed in l/kg.

3.8
lysimeter
large-scale experiment set-up to simulate scenario-specific exposure conditions under more controlled conditions than in full-scale field conditions

3.9
multiparametric test
test aimed at measuring the influence of interrelated specific parameters on the release from a soil in the considered scenario

3.10
parametric test
test aimed at measuring an intrinsic property of a soil or to measure the influence of a specific parameter on the release from a soil in the considered scenario

NOTE This does not exclude the fact that other parameters may be influenced at the same time.

3.11
percolation
transport of infiltration water through a layer of soil

3.12
release
emission of constituents from a soil which pass through the external surface of a soil mass as specified in the considered scenario

3.13
scenario
case defined by a set of normal and exceptional conditions relevant to a particular disposal or utilisation situation for soil for the determination of the leaching behaviour within a specified time frame

3.14
simulation test
test aimed at simulating the combined effect of various parameters on the release in the scenario under consideration

3.15
soil material
material coming from soil and displaced and/or modified by human activity, including excavated soil, dredged materials, manufactured soils, and treated soils and fill materials

[ISO 17402:—[1]]

3.16
source term
set of information characterising the release of constituents from soil

3.17
transfer term
set of information characterising the transfer of the source term through the soil and/or the groundwater

4 General approach

4.1 Aim of leaching tests

The aim of performing a leaching test is to determine the expected constituent concentrations in solution when the leachant is placed in contact with a sample specimen under specified conditions. Many factors that influence dissolution and subsequent release of organic and inorganic constituents from a soil can be assessed through leaching tests.

Two main categories of leaching tests can be identified: static and dynamic tests. Among these categories, a wide variety of test procedures is available in literature, depending on a limited set of test conditions (e.g. pH of the leachant, liquid to solid ratio, contact time). The first question to emerge then is to know how to select the appropriate leaching test. It shall be considered that this question can be reformulated in some situations as how to select the appropriate set of leaching tests.

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[1] To be published.

4.2 How to choose leaching tests

4.2.1 For which purposes are leaching tests performed?

The first question is to determine whether leaching tests are performed to determine the leaching behaviour in the framework of impact assessment, or for compliance and comparison purposes. In the first case, the general approach to assess the leaching behaviour of contaminants from soils can be relevantly derived from the methodology described in EN 12920. The second case implies that background information to which leaching test results are to be compared is available (e.g. regulation, variability study, treatment efficiency).

This general approach is illustrated in Figure 1.

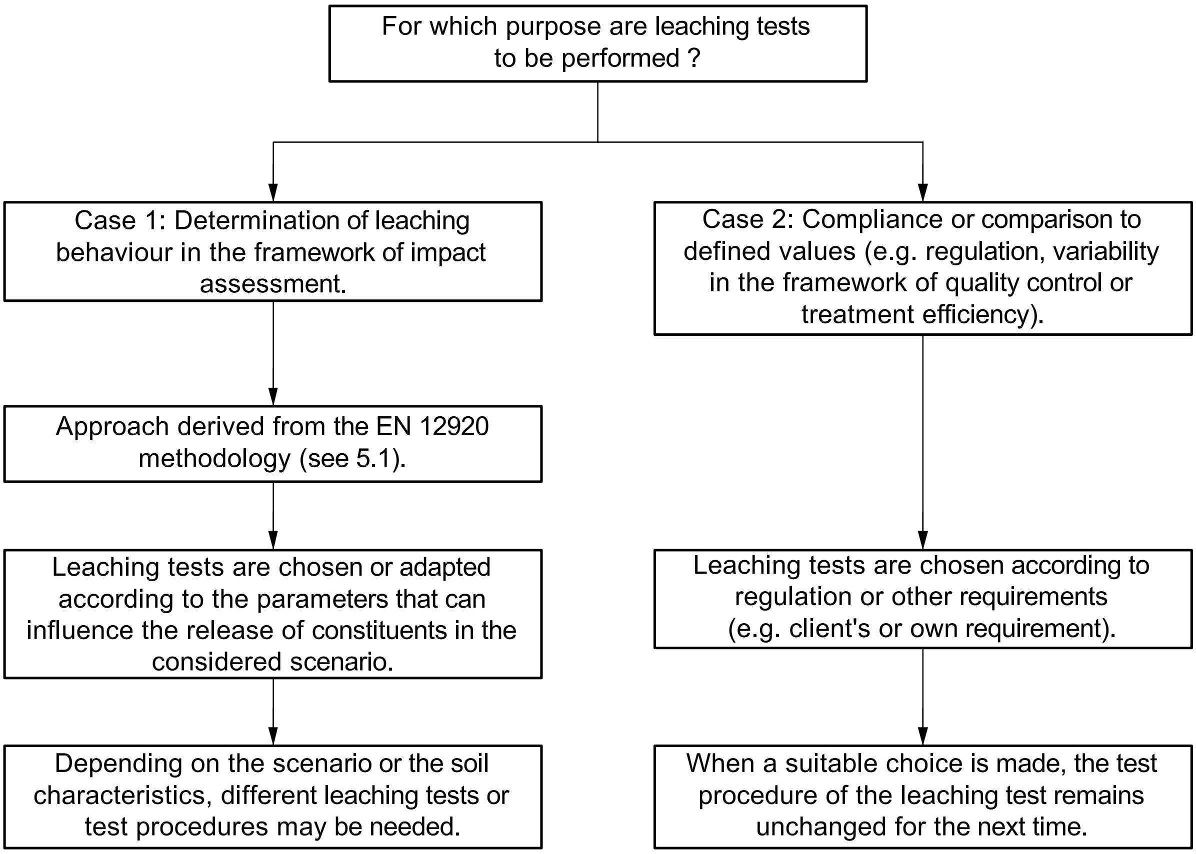


Figure 1 — General approach for the selection of the type of leaching tests

The aim of Table 1 is to allow easier identification of how to make the connection between the two cases of approach (cases 1 and 2) and the situations that stakeholders face up to in terms of soil management strategies.

Table 1 — Examples of management strategies and knowledge needed to fulfil the purpose

Management strategy	Problem	Related case	Description
Impact assessment, including beneficial use/ utilisation	Do the leaching properties of soil make it possible to comply with water quality criteria downstream of the site?	Case 1	No quality criteria are generally available that are related directly to the leaching of organic compounds from soil. However, quality criteria are available for groundwater and drinking water. In order to comply with these criteria, the leaching properties of the soil shall be determined and used as input for site-specific impact assessment.
	What is the present release of contaminants (snapshot) and what is the impact on groundwater?	Case 1	The release of contaminants from the soil under present conditions may be determined. The measured release may be used to evaluate the present impact on groundwater. This provides a first impression of the soil properties with respect to leaching and whether the soil is suitable for beneficial use/utilisation.
	What is the maximal leachable amount of contaminant?	Case 1	The maximal amount of contaminants that are available for leaching may be determined according to a defined time frame. For most soils (and other kinds of materials) there is no correlation between the total solid content of contaminants (either inorganic or hydrophobic for organic ones) and the leachable amount.
	How will the release of contaminants change with time?	Case 1	In risk assessment, it may be valuable to know if leaching from the source term is almost constant for a longer period or if it decreases within a shorter time period. The leachate or eluate quality may be estimated as a function of time.
	May the release of contaminant change significantly due to external influence over time?	Case 1	It shall be identified if there is a risk that the environment may influence the properties of the soil (e.g. pH changes) and whether the consequences of these changes with respect to leaching of contaminants from the soil should be known.
Disposal, or beneficial use/ utilisation	Do the leaching properties comply with leaching-based regulatory criteria or with an available variability frame of leaching characteristics?	Case 2	Leaching-based acceptance criteria generally only concerned inorganic compounds. Soils for disposal or beneficial use/utilisation containing organic compounds are still evaluated based on the total solid content.
Treatment	Does this treatment process change the leaching properties of the soil so that leaching criteria for disposal are fulfilled?	Case 2	Leaching properties may be determined for a given soil before and after a treatment in order to determine the ability of this treatment to make the soil comply with acceptance criteria in disposal or beneficial use/utilisation.
	Does this treatment process change the leaching properties?	Case 1	Leaching properties may be determined for a given material before and after treatment of the soil in order to evaluate if the treatment process is effective with respect to reducing the release of contaminants.
	Is it possible to improve the environmental properties of this soil with respect to the release of contaminant by leaching?	Case 1	By knowing the processes that control the release of contaminants from the soil, it may be possible to design or optimise the effective treatment processes.
Agricultural practices	To what extent will the added fertilisers or soil improvers be leached out of the soil? First assessment	Case 1	Leaching the soil sample after the addition of fertiliser or soil improver will provide information for assessing the amount remaining in the soil after exposure in the field (it will help in defining whether a new treatment is needed).
	To what extent will the added fertilisers or soil improvers be leached out of the soil? Routine testing	Case 2	After having established a relationship between laboratory testing and field exposure (see above), routine testing can be designed and performed.

4.2.2 Hierarchy in testing

A hierarchy in test use is promoted, in which more realistic and sophisticated tests are used to determine the leaching behaviour in the framework of impact assessment, whereas for quality control in soil processing or quality variations within a specific source of soil or, more generally, for compliance verification, more simplified tests are used.

The different kind of leaching tests can be gathered in three main categories, classified in ascending order of representativity and complexity.

a) **Compliance and quality control (QC) leaching tests**

These tests can be used for an initial screening of the release of soil constituents to water (contact times generally of one to a few days) or for checking compliance with respect to existing limit values or for the intercomparison and classification of different types of soils. This kind of test does not cover, and hence cannot allow, the assessment of the leaching behaviour of a soil in a given scenario. For typical compliance and QC tests, see ISO/TS 21268-1 and ISO/TS 21268-2.

b) **Basic characterisation**

This type of test can provide the intrinsic properties of soils to be used in subsequent modelling of release prediction.

- **Parametric tests.** These tests are intended for measuring an intrinsic property of a material or the effects (correlated) of specific parameters on release, on the basis of a contaminated material in an envisaged scenario. ISO/TS 21268-4 describes a typical parametric test.

