BS EN ISO 12782-5:2012

BSI Standards Publication

Soil quality — Parameters for geochemical modelling of leaching and speciation of constituents in soils and materials

Part 5: Extraction of humic substances from aqueous samples (ISO 12782-5:2012)

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The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Soil quality - Parameters for geochemical modelling of leaching and speciation of constituents in soils and materials - Part 5: Extraction of humic substances from aqueous samples (ISO 12782-5:2012)

Qualité du sol - Paramètres pour la modélisation géochimique de la lixiviation et de la spéciation des constituants des sols et des matériaux - Partie 5: Extraction des substances humiques des échantillons aqueux (ISO 12782-5:2012)

Bodenbeschaffenheit - Parameter zur geochemischen Modellierung der Elution und Speziation von Bestandteilen in Böden und Materialien - Teil 5: Extraktion von Huminstoffen aus wässrigen Proben (ISO 12782-5:2012)

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Foreword

This document (EN ISO 12782-5:2012) has been prepared by Technical Committee ISO/TC 190 "Soil quality" in collaboration with Technical Committee CEN/TC 345 “Characterization of soils” the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2012, and conflicting national standards shall be withdrawn at the latest by December 2012.

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Endorsement notice

The text of ISO 12782-5:2012 has been approved by CEN as a EN ISO 12782-5:2012 without any modification.

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Foreword

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

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

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ISO 12782-5 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

ISO 12782 consists of the following parts, under the general title *Soil quality — Parameters for geochemical modelling of leaching and speciation of constituents in soils and materials*:

— *Part 1: Extraction of amorphous iron oxides and hydroxides with ascorbic acid*

— *Part 2: Extraction of crystalline iron oxides and hydroxides with dithionite*

— *Part 3: Extraction of aluminium oxides and hydroxides with ammonium oxalate/oxalic acid*

— *Part 4: Extraction of humic substances from solid samples*

— *Part 5: Extraction of humic substances from aqueous samples*

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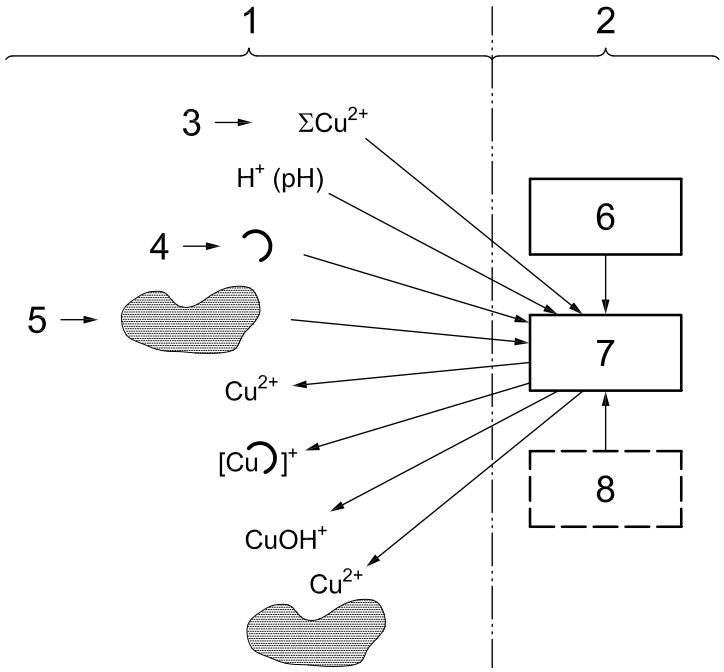
Introduction

In addition to leaching procedures for subsequent chemical and ecotoxicological testing of soil and other materials including waste, predictive models are becoming indispensable tools in the environmental risk assessment of these materials. Models are particularly required when the results of laboratory leaching tests are to be translated to specific scenarios in the field, with regard to assessing the risks of both contaminant migration and bioavailability.

In the past few years, geochemical models have been shown to be valuable tools to be combined with the data obtained from characterization leaching standards, such as pH-dependence and percolation tests. These models have the advantage of being based on fundamental thermodynamic parameters that have a general validity. In order to enable extrapolation of laboratory leaching data to the mobility and/or bioavailability of a constituent in a specific field scenario, these models require additional input parameters for specific soil properties (see Figure 1).



Key

1 experiment
2 geochemical speciation modelling
3 available metal concentration
4 dissolved humic substances
5 reactive (solid) surfaces
6 database with stability constants
7 computer program
8 assumptions

Figure 1 — Relationships between experimental data, as obtained from laboratory leaching/extraction tests, and geochemical modelling of the speciation of a heavy metal in the environment (modified after M. Gfeller & R. Schulin, ETH, Zürich)

Characterization leaching standards provide information on the concentrations of the contaminant of interest as a function of, in particular, pH and liquid/solid (L/S) ratio. In addition, a more complete analysis of the leachates also provides information on the major ion composition and dissolved organic carbon (DOC), parameters that are particularly important for the chemical speciation of constituents through processes such as precipitation, complexation and competition for adsorption on reactive mineral and organic surfaces in the soil. As illustrated

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in Figure 1, for the example of copper, geochemical modelling enables calculation of the metal distribution among these different chemical species in the system of interest. This provides necessary information for risk-assessment purposes, as these different chemical forms play distinct roles in the mobility and bioavailability of the metal in the soil. In addition to information obtained from the leaching standards (in their current state of development/definition), two additional types of information are required.

a) The “available” (sometimes also referred to as “active” or “exchangeable”) concentration of the constituent in the solid phase, as opposed to the total concentration determined by acid destruction of the solid matrix. This “available” concentration can be obtained by leaching at low pH, a condition that can be obtained by extending the pH range in the pH-dependent leaching test (ISO/TS 21268-4) down to pH ≈ 0,5 to pH ≈ 1.

b) The concentration of reactive organic and mineral surfaces in the soil, which constitute the major binding (adsorption) sites for most constituents in the soil matrix.

