

**Sludge, treated biowaste and soil - Guidance for sample pretreatment**

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## EESTI STANDARDI EESSÕNA

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Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 22.08.2012.	Date of Availability of the European standard is 22.08.2012.
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ICS 13.030.01, 13.080.05

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ICS 13.080.05; 13.030.01

English Version

## Sludge, treated biowaste and soil - Guidance for sample pretreatment

Boues, bio-déchets traités et sols - Lignes directrices pour le prétraitement des échantillons

Schlamm, behandelter Bioabfall und Boden - Anleitung zur Probenvorbehandlung

This European Standard was approved by CEN on 23 June 2012.

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## Foreword

This document (EN 16179:2012) has been prepared by Technical Committee CEN/TC 400 "Project Committee - Horizontal standards in the fields of sludge, biowaste and soil", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Introduction

This document has been developed upon existing International Standards for the pretreatment of soils of specified particle fractions.

Historically this has been the case for analysing organic compounds after pretreatment according to e.g. ISO 14507. Standards describing pretreatment for chemical and physico-chemical parameters, e.g. ISO 11464, historically have divided the samples into fractions < 2 mm and > 2 mm where the fraction < 2 mm was taken for testing. By this the concentrations reported for organic compounds could be related to another part of the sample than those of the chemical and physico-chemical parameters. This European Standard stems on the assumption that the same part of the original sample is used for all parameters to be analysed. For environmental investigation it is assumed that generally the whole sample is of interest and will be pre-treated. Only extraneous materials may need to be removed under specific circumstances (and usually then will be reported accordingly).

Depending on legislative or other demands only specific fractions of the sample may be analysed (e.g. the fraction < 2 mm). In that case, the sample will be sieved prior to the pretreatment and the laboratory will report that only the fraction < 2 mm was analysed.

The pretreatment procedures described in this European Standard are not applicable if they affect the results of the determinations to be made. For example, the properties of the parameters to be analysed may differ greatly depending on chemical species:

- they can range from non-volatile to very volatile compounds (low to high vapour pressure);
- they may be labile or reactive at ambient or elevated temperatures;
- they may be biodegradable or UV-degradable;
- they may have considerable different solubilities in water.

Some properties of chemical species require different analytical procedures.

Because of these differences, it is not possible to specify one general pretreatment procedure to fit all materials and goals of investigation. The aim of a pretreatment procedure is to prepare a test sample of which the content of a substance or a characteristic is equal to the original material, provided that the applied pretreatment procedure does not considerably alter the characteristic or the chemical nature of the substance to be analysed. It should be noted that every type of pretreatment will have an influence on several material properties.

Important for both sampling and pretreatment are the particle size distribution and form and the degree of chemical heterogeneity of the sample in relation to the minimum required mass of the sample. In general it can be stated that the smaller the particle size and form, and the less the chemical heterogeneity of the original material, the less sample mass is required for a reliable test or – the other way around – the coarser the particle size or the greater the range of particle size and forms, and the greater the chemical heterogeneity might be, the bigger the (sub)sample mass needs to be in order to perform a reliable test. Clause 5 and 8.3 deal with this subject.

**WARNING** — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

**IMPORTANT** — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

## 1 Scope

This European Standard specifies the pretreatment required for sludge, treated biowaste and soil (including soil-like materials), that are subject to the analysis of organic as well as inorganic chemical and physico-chemical parameters.

The pretreatment of samples aims at preparing a (small) test sample which is representative for the original sample.

This European Standard describes the pretreatment which could be performed under field conditions if necessary (see Clause 8) and the sample pretreatment under laboratory conditions (Clause 10).

For determining inorganic chemical and physico-chemical parameters this European Standard describes procedures (see 10.2) to prepare:

- test samples for tests under field moist conditions;
- test samples for testing after drying, crushing, grinding, sieving etc.;
- test samples of liquid sludge.

For determination of organic compounds three pretreatment methods are specified:

- a pretreatment method if volatile organic compounds are to be measured (see 10.3.2);
- a pretreatment method if moderately volatile to non-volatile organic compounds are to be measured and the result of the following analysis will be accurate and reproducible (see 10.3.3);
- a pretreatment method if moderately volatile to non-volatile organic compounds are to be measured and the extraction procedure prescribes a field moist sample or if only indicative results are required (see 10.3.4).

The choice of the method depends above all on the volatility of the analyte. It also depends on the particle size distribution of the material (see Clause 5 and 8.3), the heterogeneity of the sample and the following analytical procedure.

## 2 Normative references

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

EN 15933, *Sludge, treated biowaste and soil — Determination of pH*

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges (ISO 5667-13)*



EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15)*

EN ISO 16720, *Soil quality — Pretreatment of samples by freeze drying for subsequent analysis (ISO 16720)*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 10381-8, *Soil quality — Sampling — Part 8: Guidance on sampling of stockpiles*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

### 3 Terms and definitions

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

#### 3.1

##### **composite sample**

average sample

aggregated sample

two or more increments/subsamples mixed together in appropriate proportions - either discretely or continuously (blended composite sample) - from which the average value of a desired characteristic may be obtained

[SOURCE: ISO 11074:2005, 4.3.3]

#### 3.2

##### **extraneous material**

materials not belonging to the matrix or particle fraction to be analysed

Note 1 to entry: Due to the variety of the matrices covered by this European Standard the definition of extraneous material is very broad. The decision to remove certain parts of the material depends on the analytical task.

#### 3.3

##### **field sample**

quantity (mass or volume) of material obtained through sampling without any subsampling

[SOURCE: EN 14899:2005, 3.3]

#### 3.4

##### **increment**

sampling unit collected by a single operation of a sampling device and being used in a composite sample

Note 1 to entry: When an individual portion of material is collected in a single operation of a sampling device and this portion is analysed as an individual unit, it is by definition a sample.

[SOURCE: ISO 11074:2005, 4.1.8]

#### 3.5

##### **laboratory sample**

sample intended for laboratory inspection or testing

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or analysis.