  NOTE Diffusion coefficients, solubility or physical properties are examples of intrinsic properties of materials.

  Temperature, pH-value, liquid/solid ratio, redox potential, chemical properties or leaching-agent flow rate are examples of specific parameters which influence the behaviour towards leaching.

- **Multiparametric tests.** These tests are intended for measuring the combined effect of different parameters on release in the relevant scenario. For a typical multiparametric leaching test, see ISO/TS 21268-3.

For the first characterisation of a soil with these kinds of leaching tests, a direct use of test results, such as multiplication by a factor to extrapolate from laboratory scale to field scale, is generally not possible.

c) **Simulation tests**

These tests are aimed at reproducing, as well as possible, the field conditions and/or conditions when checking, on a large scale, the behaviour towards leaching predicted on the basis of the previous parametric or multiparametric tests. Lysimetric tests (so-called lysimeters) or large-scale column tests are examples of simulation tests.

Further information is given in 7.1.4.

4.3 Usefulness of leaching tests to understand and characterise different mechanisms occurring in soil

Soils are made up of three distinct phases: the solid matrix itself, the liquid phase (sometimes including the non-aqueous liquid phases) and the gaseous phase. Soil is also an ecosystem where biological activity takes place. The behaviour of the soil constituents (inorganic constituents consisting of major, minor and trace elements and organic constituents consisting of compounds of varying volatility and water solubility) within these porous media is governed by very diverse mechanisms, among which can be cited:

a) mobilisation and release of constituents and their chemical or mineralogical form;

b) pattern of water circulation through the granular bed (convection, dispersion, preferential flow) which regulates both the transfer and transport of constituents;

c) possible transport of dissolved constituents, especially constituents associated with organic carbon, and substances bound to fine particles (e.g. colloids, clay particles) within the granular bed;

d) physico-chemical interactions of the liquid phase with the solid matrix (e.g. mineral oxides, organic matter): adsorption/desorption, diffusion in stagnant water or other liquid or solid phases, diverse physico-chemical reactions (precipitation/dissolution, complexing, acid/base neutralisation, oxido-reduction, carbonation, ionic association/dissociation, etc.);

e) possible biological interactions (action of micro-organisms, mainly biodegradation or bioaccumulation).

The usefulness of leaching tests to the understanding and characterisation of the above mechanisms is presented in Table 2 (in this table each mechanism is referenced by the letter of the above list).

Table 2 — Usefulness of leaching tests to understand and characterise different mechanisms occurring in soil

Mechanism	Compliance and quality control tests	Parametric tests	Multiparametric tests	Simulation tests/ lysimeters
a)	x	x	x	x
b)	—	—	x [a]	x [a]
c)	—	—	x [b]	x [c]
d)	—	x	—	x [c]
e)	—	x [d]	—	x [c]

[a] With hydrodynamic characterisation, scenario information and modelling.

[b] Without filtration device.

[c] Previous information obtained with parametric and/or multiparametric tests, together with modelling, are needed to help in interpreting results and in qualifying the mechanisms.

[d] For example, by carrying out two tests in parallel, the first under biotic conditions and the second under abiotic conditions (all other test conditions being equivalent), it is possible to determine the effect of biological activity on the release.

5 Case 1: Application of leaching tests to determine the leaching behaviour of soil in the framework of impact assessment

5.1 Presentation and description of the assessment methodology

5.1.1 General

These issues are addressed according to the following 7-step procedure. More detailed information related to the methodology can be found in EN 12920.

- Step 1: Definition of the problem and the solution sought
- Step 2: Description of the scenario
- Step 3: Description of the source
- Step 4: Determination of the influence of parameters on leaching behaviour
- Step 5: Modelling of the leaching behaviour
- Step 6: Behavioural model validation
- Step 7: Conclusions

In that case, more than one test is needed to predict the release from soil under field conditions within a certain time frame. The outcome of this assessment is a source description in terms of release, as a function of time and external influences, for a given scenario.

5.1.2 Step 1: Definition of the problem and the solution sought

This step consists of describing what kind of soil is under study, what is the framework of the assessment (e.g. regulation requirements) and what is the question to be answered (e.g. release of organic contaminants within a certain time frame of a soil located in a contaminated site and for which the exposure conditions are well known). In this step, the time scale, expected results and constituents under investigation shall be defined.

Examples of questions to be answered are given in Table 1.

5.1.3 Step 2: Description of the scenario

5.1.3.1 General

This step consists of the description of the following:

- usual and exceptional exposure conditions of the soil in the studied scenario which may influence properties (e.g. release) within the chosen time frame;
- identification and relevance of the main influencing factors (and their related parameters) (e.g. rain and infiltration rate into the soil).

5.1.3.2 Typical situations

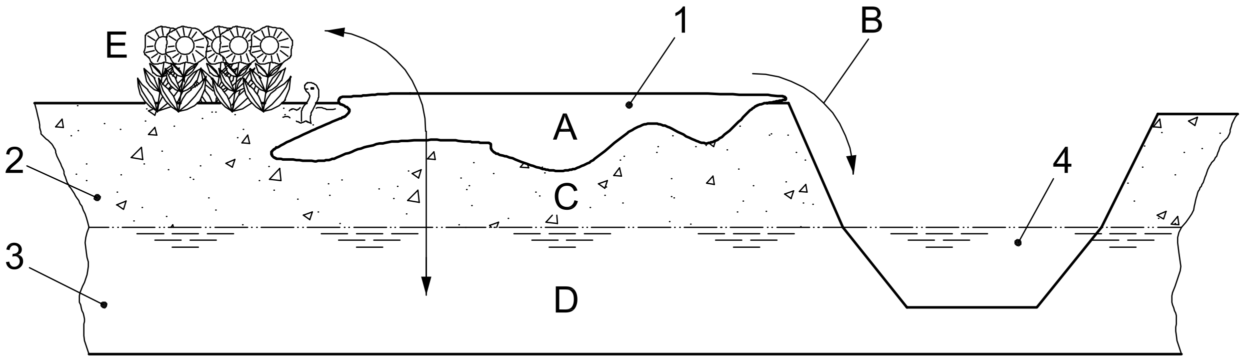
Typical situations frequently encountered on agricultural, natural and (de)contaminated soils are presented in Figures 2 and 3. For each of them, the different transfer processes involved are presented, the latter being symbolised by the letters A, B, C, D, E:

- A: source-term production of the contamination;
- B: transfer by run-off water;
- C: transfer into the unsaturated zone of the soil;
- D: transfer into the saturated zone of the soil;
- E: transfer towards organisms.

Scenario 1 (see Figure 2) is a generic case meaning that, depending on the situation, several variations may occur as follows:

- the depth of the contaminated area (A), because in natural or agricultural situations the contamination is in most cases concentrated only at the surface (less than 30 cm), whereas in industrial polluted sites the contamination may be deeper (a few meters);
- the saturated zone (D), which may be in contact with the polluted area (A), meaning that redox conditions may change.

In Annex A, a schematic representation of a contaminated site with relevant targets is given.



Key

1 backfill, soil, industrial soil

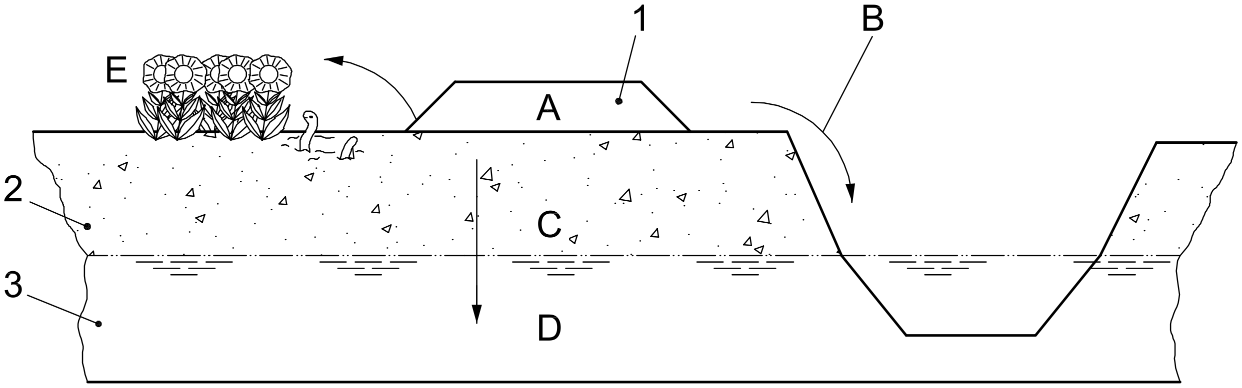
2 soil

3 groundwater

4 water

Figure 2 — Scenario 1 — Natural, agricultural and (de)contaminated soil

In scenario 2 (see Figure 3) the soil is used as a construction material that can stay more or less in contact with other environmental media (e.g. air, water, non-contaminated soil).



Key

1 backfill, soil, industrial soil

2 soil

3 groundwater

Figure 3 — Scenario 2 — Use of soil as a construction material

5.1.3.3 Identification and relevance of key factors

The critical factors that play an important role in the leaching of contaminants shall be identified and their relevance should be determined. The following factors, depending on the situation, might be of importance:

- mechanical and geotechnical conditions (e.g. permeability, compaction);
- hydrogeological and climatic conditions (e.g. temperature, rain, water-table modification);
- chemical conditions (e.g. changes in pH due to the use of lime, redox as a result of biological activity or water-table modification);
- biological activities;
- agricultural conditions (e.g. fertilisation, soil improvers, irrigation, ploughing) and conditions for the use of the site at different points in time.

5.1.4 Step 3: Description of the source

In this step, the present/initial properties of the soil are described and documented. These properties should be relevant to the type of soil and to the considered scenario. In this context, the following information might be needed:

- history of the site (e.g. land use, age of contamination);
- soil pedology, physical and mechanical characteristics (e.g. grain size distribution, clay and organic matter content, intra-particle porosity, bulk density, internal surfaces);
- mineralogy (e.g. amount of carbonates, of sulfides, of Fe, Mn, and Al oxides);
- chemical characterisation (e.g. soil pH, type of contamination, pools of non-aqueous liquid phases, tar-oil blobs);
- biological activity (e.g. microbial biomass).

