SLOVENSKI STANDARD
SIST EN 16179:2013

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Blato, obdelani biološki odpadki in tla - Navodilo za pripravo vzorcev

Sludge, treated biowaste and soil - Guidance for sample pretreatment

Schlamm, behandelter Bioabfall und Boden - Anleitung zur Probenvorbehandlung

Boue, biodéchet traité et sol - Usure par prétraitement des échantillons

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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

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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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.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes.

[SOURCE: ISO 11074:2005, 4.3.6]

3.6
maximum particle size
D_{95}
particle size that concurs with the mesh width of a sieve on which a maximum of 5 % (mass fraction) of the material remains

3.7
particle size reduction
grinding or crushing the sample in order to reduce the particle size of the whole (sub)sample without reducing the sample size (mass)

[SOURCE: ISO 11074:2005, 4.6.4]

3.8
sample
portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be described in the sampling plan.

[SOURCE: ISO 11074:2005, 4.1.16]

3.9
sample pretreatment
collective term for all procedures used for conditioning a sample to a defined state which allows subsequent examination or analysis or long-term storage

Note 1 to entry: Sample pretreatment includes, e.g. grinding, mixing, splitting, drying, crushing, stabilization.

3.10
sampling plan
predetermined procedure for the selection, withdrawal, on-site pretreatment, preservation, transportation and preparation of the portions to be removed from a population as a sample

[SOURCE: ISO 11074:2005, 4.1.22]

3.11
subsample
sample taken from a sample of a population

Note 1 to entry: A subsample may be:

a) portion of the sample obtained by selection or division;

b) an individual unit of the lot taken as part of the sample;

c) the final unit of multistage sampling.

Note 2 to entry: The term 'subsample' is used either in the sense of a 'sample of a sample' or as synonym for 'unit'. In practice, the meaning is usually apparent from the context or is defined.

[SOURCE: ISO 11074:2005, 4.1.30]

3.12
subsampling
sample division
process of selecting one or more subsamples from a sample of a population

[SOURCE: ISO 11074:2005, 4.6.9]

3.13
test sample
analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

3.14
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 to entry: The test portion may be taken from the field sample or from the laboratory sample directly if no preparation of the sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

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

[SOURCE: ISO 11074:2005, 4.3.13]

4 Safety remarks

Special precautions should usually be taken for samples from contaminated material. It is important to avoid any contact with the skin and special measures should be taken when drying such samples (ventilation, air removal, etc.). Samples may be hazardous because of the presence of chemical contaminants, fungal spores, or pathogens such as *leptospirosis*.

Appropriate national safety precautions shall be followed.

5 Principle

5.1 General

Pretreatment in this European Standard is the process of preparing the test portion from the field sample. The operations and treatment steps are divided into pretreatment procedures suitable in the field (Clause 8) and pretreatment procedures that are restricted to be performed in the laboratory (Clause 10).

It is important to note that the methods and instruments described are meant as examples for suitable routine procedures. A laboratory may decide to use other procedures and/or instruments as long as the requirements of the analytical task are met (see Clause 10).

Another important item concerns the part of the sample delivered to the laboratory which shall be taken for the determinations and the part of the sample which is removed before starting the tests. Depending on legislative or other demands only specific fractions of the sample may be analysed (e.g. the fraction < 2 mm). Extraneous materials shall be separated and recorded.

In case parameters are analysed in the fraction < 2 mm usually the results of the tests refer to the fraction < 2 mm. Using the mass fraction > 2 mm and the extraneous material removed from the sample the results also can be calculated referring to the mass of the whole sample. The report shall clearly state to which fraction the results refer.

Beside the requirements given in this European Standard, the subsequently applied analytical standards shall be regarded for particular requirements to be observed. The determination of some parameters requires sample pretreatment soon after sampling as specified in the respective analytical methods.

If several parameters are being investigated, the sample pretreatment shall be designed so that the parameters of major importance are determining the pretreatment. If this is not possible, e.g. the required precision for each parameter can not be achieved, separate pretreatment shall be set up for each group of parameters.

Whenever volatile compounds are to be determined, the process of sample pretreatment can result in a substantial loss of these compounds. Sample pretreatment shall be omitted in these cases by taking specific samples for the determination of volatile components. These samples shall be pretreated in accordance with the appropriate analytical standard and analysed as soon as possible after sampling.

When preparing composite samples the analytical requirements shall be considered. For example, composite samples are not appropriate if volatile compounds are to be determined.

Figure 1, Figure 2 and Figure 3 show flow charts for:

— pretreatment for inorganic and physico-chemical parameters in solid sludge, treated biowaste and soil (Figure 1),

— pretreatment for organic parameters in solid sludge, treated biowaste and soil (Figure 2),

— pretreatment for inorganic, physico-chemical and organic parameters in liquid sludge (Figure 3).

Aside from the parameter to be determined, the procedure to be applied also depends on the required minimum mass of the sample to be used in relation to the maximum particle size D_{95} of the sample. For this the relationship described in ISO 10381-8 is used (see Table 2 in 8.3).

Grinding of the sample depends on the mass of the subsample being taken. Table 1 gives the requirements for a wide group of parameters.

For some parameters the requirements stated in 8.3 need not to be followed exactly as the homogeneity of the samples is enough to obtain adequate results for those parameters.

EXAMPLES dry matter, total organic compounds (TOC), pH, EC.

Table 1 — Required maximum particle size (D_{95}) for the determination of several parameters.*(1 of 2)*

Parameter	Matrix[a]	Test portion on dry matter basis	D_{95} required, remarks	Procedure[b]
Ammonium- and nitrate-nitrogen	Solid	> 15 g	No D_{95} requirements, sample as received, no heating and no freeze drying allowed	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Anions (Cl^- , F^- , Br^- , PO_4^{3-} , SO_4^{2-})	Solid	> 15 g	No D_{95} requirements, sample as receive	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Adsorbable organic halogens (AOX)	All	5 mg to 100 mg	≤ 250 µm	Fig. 3
Cyanide	Solid	10 g to 40 g	No D_{95} requirements, sample as received, no heating allowed	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Dry matter fraction	Solid	30 g to 50 g	No D_{95} requirements	Fig. 1, Route A
	Solid, air dried	10 g to 15 g	No D_{95} requirements	
	Sludge	> 0,5 g	No D_{95} requirements	Fig. 3
Electrical conductivity	Solid	20 g	No D_{95} requirements, drying (optional)	Fig. 1, Route C
	Sludge		No grinding, direct measurement in liquid phase	
Loss on ignition (LOI) at 550 °C	All	< 0,2 g	≤ 250 µm	Fig. 1, Route A
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	No D_{95} requirements, dried sample for determining dry matter is taken	
Metals: see trace elements				
Organic matter: see LOI				
Particle size distribution			No pretreatment allowed	
pH	Solid	5 ml	No D_{95} requirements, drying (optional)	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in liquid phase	Fig. 3