The major reactive surfaces that control the binding of constituents by sorption processes to the soil matrix are particulate organic matter and iron and aluminium (hydr)oxides. It is generally accepted that the reactivity of these mineral and organic surfaces can strongly vary as a function of their specific surface area/crystallinity [iron and aluminium (hydr)oxides] and composition (organic matter). When the results are intended to be used for the above-described purposes of geochemical modelling in conjunction with leaching tests, it is important that the methods be selective for reactive surfaces for which generic thermodynamic adsorption parameters are also available for the most important major and trace elements.

These reactive surfaces have been identified in soils, as well as in a variety of other materials for which the leaching of constituents is of relevance. It has been shown that the binding properties of these surfaces play a generic role in the speciation and leaching of constituents among these different materials. As an example, a similar geochemical modelling approach, using model input from the partial or complete ISO 12782 series, has been successfully applied to different soils[4], amended soils[5][6], municipal incinerator bottom ash[7], steel slag[8][9], bauxite residues[10], and recycled concrete aggregate[11]. Hence, the scope of the ISO 12782 series extends from soils to materials including soil amendments and waste materials.

This part of ISO 12782 aims to determine important reactive organic surfaces in soil and materials, for which generic thermodynamic adsorption parameters exist, i.e., humic and fulvic acids. The procedure is based on Reference [12], while generic thermodynamic adsorption parameters for humic and fulvic acids are available in References [13] and [14].

Thermodynamic parameters for adsorption models other than those used in References [13] and [14] are also available in the literature and may also be used to model the binding of constituents to humic and fulvic acids.

The method[15] is based on a conventional isolation and purification method[12] that is also used by the International Humic Substances Society (IHSS).

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Soil quality — Parameters for geochemical modelling of leaching and speciation of constituents in soils and materials —

Part 5: Extraction of humic substances from aqueous samples

1 Scope

This part of ISO 12782 specifies a procedure to determine the concentration of humic substances in aqueous samples. These samples may be obtained as such or as eluates from leaching procedures applied to soil or other materials. Other materials also include waste. The content of humic substances can be used as input in geochemical models.

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 8245, *Water quality — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

3 Terms and definitions

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

3.1
dissolved organic carbon
DOC
sum of organically bound carbon present in water originating from compounds (including cyanate and thiocyanate) which will pass a membrane filter of pore size 0,45 µm

3.2
humic substance
HS
(partial) decomposition product from plant and animal tissue

NOTE 1 Humic substances form a series of relatively high-molecular-weight, brown-to-black-coloured substances formed by secondary synthesis reactions.

NOTE 2 The term is used as a generic name to describe coloured material or its fractions (e.g. humic and fulvic acids) obtained on the basis of solubility characteristics.

3.3
humic acid
HA
fraction of a humic substance that is not soluble in water under acidic conditions (pH <1 to 2) but is soluble at higher pH values

NOTE Humic acids are dark brown to black in colour.

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3.4
fulvic acid
FA
fraction of a humic substance that is soluble in water under all pH conditions

NOTE 1 Fulvic acids remain in solution after removal of humic acid by acidification.

NOTE 2 Fulvic acids are light yellow to yellow-brown in colour.

3.5
hydrophilic organic carbon
Hy
organic carbon compound consisting of non-humic and humic-like substances

NOTE In this part of ISO 12782, Hy is essentially regarded as the extractable organic carbon fraction that is not identified as humic acid, fulvic acid or hydrophobic neutral organic carbon in accordance with the procedure specified in Clause 8. Hydrophilic organic carbon generally consists of molecules with a lower molecular weight and higher COOH/C ratios than humic acids and fulvic acids. Examples of compounds are: oxidized carbohydrates with carboxylic acid groups, low-molecular-weight carboxylic acids, and sugar phosphates.

3.6
hydrophobic neutral organic carbon
HON
difference between the amount of adsorbed fulvic acid and hydrophilic organic carbon and the amount of desorbed fulvic acid

NOTE Hydrophobic neutral organic carbon can include non-humic and humic-like compounds.

3.7
laboratory sample
sample intended for laboratory inspection or testing

[ISO 11074:2005]

3.8
test sample
sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis; this portion of material, resulting from the laboratory sample by means of an appropriate method of sample pretreatment, and having the size (volume/mass) necessary for the desired testing or analysis

NOTE Adapted from ISO 11074:2005.

3.9
test portion
analytical portion
quantity of material, of proper size, for measurement of the concentration or other property of interest, removed from the test sample

NOTE 1 The test portion may be taken from the primary sample or from the laboratory sample directly if no preparation of sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

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

[ISO 11074:2005]

3.10
soil material
excavated soil, dredged material, manufactured soil, treated soil and fill material and other relevant materials, including soil amendments and waste materials

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