5.1.5 Step 4: Determination of the influence of parameters on leaching behaviour

This step consists of identifying and determining the influence of the key chemical, physical, geotechnical, mechanical and biological parameters identified in step 2 on relevant properties of the soil determined in step 3 on the release dynamics as a function of time (e.g. the influence of the liquid to solid (L/S) ratio or of the content of organic matter on the release dynamics).

Based on the soil properties and the considered scenario, the appropriate leaching tests are selected and performed. Those tests can be (see Clause 4)

- parametric or multi-parametric tests, or
- simulation tests.

The leaching tests performed under that step are either more specific (by adapting the operating parameters) or more representative of real field exposure than those described in Clause 6. Generally, choosing a limited set of test conditions (e.g. pH and redox of the leachant, liquid to solid ratio, contact time) can allow key processes and influencing factors to be addressed in by far the majority of cases. In that respect, the application of upflow percolation test (see ISO/TS 21268-3) allows the determination of the release of constituents as a function of the L/S ratio under standardized conditions, both in terms of concentration and cumulative release. The pH-dependence test (see ISO/TS 21268-4) allows the elaboration of a relationship between the variation of pH and the released concentration of soil constituents.

5.1.6 Step 5: Modelling of the leaching behaviour

This step consists of developing and subsequently applying a behavioural model from a logical hierarchy of the influence of the relevant physical, geotechnical, biological and chemical parameters identified in step 4. Thus, a set of relationships is provided that describe the leaching behaviour of the soil in the considered scenario (especially in the specified time frame). The logical hierarchy described above includes simplification, i.e. the elimination of parameters shown to be negligible or irrelevant. The sophistication of the model depends on the defined problem and the solution sought.

Practically, applying a behavioural model may consist of the following:

- quantifying the chemical speciation of the system;

  The chemical form of the substances (inorganic, organic and natural radionuclides) of interest and the factors controlling release to the soil solution are crucial, as constituents present as free ions in solution or complexed by association with dissolved organic carbon (DOC) or particulates behave entirely differently in terms of transport (DOC-associated: more mobile) and uptake by organisms.

- verifying the relevance of the model on some leaching test results, e.g. the dynamic release (step 4);
- modelling the release within a certain time frame well beyond the duration of the test procedure under the exposure conditions of the considered scenario, by using the results of parametric tests carried out during step 4.

Depending on the scenario considered and the relevant related parameters, different kinds of modelling may be implemented in the following manner.

a) Geochemical. Such modelling can be performed by using a chemical speciation program with the results of ISO/TS 21268-4 if the following elements and parameters have been measured in eluates (the electroneutrality of each eluate shall be checked, i.e. as large a quantity of cationic species as anionic ones shall be obtained):

   - full analysis of major, minor and trace inorganic elements and of organic compounds and contaminants and, if relevant, natural radionuclides;
   - pH, conductivity, Eh and DOC in the eluates.

Knowledge of the following information on the soil sample is also of importance:

- organic matter (e.g. measurement of total organic content);
- mineralogical composition, in particular the quantity of reactive Fe-oxides and Al-oxides, and clay fraction.

b) Multiphase (e.g. solid/liquid/gas).

c) Transport. This kind of modelling shall consist both of “water” circulation (if relevant for the scenario under consideration) characterised by hydrodynamics, and of the interaction between the liquid phase and the soil matrix, either physically (exchange between water in movement – connected porosity, and stagnant water – unconnected porosity) and/or chemically (e.g. kinetics of sorption/desorption of organic compounds, cationic exchange). The transport of solid particles and of colloids is also of great importance.

d) Degradation of organic matter/sequestration (e.g. plant uptake).

e) Degradation of organic contaminants.

Results coming out from the different modelling stages are gathered to provide relevant information on the overall source-term description (for the implementation of the source term into the soil and groundwater impact model, see Clause 8).

The different stages cannot be seen as separate independent items. This implies that a proper behaviour model shall integrate all relevant aspects simultaneously. For instance, degradation produces DOC, which affects release of the metal and organic contaminants.

Modelling is a tool that may provide suitable and/or useful information or results either for case-by-case studies or even for generic studies. It is a key aspect for describing a time-dependent source term.

5.1.7 Step 6: Behavioural model validation

Validation of behavioural models describing release in specific scenarios is important to allow confident decision making based on these models. No absolute validation can be expected for the long-term models of leaching behaviour, especially when data have been extrapolated over periods of several hundreds or thousands of years. However, certain validation procedures may result in a satisfactory level of confidence given the inherent uncertainty in any prediction. It may be appropriate to assess this level of uncertainty.

This step consists first in validating the behavioural model and secondly in verifying consistency of the model prediction with one of the following procedures.

a) Verification of the consistency between parameter-specific tests and simulation tests; this procedure consists of checking the coherence and consistency (given the forecast level targeted) between, on the one hand, the results obtained by modelling release based on the parameter-specific tests and, on the other hand, the simulation tests.

b) Field verification of predicted behaviour (lysimeters or field testing); although limited due to the high cost involved, the verification of release in actual practice on sites that have been placed as full-scale demonstrations and which are generally well documented provides one of the best means to compare model predictions based on laboratory leaching data to actual field work.

In performing this validation step, the importance of scale effects between the results of modelling using the laboratory results, and more realistic tests or field monitoring shall be considered and taken into account, as far as possible.

As a preliminary step to the verification of modelling validation results, a useful stage is to compare dynamic tests results obtained at different scales (e.g. laboratory scale with ISO/TS 21268-3, lysimeter and field scale — see example for mixed inorganic waste with predominantly soil materials in Annex B). This comparison should be done by using the L/S as a basis of reference. The observation of potential differences in slopes and in curve shapes may allow for non-interacting species such as chloride to

- provide information on the preferential flow in full-scale operations,
- extrapolate from one scale to another and, as a consequence, forecasting as a function of time with more confidence.

For other elements of which the release is controlled by solubility (gradient and/or kinetics), the comparison in terms of L/S ratio generally does not allow such use and interpretation of data.

5.1.8 Step 7: Conclusions

This step consists of examining the conclusions reached in the previous steps and in determining whether the defined problem has been solved or whether information is lacking. In the situation where the defined problem has not been solved, either the previous steps shall be redone with modifications (e.g. a key parameter may have been neglected, further/more detailed/more complex experiments shall be done to determine the lacking parameters which are crucial for understanding the problem) or it shall be considered that, given the current state of knowledge, it is not possible to evaluate the leaching behaviour within the considered time frame for the type of soil in the scenario under consideration.

The evaluation shall be carried out by relating the results (and their associated level of confidence) to the originally defined problem. From this, one of the following conclusions shall be drawn.

a) The results may provide the solution (e.g. take the decision on how to manage the soil).

b) The results may not be conclusive and it may be necessary to repeat previous steps in an iterative way, e.g. amending the model, changing the scenario, assessing a new soil property. Return to the appropriate step of the methodology.

c) The results may indicate that it is impossible to reach a solution based on available state-of-the-art tests and information. In this case, state the reason why the assessment is not providing a solution.

6 Case 2: Compliance and comparison purposes

Leaching tests developed for compliance and comparison purposes are generally simpler and easier to carry out than those used in Case 1. The leaching procedure is generally standardized by setting conventional operating parameters allowing a higher degree of robustness, repeatability, reproducibility and a broad applicability to different kinds of soil. For typical compliance and quality control tests, see ISO/TS 21268-1 and ISO/TS 21268-2.

In general, they do not provide, nor aim to provide, the same level of information as that provided by most of the (characterisation) tests used in Case 1. The outcome of these tests is a leached amount of constituents. They are often used within regulatory frameworks to assess compliance with criteria, which ideally should be based on assessments such as those described under Case 1. They can also be used to

- provide a screening of the release of soil constituents to water,
- check compliance with respect to existing relevant data previously obtained in the framework of quality control, with the same test, and
- allow intercomparison and classification of different types of soils.

Concerning compliance and comparison purposes, a distinction shall be made. For compliance, the leaching test is usually defined by regulations and generally allows no deviation nor adaptation (the test is often applicable to a wide range of material because of its generic conditions) whereas for comparison purposes (e.g. treatment efficiency) background information on the soil (e.g. origin, nature of contaminants, existing documented information, leaching behaviour) is needed to fit the test with the objectives (e.g. modification of the test conditions, determination of specific properties or parameter such as redox, DOC).

A direct link between characterisation test results and compliance test results is crucial for proper decision making as criteria can be based on characterisation rather than compliance test results. In fact, linking the test lends more credibility and power of decision to the compliance test results.

These methods and the different operating parameters (choices and description) are presented exhaustively in 7.1.2.

7 Description of test methods

7.1 Laboratory methods for basic characterisation and compliance/quality control testing

7.1.1 Principle of the methods

These tests consist of placing the soil sample in contact with a leaching solution, in a unique batch or in a dynamic way. After a defined duration, the eluate is recovered in order to perform the physical and/or chemical and/or ecotoxicological characterisations.

7.1.2 Key factors

7.1.2.1 General

Depending on the tests, the following key parameters of the leaching procedures, related to key factors of exposure of a soil in a field situation, can vary or be changed:

- the contact mode between soil and leaching solution (static or dynamic) (see 7.1.2.2);
- the ratio of the mass of leaching solution to the mass of soil sample (L/S dry mass ratio) (see 7.1.2.3);
- the type and characteristics of the leaching solution (see 7.1.2.4);
- the particle-size distribution and state (dry or wet) of the soil sample to be leached (see 7.1.2.5);
- the method of agitation/stirring (batch tests) (see 7.1.2.6);
- the column dimensions (percolation tests) (see 7.1.2.7);
- the flow mode: up-flow/down-flow (percolation tests) (see 7.1.2.8);
- the flow rate of the leachent (percolation tests) (see 7.1.2.9);
- the duration and contact time (see 7.1.2.10);
- the temperature (see 7.1.2.11);
- the method of recovery of the eluates (see 7.1.2.12);
- the biological activity (see 7.1.2.13);
- the presence of organic constituents (see 7.1.2.14).

7.1.2.2 Contact mode between soil and leaching solution (static or dynamic)

Two main categories of leaching tests can be identified: static and dynamic tests. Static tests are currently performed in tanks and are usually called “batch tests”. They can be performed in one run or with multiple renewal of the leachant.

Dynamic tests are generally performed in a column under continuous flow conditions with permeable soils (e.g. permeability $> 10^{-8}$ m/s).

7.1.2.3 Ratio of the mass of leaching solution to the mass of soil sample (L/S dry mass ratio)

7.1.2.3.1 Batch tests

There are different L/S specified (see ISO/TS 21268-1 and ISO/TS 21268-2), 2 and 10 respectively, 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 soil and, on the other hand, by different leaching conditions dictated by the soil itself (as a result of the compounds of the soil dissolved in the eluate). It shall be noted that there is generally 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.

NOTE Leaching at different L/S values can lead to the same results for strongly sorbing compounds [e.g. polycyclic aromatic hydrocarbons (PAH), some metals] and soils with theoretically high sorption capacities. This is because a very low equilibrium concentration is reached in both cases.

At lower L/S, some species are present in the eluate at higher concentration, as a result of the smaller volume of leachant, where the entire content of the element is present in solution (non-interacting species, e.g. Cl, Na, K). A different pH, as a result of the same amount of alkalinity dissolving in a smaller amount of leachant, can also lead to different concentration levels in the eluate.

For some other elements, for which release is solubility controlled, a difference between low L/S and high L/S can be observed depending on the saturation level of the element in the eluate. In exceptional cases, the concentration at L/S = 10 is higher. This can occur, when a significant pH difference exists between L/S = 2 and L/S = 10 and the leachability of the constituent of interest is very sensitive to such a pH change.

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

Table 3 presents the main advantages and limitations concerning the use of each of these two categories of L/S ratio.

Table 3 — Advantages and limitations of low (e.g. L/S = 2) or high (e.g. L/S = 10) L/S mass ratios

L/S mass ratio	Advantages	Limitations
Low L/S (e.g. L/S = 2 l/kg)	Limits the dilution of the extracted constituents which are therefore less likely to be present in concentrations below the limit of detection of the methods used for chemical analysis. Closer to the pore water concentration but still quite far from true pore water (L/S about 0,3). For highly contaminated soil, the low L/S may ensure that certain complexing effects, which would occur under field conditions, are taken into account in the test.	The extract is not applicable to extreme clayed soils and organic soils. These soils limit the recovery of the eluate or even make it impossible. Thus, depending on the type of the sample, the volumes of eluate recovered differ, making the forecasting of the volumes available for the subsequent characterisations impossible. If the leached sample is highly contaminated with soluble constituents, the extraction capacities of the solution may be limited, as the solution may become saturated. The recovery of the eluate may be more difficult (the quantity of soil to be eliminated is greater) and, depending on the retention capacities, centrifugation methods are required. Depending on the subsequent characterisations to be performed (physical and/or chemical and/or ecotoxicological [a]), the available volumes may be insufficient and may make it necessary to perform more than one test (e.g. parallel leachings) to produce the needed volume of eluate. In the case of subsequent ecotoxicological testing, the dilution, if any, of the eluates may reduce the toxic response of organisms.
High L/S (e.g. L/S = 10 l/kg)	The method is applicable to most soil typologies. The volume provided is considerably greater than the water retention capacity. The extraction capacity is high [b] (saturation of the solution being more difficult, taking into account the volume). The transfer is thus maximised. The obtention of the eluate is generally simpler (less solid sample to separate). The volumes of eluate provided are more likely to be sufficient for the subsequent physical and/or chemical and/or ecotoxicological characterisation to be performed. [a]	The high volume of leaching solution generally produces a “diluted” eluate compared to the ones obtained at a low L/S ratio. This may cause the concentration of constituents to be below the limit of detection of the chemical analytical methods used. Similarly, the levels may be too low for detection in ecotoxicological tests. This is particularly problematic for slightly contaminated soils. Even for highly contaminated soils, the high L/S of the eluates produced are likely not to account for certain complexing effects which would occur under field conditions.

[a] Large volumes may be necessary for conducting the chronic toxicity tests and the chemical analysis of the organic contaminants.

[b] When determining the organic contaminants by extraction and concentration, this method allows an increase of the detection thresholds.

The choice of an L/S mass ratio for leaching therefore depends on the objectives:

- high L/S ratios (e.g. L/S = 10) can be applied to a wide range of soils, and can maximise the transfers of contaminants and produce enough volumes for performing the physical and/or chemical and/or ecotoxicological characterisations;
- low L/S ratios (e.g. L/S = 2) can be applied to study situations where it is important to maximise the concentration level in the eluate rather than the transfer, e.g. to be as close to pore water concentrations as possible, or to make it less likely that concentration levels are below the limit of detection or toxic response for the characterisation methods used.

It should be noted that the L/S ratios of the tests to be used in regulatory frameworks are generally specified.

7.1.2.3.2 Percolation tests

For a largely percolation-dominated situation, such as contaminated site evaluation, a percolation test is the most crucial test to evaluate release.

For rather impermeable soils (e.g. clay or clayey soil), specific test methods should be carried out to take into account the fact that these granular materials, in a given scenario, behave more like a flow-around “monolith” than as granular percolating material.

In the up-flow percolation test, the following eluate fractions are collected: L/S = (0,0 to 0,1) l/kg, (0,1 to 0,2) l/kg, (0,2 to 0,5) l/kg, (0,5 to 1,0) l/kg, (1,0 to 2,0) l/kg, (2,0 to 5,0) l/kg and (5,0 to 10) l/kg. The size of the fractions is seen to increase with increasing L/S.

The L/S of many of the field leaching scenarios (landfills and field applications), which could be modelled on the basis of percolation test results, often requires many years to reach a value of L/S = 1 l/kg or 2 l/kg. To place the L/S ratios in perspective, two simple scenario calculations can be considered. For a 2 m thick layer of soil with a density of 1 t/m^3 through which water (e.g. infiltrating rainwater) is percolating at a rate of 200 mm/year, a L/S ratio of 2 l/kg and 10 l/kg is attained in 20 years and 100 years, respectively. For a 20 m thick layer of soil with a similar density and percolation rate, L/S ratios of 2 l/kg and 10 l/kg are attained after 200 years and 1 000 years, respectively.

It should be noted, that the collection of the eluate as fractions is well suited to describe the amount of constituents leached at a given L/S. This procedure is, however, less suited to describe the actual eluate composition at various values of L/S since the concentrations are measured as averages over increasing ranges of L/S. If a description of eluate composition at specific L/S values is desired, small eluate samples could be collected and analysed at those L/S values. An alternative to measurement is to calculate the average concentration from the release curve by focussing on a small L/S interval.

7.1.2.4 Type and characteristics of the leaching solution

7.1.2.4.1 General

When performing leaching tests on soil, it is recommended (and prescribed in several tests such as in ISO/TS 21268-1, ISO/TS 21268-2, ISO/TS 21268-3) that a weak solution of $CaCl_2$ in demineralised water (0,001 M) is used as a leachant rather than, for example, demineralised water alone. The intention of this is to

- provide a “natural” ionic strength typical of soil pore water, and
- reduce clogging of filters by enhancing the agglomeration of soil particles, thus allowing limitation of the presence of colloids (when filtering is advisable).

To produce this weak solution of $CaCl_2$, instead of demineralised water, water of equivalent purity with a pH between 5 and 7,5 and a conductivity $<$ 0,5 mS/m, such as distilled water or deionised water, can be used. The leachant is water made up to 0,001 M $CaCl_2$.

In other non-standardized leaching tests, other solutions have been used (e.g. humic acid solution, methanol, DiethyleneTriamine Pentaacetic Acid (DTPA), $NaNO_3$) for the determination of specific parameters. In this case, the performance of possible ecotoxicological tests on the obtained eluates shall be accompanied by a blank test.

NOTE Sequential and selective extractions for speciation and bioavailability determination are not covered.

For organic contaminants, where the quantification of the water leaching potential shall be carried out eliminating all possibility of biological degradation, a solution containing a biocide (e.g. NaN_3) may be used. In this case, the ecotoxicological analyses cannot be performed (see 7.1.2.13).

The influence of the pH and of the reducing/oxidising properties of the leaching solution is specifically discussed below.

7.1.2.4.2 pH of the leaching solution

In ISO/TS 21268-1, ISO/TS 21268-2 and ISO/TS 21268-3, the final conditions of the tests are currently imposed by the soil itself. This is generally the case for the pH. The sensitivity of leaching to relatively small changes in pH may be significant. Such sensitivity may 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 may affect the test results, as they may lead to pH/redox changes in the eluate.

In ISO/TS 21268-4, the pH is the parameter that is made to vary in order to study its influence on the solubilisation of the soil constituents.

7.1.2.4.3 Oxidising/reducing properties of the leaching solution

Soils to be tested may exhibit reducing or oxidising properties, which is evident respectively from a low or high redox potential in the eluate. For a proper evaluation of soil, it is important to be aware of this aspect as different degrees of oxidation in sample handling and storage may induce varying results.

Another aspect is the issue of oxidised conditions in laboratory testing (see ISO/TS 21268-1, ISO/TS 21268-2, ISO/TS 21268-3, ISO/TS 21268-4), whereas in the field or in lysimeter experiments, reducing conditions may prevail.

The effect of oxidising/reducing conditions may be studied by the application of ISO/TS 21268-4 in which different oxidising or reducing leachants are used instead of making the pH vary. For a better interpretation of the results, this test should be carried out at only one pH condition. In such a test, care should be taken in the preservation and analysis of the eluates.

7.1.2.4.4 Electric conductivity of the leaching solution

The level of electric conductivity gives a first approximation of the ionic strength of the solution. This level also decides on the amount of colloids leached. Distilled water results in an extreme mobilisation of colloidal materials.

The understanding of the level of electric conductivity is an important condition to decide whether or not thermodynamic laws on which geochemical models are based are applicable.

7.1.2.5 Particle-size distribution and state of the soil sample to be leached

The maximum size of the soil particles submitted to leaching should be standardized in order to homogenise the exchange surface between the leached sample and the leaching solution. Sieving of the sample through a 4 mm mesh sieve is recommended, the oversize being weighed and its type identified. A larger size of particles (< 10 mm) reduces the contact surface and may limit the extraction of the contaminants. In the same way, as the objective is not to extract a maximum amount of contaminants, a fine particle-size reduction is not desirable. Where sieving is not possible as such, moderate drying of the sample (at 40 °C maximum) may be performed before.

NOTE 1 If national regulations specify other particle sizes, other sizes can be used.

Moreover, in a column during a percolation test, the particle size/particle-size distribution has an influence both on the potential representativity of the test portion in the column and on the time required to approach the initial equilibrium between the dissolved and the solid phases for various components. Both properties improve with decreasing particle size, i.e. the representativity increases and the pathway for diffusion in the solid phase shortens, thus decreasing the time needed to approach equilibrium conditions. As a rule of thumb, the largest particle size should be at least 10 times smaller than the diameter of the column (see Table 1 and Clause C.3, of ISO/TS 21268-3:2007). Also, the length of the column shall be at least three to four times the diameter of the column. If possible, the particle-size distribution of the soil to be tested should remain unchanged. Crushing the sample should be avoided in any case, at least for soils which contain carbonates. The crushing of a granular material may alter its leaching properties by opening new surfaces, disturb surface layers that may have formed as a result of ageing processes, and affect the pH of the experiment.

The mass of the leached sample can be related to the raw or dry matter. It is essential, with the aim of comparing the results of the subsequent characterisations (and, in particular, for the ecotoxicological analyses), to always work with an identical supply of contaminated soil, expressed as a dry mass equivalent (the dry matter of the sample being previously determined on a soil sample).

NOTE 2 It is easier to convert chemical results on the basis of dry weight than ecotoxicological ones (because ecotoxicological tests are performed on subsequent dilutions of the original eluate).

7.1.2.6 Method of agitation/stirring (batch tests)

The aim of stirring is generally to ensure sufficient contact between the entire sample and the leachant, to homogenise the solution but not to create new surfaces or disturb surface layers. This also limits the abrasion of the container in which the leaching is performed.

Several stirring systems are available and the most suitable have been recommended by the different batch tests standards (e.g. end-over-end tumbler at 5 min^{-1} to 10 min^{-1} in ISO/TS 21268-1 and ISO/TS 21268-2).

7.1.2.7 Column dimensions (percolation tests)

For good reproducibility of the leaching test itself and proper interpretation of the results, the flow pattern within the column should resemble plug flow as closely as possible. Based on experience, this is assumed to be the case for linear velocities (through the empty column) in the range of $2{,}08 \times 10^{-4}$ m/h to $6{,}25 \times 10^{-3}$ m/h (i.e. 0,5 cm/d to 15 cm/d) if the length of the column is at least three to four times the diameter. From the point of view of representativity of the test portion placed in the column, and of securing relatively large fractions of eluate for chemical analysis, a large column would be preferable. However, the longer the column, the longer the time it would take to reach a certain L/S value for the same linear velocity. Experimental data obtained so far do not indicate very significant differences as a function of flow rate within the above-mentioned range. However, the number of materials for which this information has been verified is very limited and thus cannot be generalised.

It has therefore been decided to use two options for column size: one with an inner diameter of 5 cm and one with an inner diameter of 10 cm, both with a filling height of (30 ± 5) cm. The conditions prescribing which column size is to be used for a particular sample of soil have been presented in the table in 6.2 of ISO/TS 21268-3:2007.

7.1.2.8 Flow mode: up-flow/down-flow (percolation tests)

ISO/TS 21268-3 is intended to describe the leaching properties of soils being percolated by a leachant under saturated conditions. This is best achieved by passing the leachant through the column in up-flow mode. Application of the up-flow mode further reduces the risk of channelling and preferential flow, provided the flow rates are not excessive.

This way of circulating the leachant is far from field conditions, or conditions used mainly in lysimeters. Thus, this can lead to difficulty in the use of saturate dynamic conditions for modelling unsaturated (e.g. intermittent weathering) field conditions.

Down-flow mode is generally used in simulation tests [large-scale columns or lysimeters, (see 7.2)] in combination with unsaturated conditions. The leachant shall then be applied in a way which allows for homogeneous repartition of the leachant on the surface of the column or lysimeter.

7.1.2.9 Flow rate of the leachant (percolation tests)

The actual flow rate of the leachant in ISO/TS 21268-3 (expressed in ml/h) is based on and calculated from an apparent linear velocity, expressed in terms of cm/d (cm/24 h), through the empty column. This is convenient since the equilibrium/non-equilibrium conditions in the soil/leachant system are related to the linear velocity, and are independent of the pore volume of the packed column (the actual pore velocity is considerably higher than the open-column velocity). The open-column linear velocity is expressed in the same way as, and is comparable to, the rate of infiltration of precipitation into a landfill or a utilisation scenario of soils.

The major advantages of a low linear velocity of the leachant are that it is more likely to ensure that the local equilibrium conditions are fulfilled, and that it comes closer to the actual flow conditions occurring under field conditions. It should be noted, however, that the laboratory procedure is not aimed at the same linear velocity as in field conditions, as this might lead to a very long test duration. This is also the major disadvantage of low flow rates, particularly when the test is run to an L/S value of 10 l/kg. Conversely, the major advantage of a high leachant flow rate is that it limits the duration of the test.

NOTE For specific purposes (for instance, compliance testing or for a landfill scenario with top cover), it can be sufficient to know the leaching characteristics up to a predetermined L/S ratio, for instance, L/S = 2. In that case, the test can be stopped after the appropriate L/S has been reached.

It should be noted that, for a given soil, the influences of particle size and flow rate on the achievement of local equilibrium are interrelated. Small particle sizes and low flow rates favour equilibrium and vice versa. For a given soil with a given particle-size distribution, there should, in principle, exist an upper limit for the leachant flow rate below which the local equilibrium assumption is always fulfilled. This can be used to check the equilibrium situation: if similar columns with identical soil are run at two different leachant flow rates and the results are similar, the local equilibrium requirement is likely to have been fulfilled at both flow rates.

For the test in accordance with ISO/TS 21268-3, a linear leachant velocity of $6,25 \times 10^{-3}$ m/h (i.e. 15 cm/d) has been fixed. This enables the test to be carried out to a final L/S = 10 l/kg in approximately 30 d and to reach L/S = 2 l/kg within approximately one week. Test results indicate that the local equilibrium condition appear to be fulfilled for several components and several materials, but not for all. For the sake of reproducibility it is therefore important to maintain a relatively constant and precise linear velocity [$(6,25 \times 10^{-3} \pm 8,33 \times 10^{-4})$ m/h (i.e. (15 ± 2) cm/d)] when applying this flow rate compared to the sometimes very low flow rates observed in field conditions.

Ideally, the flow rate should be based on the grain size distribution as it is well established in the literature that multiple processes are governed by non-equilibrium (e.g. release of DOC, evolution of pH, EC). Thus, the effect and extent of non-equilibrium should be checked. All these parameters are affected by the flow rate of the leachant. Interruption of flow rate may be applied during the percolation test in order to check if local equilibrium is reached. A useful means is to compare concentrations before and after interruption.

7.1.2.10 Duration and contact time

Compliance/QC tests, such as ISO/TS 21268-1 and ISO/TS 21268-2, are currently conducted over periods not exceeding 24 h. Compliance/QC tests are based on the assumption that equilibrium or semi-equilibrium is reached under the test conditions, this equilibrium or semi-equilibrium being, in most cases, far away from those observed in field studies (see 7.2, Table 7). The contact time required to reach the state in equilibrium or semi-equilibrium depends on the combination of soil type and 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 (see 7.1.2.4), such as marine soil, volcanic soil and organic soil, are important factors that determine how fast equilibrium or semi-equilibrium is reached.

The duration of percolation tests is longer than the one of batch tests and depends on several factors, such as flow rate (see 7.1.2.9), column size (7.1.2.7) and grain size (7.1.2.5). For instance, the duration of the procedure defined in ISO/TS 21268-3 is between three and four weeks.

7.1.2.11 Temperature

Temperature modifies the kinetics and the chemical equilibria of the constituents within the “soil-leaching solution” system. Care should therefore be taken to ensure a leachant/eluate temperature as constant as possible, and as close as possible to the prescribed value (in general, a temperature of (20 ± 5) °C is advised). The safest way to achieve this is to ensure that the room temperature remains within the required range day and night during the entire test period. Alternatively, temperature control may be achieved in the column by applying a heated/cooled water jacket or a similar device.

The temperature, (20 ± 5) °C, has been chosen to represent common indoor conditions. Nevertheless, the range of temperatures should be limited as much as possible to (2 to 4) °C since, according to van’t Hoff’s rule, this can affect rate parameters dramatically.

7.1.2.12 Method for recovery of the eluates

After the leaching stage, the soil sample shall be separated from the eluate. Separation of solid and eluate is a critical step in the procedure. It should be kept in mind that, for hydrophobic compounds such as, for example, PAH, the separation procedure may be a critical test step. Indeed, if the separation is insufficient, the leached concentration may, for hydrophobic compounds, be significantly overestimated due to the presence of “artificial colloids” in the eluate.

This separation shall be carried out by high-speed centrifugation in case only organic compounds or both inorganic and organic constituents are to be analysed. Decantation and/or filtration are not suitable for that purpose. Table 4 presents the advantages and limitations of these different techniques.

It should be noted that the concentration of dissolved species can be affected by the acceleration applied: the higher the speed, the higher the concentration due to friction among soil particles in the eluate.

NOTE If organic compounds are analysed, it can be useful to trap the eluate directly in an organic solvent to extract them before the centrifugation step.

Table 4 — Advantages and limitations of the different methods of separation of the eluate from the solid phase

Separation technique	Advantages	Limitations
Decantation	— Technique simple to implement. — Allows the presence in the eluate of more or less coarse particles. — Depending on the duration of the decantation and on the depth at which the sampling is carried out, the maximum size of the particles recovered can be estimated. — Approximation of surface run-off.	— Difficult to standardize (duration, bottle height, speed of decantation of the particles variable). — Certain constituents may not decant (e.g. colloids, clayey soils). — If the decantation time is too long, this can result in initial degradation or transformation of the molecules. — May lead to overestimation of leaching test results.
Filtration	— Technique simple to use. — Depending on the porosity of the filter, there may be colloids present in the eluate lower than the filter cut-off which are transportable in the soil solution. — Facilitates the standardization of the eluates. — Sterilisation possible by 0,22 µm filtration. — Separation is independent of the density of the particles.	— Depending on the porosity of the filter, there may be colloids present in the eluate lower than the filter cut-off which can lead to an overestimation of the solubility. — The formation of a filter cake can result in the filtration of particles smaller than the filter cut-off (the regular replacement of the filter when performing filtration allows this effect to be limited, but may lead to excessive use of filters). — The type of the filter can interfere with the recovery of certain contaminants (particularly organic contaminants). The filter should be chosen on the basis of the type of molecules being sought. — Glass-fibre filters may leach metals.
Centrifugation	— Depending on the time and on the acceleration, there may be particles present in the eluate. — Facilitates the standardization of the eluates. — Prevents sorption of organic contaminants on filter materials. — High-speed centrifugation (20 000 g to-30 000 g) may result in the elimination of fine particles, depending on their density.	— Requires a high investment in equipment because inert materials have to be used (e.g. centrifugation bottles made of glass or steel).

In the context of these water-leaching primary characterisation tests, the technique to be used varies as a function of the objective.

- If the objective is to quantify the concentrations and the toxicity of the dissolved molecules, centrifugation (e.g. 20 000 g to 30 000 g) should be employed. For the determination of inorganic contaminants, 0,45 µm filtration is conventionally employed.
- If the objective is to quantify the concentrations and the toxicity of the dissolved molecules and of the ones associated with fine particles, the three techniques can be used, provided that the maximum size of the suspended particles can be estimated.

7.1.2.13 Biological activities

Biological organisms and respective activities may affect leaching of inorganic and organic constituents by absorption/adsorption, changing redox condition or degrading organic matter. As an example, in column tests under saturated conditions, bacterial activity can induce reducing conditions and mobilisation of oxide-bound species, in particular when organic-rich soil is investigated. Effects of biological activities can be studied by performing the tests in biotic and abiotic conditions. As a corollary, undesired biological activity may be prevented by performing the test under abiotic conditions. Abiotic conditions may be obtained by sterilisation of the soil sample (e.g. gamma ray irradiation, use of biocides). Biological activity may also be reduced by lower temperatures, such as 5 °C. In the latter case, one should keep in mind that this change of temperature also affects the release kinetics of some constituents (see 7.1.2.11).

Sterilisation of the soil samples and use of biocides, e.g. higher concentrations of NaN_3 may also affect the release kinetics of some constituents by changing the ionic strength of the eluate or changing the properties of the organic matter by sterilisation.

Another effect of biological activity may be the degradation of the (trace) organic compounds which are being studied in the leaching test. A sample of soil contaminated with one or more organic compounds is likely to contain micro-organisms that have been adapted to those particular compounds. Due to the optimised conditions in a laboratory leaching test as compared to the field (suspension in, or saturation with, water, often higher temperature), biodegradation of organic components of interest may be unrealistically enhanced in the laboratory if preventive measures are not taken.

Long duration percolation experiments should be performed in the dark in order to avoid algal growth at any surface where there is light. Algae yield oxygen and consume nutrients, thus the soil column becomes more anaerobic than in the dark.

7.1.2.14 Specificity of organic compounds compared to inorganic compounds

The release of organic compounds should be assessed as well as that of inorganic compounds. But, for the time being, the leaching of organic contaminants from soil 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 that of inorganic contaminants. In addition, the properties of organic contaminants in relation to sorption on different materials in which they come in contact (e.g. bottles, filters) are different for organic contaminants than 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) may be more crucial.

Moreover, several steps should be avoided when handling the eluate since this may lead to losses of constituents due to sorption onto test equipment.

Therefore, special attention has been paid during the elaboration of the test procedures in ISO 21268 (all parts) to the scope, material to be used, potential degradation, volatilisation, adsorption, and separation (see 7.1.2.12). If both organic and inorganic contaminants shall be assessed, the most constraining requirements of each leaching test to be carried out shall be used.

7.1.3 Representativity and gaps between test conditions and field conditions

For practicable reasons, conditions reflecting the key factors described above are generally chosen or fixed in a laboratory leaching test away from the real conditions occurring in the field.

This is not critical when the influence of a given key factor is not of first importance on the release. If it is, however, when performing a leaching test in the framework of impact assessment, the extrapolation from laboratory to the field shall be taken into account. This can be determined when implementing the Case 1 approach.

Table 5 presents and justifies, for some selected key factors, the representativity and gaps between test conditions and field conditions. The effects on release of contaminants expected from these differences are also discussed.

Table 5 — Representativity and gaps between test conditions and field conditions

Test condition	Justification	Field condition	Effect
Temperature set at (20 to 22) °C due to normal laboratory conditions	It is not possible to simulate the real soil temperature evolution in the field, especially because it is often soil from the upper layer that is of interest, which is under the influence of climatic conditions.	Temperature varies both over a day and over the years.	A higher temperature increases the volatility of the organic compounds and enhances degradation. Temperature also has an effect on the kinetics of chemical reactions.
Biological degradation prevented by, for example, NaN_3 and darkness	It is not possible to simulate field conditions in a leaching test similar to how biological degradation could occur in the field. Depending on the country, temperature may be higher in the laboratory than in the field, which may increase degradation.	Most organic compounds are more or less degradable.	Biological degradation has an influence on organic content and characteristics, especially towards leaching. It can also affect the chemical speciation of inorganic elements, such as chromium and arsenic, thus influencing the release. Thus, in the field, the leaching from the soil may be lower than estimated by the leaching test due to biological degradation.
Release at equilibrium or local equilibrium	Laboratory tests aim at an equilibrium condition between soil and the leachant, for an easier interpretation of results.	Equilibrium between soil and a leachant is not always reached, and is generally different from the ones obtained under laboratory conditions.	Laboratory conditions do not simulate field situations, meaning that laboratory results shall not be directly used for prediction. The release of organic compounds in the field may deviate from that measured in the laboratory, if equilibrium conditions are not approached either in the laboratory (underestimation) or in the field (overestimation).
Saturated leaching conditions	To be able to study the dynamic of release, a leaching test shall be conducted under controlled conditions, and water-saturated leaching can be controlled much more easily than unsaturated leaching.	In the field, the leaching may be unsaturated with wet and dry periods and the influence of atmospheric conditions (e.g. oxidation, carbonation).	The effect is unknown. The largest effect would be expected on volatile compounds. However, wet and dry periods could probably also cause significant changes in the leached concentrations of non-volatile organic compounds. Lysimeter leaching experiments may be useful for the investigation of this topic.

Table 5 (*continued*)

Test condition	Justification	Field condition	Effect
Application of reducing conditions	In the laboratory, test conditions are often oxidising (e.g. contact with the atmosphere, or use of demineralised water or 0,001 M $CaCl_2$).	Different stages of redox conditions may occur in the field, depending especially on the depth of the soil layer under consideration.	For organic compounds, changes in redox conditions mainly affect the biological processes. If biological activity is prevented, changes in redox conditions do not affect leaching significantly. However, generally, desorption of most compounds is not significantly sensitive to redox changes. For inorganic elements of which release is speciation-dependent, applying reducing conditions may lead to significant changes in release.
Grain size	In the laboratory, the grain size is generally defined in order to optimise the reproducibility of the test.	In the field, the diameter of particles is not controlled and, generally, oversize particles are present.	It is generally accepted that, as contaminants are associated with fine particles, laboratory tests may generate a higher release.

7.1.4 Beneficial use of results

7.1.4.1 Parametric and multiparametric tests (Case 1 approach)

The test results of ISO/TS 21268-3 and ISO/TS 21268-4 can be used to provide information and input data in predicting the release of constituents from soil in a specific scenario, as a function of time. Such a prediction can be preferentially done by means of modelling under the appropriate exposure conditions of the scenario. These exposure conditions can be one of, or a combination of, the following: hydraulic, geotechnical, hydrological, chemical, physical and biological. As a part of a complete characterisation of the leaching behaviour of soil under specified conditions, one test cannot generally be used alone: the application of several test methods may be required (see 8.2).

The up-flow percolation test (see ISO/TS 21268-3) provides the evolution of constituents' concentrations as a function of the L/S ratio under continuous dynamic conditions.

Under certain conditions, the test may also provide information on pore water concentrations in scenarios in which soil and aqueous solutions are in equilibrium, both in the short and longer term (after wash-out of the labile compounds), if the modelling step described in the Case 1 approach (see Clause 5) is applied.

Results from the up-flow percolation test (see ISO/TS 21268-3) may be comparable to those obtained by carrying out the batch compliance tests (see ISO/TS 21268-1 and ISO/TS 21268-2), either at L/S = 2 l/kg or at L/S = 10 l/kg. However, users shall be careful as the sequence of leaching events in the dynamic percolation test may influence the kinetic-limited release of some constituents, which may lead to end results different from those obtained under the static conditions of a batch test, such as ISO/TS 21268-1 and ISO/TS 21268-2.

The pH dependence test (see ISO/TS 21268-4) provides the following information.

- The evolution of the soil response, in terms of pH, as a function of the quantity of acid or base added. This information allows the determination the ANC (Acid Neutralisation Capacity) or BNC (Base Neutralisation Capacity) of the soil. The ANC or BNC is defined as the amount of acid or base (± mol H^+/kg dry matter) needed to reach a given user-defined end-pH. It is graphically or numerically derived from a curve representing each end-pH obtained as a function of the amounts of acid or base added.

NOTE An environmentally relevant end-pH is 7, the neutral pH.

- The evolution of the released concentration of the soil constituents as a function of the pH (reached after the addition of a certain quantity of acid or base).

Tests that allow studying the influence of pH on leaching properties, as in ISO/TS 21268-4, are also useful for the following purposes.

- To identify the chemical behaviour trends and availability approaching levels of components at different pH values under the experimental conditions specified in this test. These values can be used as input for modelling of chemical behaviour using geochemical speciation models. In many cases, it also provides insight into the relevance of particular solubility controls and release mechanisms (e.g. formulate a hypothesis on the dissolution mechanisms).
- To provide data to feed dynamic behavioural models, for instance under the following relationship: solubilisation = f(pH or meq H^+/g) in the physico-chemical context linked with the presence of the other compounds in the material. This is not always possible with available literature data.

On the contrary, this kind of test is not meant for the following.

- Quantifying a maximum removable fraction as the concentrations obtained correspond to a steady state situation close to chemical equilibrium. For example, the values obtained for the lowest pH can only be considered as an approach to the maximum removable fraction.
- Simulating actual situations in specific scenarios.

For these kinds of basic characterisation tests, a direct use of test results, such as multiplication by a factor to extrapolate from laboratory scale to field scale, is generally not possible. Upscale extrapolation using the L/S ratio is generally only possible for, and shall thus be reserved for, easily soluble constituents such as sodium and chlorides. For other constituents, for which release is kinetically or chemically controlled, upscale extrapolation is only possible through combined geochemical and transport modelling.

7.1.4.2 Compliance and quality control tests (Case 2 approach)

The test results obtained with the compliance tests specified in ISO/TS 21268-1 and ISO/TS 21268-2 only allow a direct comparison with regulatory limits on a pass/fail basis, or with data previously obtained with the same test in a quality control process.

7.2 Large-scale columns and lysimeter

Experiments with a large soil column (e.g. diameter $>$ 10 cm and length $>$ 40 cm) conducted with a down-flow supply of leachant can provide substantial information on both the release of contaminants (source term) and sorption, transformation and binding processes (fate) in the unsaturated zone. In contrast to leaching tests with small columns (e.g. diameter $<$ 10 cm and length $<$ 40 cm), abiotic and biotic reactions occur, allowing a more realistic investigation of the transfer processes of contaminants from the source towards the target. Large soil columns are run for three to nine months, simulating moderate water fluxes which are more or less comparable to natural conditions. This means that contaminants will stay for a longer time in the column. By analysis of the different leachate fractions and of different soil layers at the end of the study, essential data for the modelling transport and fate of contaminants in the unsaturated zone are generated. By using isotope techniques (e.g. ^{14}C or ^{15}N), information on the formation of bound residues or the mineralisation of contaminants can be obtained.

Lysimeters are undisturbed soil cores of a surface of about 1 m^2 and a depth of 1,0 m to 2,0 m representing a small section of a field or contaminated area. They are , for instance, routinely used as a higher tier testing in pesticide registration, when laboratory leaching tests and subsequent modelling cannot exclude a potential for groundwater contamination. Large-scale outdoor lysimeters are integrative test systems providing information on all topics listed in 5.1. The test design of lysimeters as a validation study (see 5.1.7) is based on the existing information on laboratory studies, that is on basic information on leaching and degradation/metabolism. The advantages/challenges and limitations of lysimeter studies are summarised in Table 6. As lysimeters are fully monitored or controlled systems including the recording of exposure conditions (e.g. in an outdoor configuration: climatic data and rainfall), a modelling, and corollary identification, of the processes is possible.

By modelling, some generalisation of results is possible, e.g. modelling for other climatic conditions. Comparison of results obtained by large-column studies with disturbed soil cores and by lysimeter experiments can help elucidate the difference between maximum release caused by disturbing the soil, e.g. by excavation activities and the site-specific release and transport. By outdoor lysimeter experiments the physio-chemical and biological interactions can be studied by comparing the concentrations of the contaminants after specific events, for example the concentration in the first leaching in autumn after a longer dry period and the concentration profile during a longer rain period. However, lysimeters of 1,0 m^2 surface are too small for elaborating valid data for preferential flow processes, as 1,0 m^2 is too small for full-scale representative results.

As radioactive test substances can be applied to lysimeters, information on irreversible sorption, formation of bound residues, inhomogeneous transport and distribution in the soil profile and a mass balance can be obtained. In addition lysimeters can be cultivated for the elaboration of data for plant uptake.

Table 6 — Advantages and limits of lysimetric experimentations compared to laboratory testing

Advantages/challenges	Limitations
— outdoor, realistic exposure conditions — indoor, fully controlled system — undisturbed soil pore system — contaminants in their original position, e.g. lumps, reduced availability, hydrophobic spots — application of radio-labelled test substances — integrative system ⇒ long-term release in combination with leaching, degradation and fixation processes — sampling of total leachate ⇒ mass balance — at the end of the study, analysis of the different soil segments of the lysimeter — modelling of the processes and generalisation of results — validation of leaching as a function of time (see 5.1.7)	— high costs: not for routine testing — applicable for unsaturated zone; lysimeter can be taken up to a depth of approximately 3,0 m to 4,0 m — the subsoil should be a sandy to loamy soil; stones of a diameter of approximately 10 cm are tolerable

Table 7 gives a rough estimate of the time needed to perform the different tests.

Table 7 — Estimation of the time needed to perform different leaching tests

Test	Batch test	Percolation test	Large column test	Lysimeter
Duration	1 day	3 weeks to 4 weeks	3 months to 9 months	2 years to 3 years

8 Example: how to use leaching test results to assess the impact of soil on groundwater

8.1 General

Groundwater impact assessment is currently based on a three-level approach as presented in the introduction.

- Source: assess the release, identify speciation of constituents and retention mechanisms in the soil.
- Receptor: determine where and when the groundwater quality is affected or where and when a groundwater quality objective is exceeded.
- Pathway: estimate the transfer of the source towards the target.

Leaching tests are used to characterise the source.

As it is not realistic to routinely perform an *in situ* experiment to evaluate what could be the impact of the release of constituents from soil under exposure conditions such as those referred to in typical situations presented in 5.1.3.2, a modelling step is necessary.

To ensure sufficient confidence in modelling results, validation shall be performed at least for the source term and pathway modelling.

The determination of the leaching behaviour shall be done according to the methodology described in 8.2. The results of this determination are a modelled release that can be subsequently used as an input for transfer assessment.

8.2 Use of leaching behaviour determination in subsequent transfer and impact assessment

Transfer of elements released from the source (determined according to 5.1) towards the target is done by infiltration of water (e.g. due to rainfall) in the unsaturated zone and by groundwater circulation in the saturated zone (also called dispersion).

NOTE 1 In clay soils, the infiltration of water can occur by diffusion or by preferential flow in cracks.

An important aspect, that has a major influence on transfer modelling is the attenuation caused by interaction between the constituents released from the soil and the underlying subsoil. This attenuation is often expressed in a simplified way in terms of adsorption/desorption coefficients, the Kd values. Kd is the ratio between the concentration of an element in the solid phase (soil) and the concentration of the same element in the liquid phase (leachate or groundwater) at equilibrium conditions. Retardation in the transfer of this element through the soil layer increases with increasing Kd. Some constituents (e.g. Cl) are not retained in the underlying soil (Kd = 0). Kds may vary quite substantially from one element to another, from one type of soil to another and also as a function of soil pH. Thus, the use of these coefficients shall be made with precaution, especially when extrapolating from one soil, or one element, to another. In view of the wide range in Kd values for different soils, the selection of Kds for the modelling is usually made in such a manner that conservative values are used for all parameters.

It shall be understood that using a Kd concept does not take into account the mutual interaction of elements and competition for the same sorption sites. This can only be achieved with a full thermodynamic model. There are very few models that can deal with full mineral speciation, Fe- and Al-oxide sorption and, on top of that, interaction with dissolved and particulate organic matter. However, such models can provide a much more realistic description of actual practice.

Recent developments are leading to user-friendly models to deal with the required complexity. For organic constituents, modelling still remains a field of research.

The hydrological situation is also important as it determines the impact. The following characteristics shall at least be taken into account to assess the transfer of elements into soil and groundwater:

- net infiltration amount (e.g. 25 % to 30 % per year under oceanic conditions in a temperate climate);
- groundwater flow velocity; it shall be assumed to allow transport to a point of compliance outside the affected area (a reasonable estimate is 15 m/y in Western Europe);

NOTE 2 A point of compliance is a point located in the soil or in the groundwater chosen as a reference where a given water quality objective is to be met.

- pore flow velocity.

The final result of this modelling approach is to forecast possible concentrations of given contaminants in groundwater in a given time frame. Those concentrations are generally compared to available water quality criteria.

Annex A
(informative)

Schematic representation of a contaminated site with relevant targets

Figure A.1 gives a schematic representation of a contaminated site, where the process, influencing factors and targets are illustrated. A major distinction can be made between the source term and the means to assess the release to the subsoil, as a function of time, under the influence of varying controlling factors with time and the transport of contaminants from the boundary of the site to subsoil and from groundwater to an identified target.

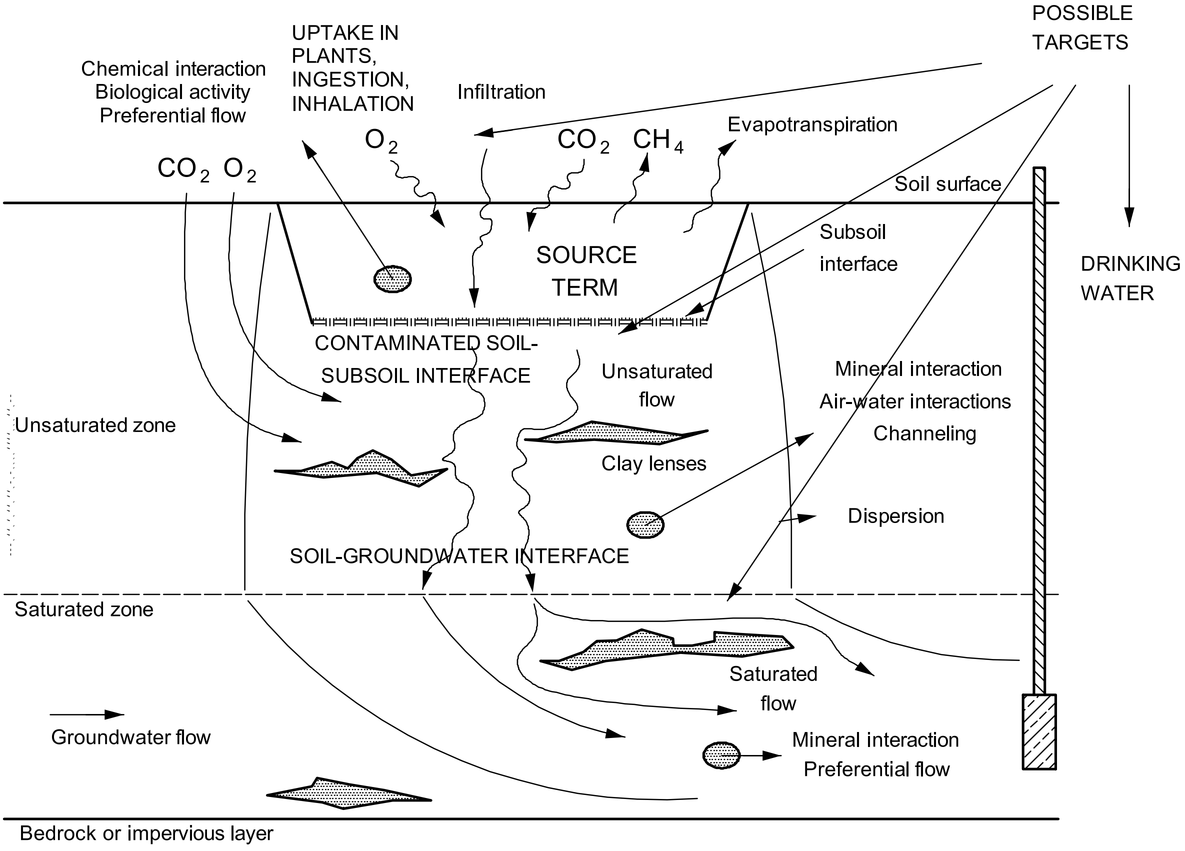


Figure A.1 — Schematic representation of a contaminated site with relevant targets for judgement

Annex B
(informative)

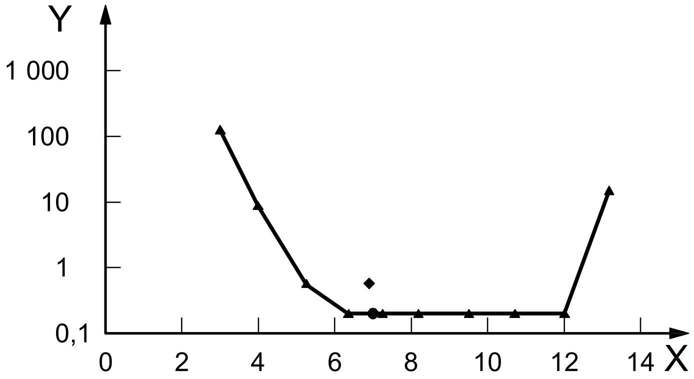
Comparison at different scales of testing (laboratory, lysimeter and field scale)

For assessing release to subsoil and groundwater, testing at different scales is crucial, as the test results obtained in the laboratory cannot be used directly in predicting what will happen in the field. This relates to other infiltration conditions (e.g. saturated versus unsaturated), exposure to the atmosphere, preferential flow aspects, etc. This is largely described in Clause 5.

In the framework of a Dutch national research project on sustainable landfill, laboratory experiments (percolation test — CEN/TS 14405; pH dependence test — CEN/TS 14429; these two tests have been used as a basis to develop ISO/TS 21268-3 and ISO/TS 21268-4 respectively), lysimeter studies (1,0 m^3 to 1,5 m^3) and a 12 000 m^3 pilot demonstration project at landfill site Nauernasche Polder (the Netherlands) were carried out in conjunction with chemical speciation modelling and release modelling. The material studied consisted mainly of soil-cleaning residues, sediments, contaminated soil and some minor industrial waste streams. This implies that the character of this mixture is very much soil-like in nature.

The filling of the 12 000 m^3 pilot was completed in November 2001. The test cell is isolated from the rest of the landfill site by a HDPE (high density polyethylene) membrane. Leachate is collected in the lower corner of the test cell, and the amount of leachate pumped out of the test cell is measured. L/S is determined from the ratio of collected leachate versus the volume of the cell. Samples were taken from all material deposited in the cell and the weight of each stream was recorded. From all samples collected, an integrated mix was prepared by taking the mass per charge into account. This mix was used for the laboratory testing according to CEN/TS 14405 and CEN/TS 14429 and for filling three lysimeters with a representative mixture (October 2001). The filling of the lysimeter to 1,5 m^3 was carried out in order of delivery, as practised at Nauerna. The studies at the field, lysimeter and laboratory scale represent different time scales through the liquid to solid ratio to which the mix was exposed.

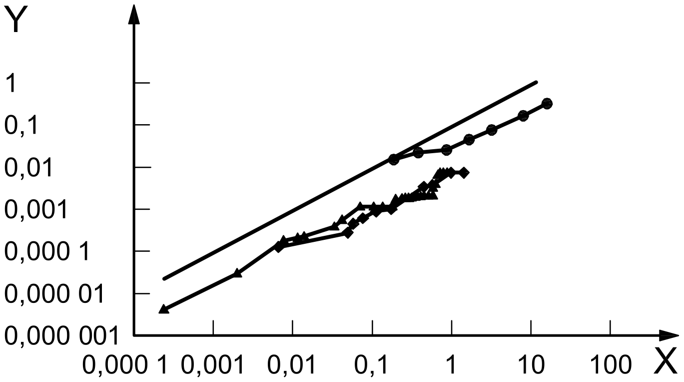
The mass of all materials delivered to the pilot cell was recorded. This implies that, by constituting the mix in proportion to the mass input, a rather good balance of all constituents in the pilot cell is obtained. A comparison between the release of mobile constituents (Cl, Na, K) in lysimeters and field leachate with the column-leaching test data obtained with up-flow (minimal channelling) allowed conclusions to be drawn on the possible role of preferential flow. In fact, it was found that, under usual infiltration conditions, about 75 % of the material was not effectively leached.



a)

Key

—●— material mix + organic rich material
—▲— mix of predominantly soil material
—◆— material mix + sewage sludge
X pH
Y release (mg/kg)



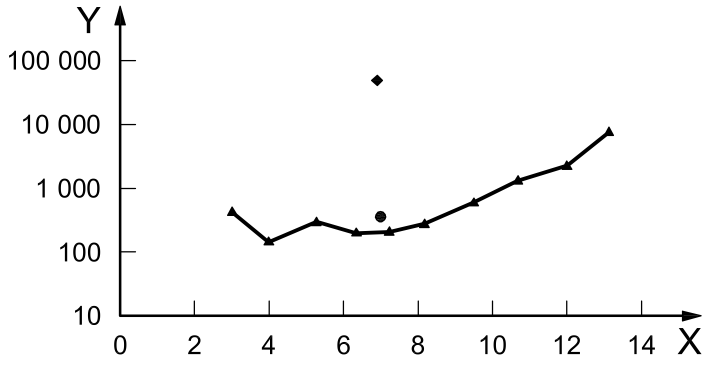
b)

Key

—●— laboratory test of material mix 0,000 5 m^3
—▲— lysimeter study 1,5 m^3
—◆— pilot study 12 000 m^3
—— slope = 1,0
X L/S (l/kg)
Y cumulative release (mg/kg)

Figure B.1 — Release curves of lead as a function of pH (left plot) and as a function of L/S ratio (right plot)

In Figures B.1 and B.2, the data for DOC and lead from laboratory testing and leachate analysis in lysimeters and the field are combined. The leaching behaviour of lead from the mix is consistent with observations on soil. The resultant integral mix behaves very consistently and, judging from the slope in the release L/S plot, its behaviour is controlled by solubility. The end-point of the column test at L/S = 10 matches very well with the corresponding leached amount at L/S = 10 in the pH-dependence test at the appropriate pH (around 7). The agreement between the different levels of testing (laboratory-lysimeter-field scale) indicates solubility control. If solubility control can be demonstrated, prediction of long-term behaviour and identification of possible stresses at long term are quite feasible. It was shown that DOC is the solubility-controlling phase (see Figure B.2).



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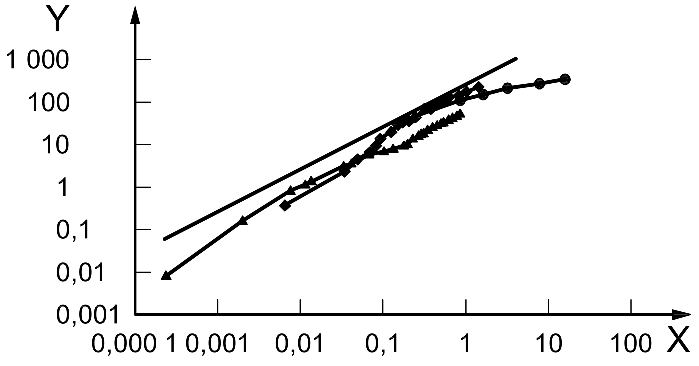
- —▲— mix of predominantly soil material
- —◆— material mix + sewage sludge
- X pH
- Y release (mg/kg)



b)

Key

- —●— laboratory test of material mix 0,000 5 m^3
- —▲— lysimeter study 1,5 m^3
- —◆— pilot study 12 000 m^3
- —— slope = 1,0
- X L/S (l/kg)
- Y cumulative release (mg/kg)

Figure B.2 — Release curves of Dissolved Organic Carbon (DOC) as a function of pH (left plot) and as a function of L/S ratio (right plot)

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