SLOVENSKI STANDARD
SIST EN 15002:2006

01-maj-2006

Karakterizacija odpadkov – Priprava preskusnih vzorcev iz laboratorijskega vzorca

Characterization of waste - Preparation of test portions from the laboratory sample

Charakterisierung von Abfällen - Herstellung von Prüfmengen aus der Laboratoriumsprobe

Caractérisation des déchets - Préparation de prises d'essai a partir de l'échantillon pour laboratoire

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Characterization of waste - Preparation of test portions from the laboratory sample

Caractérisation des déchets - Préparation de prises d'essai à partir de l'échantillon pour laboratoire

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Foreword

This European Standard (EN 15002:2006) has been prepared by Technical Committee CEN/TC 292 “Characterisation of waste”, 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of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

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

Introduction

In laboratory praxis, very often different analytical procedures have to be applied to the laboratory sample that has been taken according to the sampling plan. For this purpose subsampling has to be applied in a way, that the different test portions are representative for the original laboratory sample with respect to the compounds of interest and the specific analytical procedures. The representativity of the laboratory sample and of the test portions is of major importance to guarantee the quality and accuracy of analytical results. The representativity of the laboratory sample is specified by the sampling plan. This European Standard specifies the correct sequence of operations to ensure the representativity of the test portions.

Safety remarks

Anyone dealing with waste and sludge analysis has to be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e.g. toxic, reactive, flammable and infectious) substances, which can be liable to biological and/or chemical reaction. Consequently it is recommended that these samples should be handled with special care. The gases that may be produced by microbiological or chemical activity are potentially flammable and will pressurise sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

1 Scope

This European Standard is applicable for the preparation of representative test portions from the laboratory sample that has been taken according to the sampling plan (EN 14899), prior to physical and/or chemical analysis (e.g. preparation of eluates, extractions, digestion and/or analytical determinations) of solid and liquid samples and sludge. It is also applicable for the preparation of test portions from digests and eluates for the subsequent analyses.

This European Standard is intended to find the correct sequence of operations and treatments to be applied to the laboratory sample in order to obtain suitable test portions in compliance with the specific requirements defined in the corresponding analytical procedures.

2 Normative references

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

ISO 11465:1993, *Soil quality – Determination of dry matter and water content on a mass basis – Gravimetric method*

ISO 14507:2003, *Soil quality — Pretreatment of samples for determination of organic contaminants*

3 Terms and definitions

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

3.1
sample
portion of material selected from a larger quantity of material

3.2
laboratory sample
sample sent to or received by the laboratory

NOTE 1 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 for analysis.

NOTE 2 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 Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

3.3
test sample; analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

3.4
test portion; analytical portion
quantity of material of proper size, for measurement of the concentration or other properties of interest, removed from the test sample

NOTE The test portion may be taken from the laboratory sample directly if no preparation of sample is required (e. g. with liquids or samples of proper homogeneity, size and fineness), but usually it is taken from the prepared test sample.

3.5
sub-sample
sample obtained by procedures in which the items of interest are randomly distributed in parts of equal or unequal size

NOTE A sub-sample may be:
a) portion of the sample obtained by selection or division;
b) the final sample of multistage sample-preparation.

3.6
fraction
sample obtained by procedures from the laboratory sample where the properties of interest may be unequally distributed

NOTE A fraction may consist of metal pieces, stones etc.

3.7
volatile organic compounds
organic compound having a boiling point below 300 °C (at a pressure of 101 kPa)

NOTE This includes volatile aromatic and volatile halogenated hydrocarbons as determined in accordance with ISO 15009. Some mono- and dichlorophenols, for instance, and naphthalene also belong to this group.

3.8
moderately volatile organic compounds
organic compound having a boiling point above 300 °C (at a pressure of 101 kPa)

NOTE This definition includes:
a) mineral oil;
b) most polycyclic aromatic hydrocarbons (PAH) (see ISO 13877);
c) polychlorobiphenyls (PCB) (see ISO 10382);
d) organochlorine pesticides (see ISO 10382).

3.9
moderately volatile compounds
sum of moderately volatile organic compounds and volatile inorganic compounds (e.g. mercury, arsenic cadmium, thallium) that can be lost during sample preparation (e.g. heating)

3.10
homogenisation
process of combining of components, particles or layers into a more homogeneous state of the original samples (in the case of composite samples) or pre-treated fractions of samples in order to ensure equal distribution of substances in and properties of the sample

3.11
phase separation; fraction separation
process of dividing components, particles or layers if homogenisation of the sample is practically not applicable and/or the analysis of different fractions or phases are appropriate

3.12
drying
process of removing water from a sample

NOTE For the purpose of test portion preparation, it may be useful to remove just the amount of water that could interfere with other processes involved (e.g. during crushing or milling). In order to minimise the alteration of the sample during test portion preparation, removing the total amount of water present in the sample is not necessarily needed.

3.13
particle size reduction
mechanical friction of the sample by milling, grinding, crushing or cutting

3.14
sub-sampling
process of selecting one or more sub-samples from a sample

4 Equipment

For the purpose of preparation of test portions from the laboratory samples appropriate equipment has to be chosen depending on the procedures selected according to Annex A.

In the selection of the type of treatment techniques, one should keep in mind that each of them has some potential impact on analytical results, because it can introduce contamination or alter the physical-chemical properties of the sample.

All glassware and devices that come in contact with the sample shall be made out of a suitable material, chemically compatible with the sample, selected in order to minimize contamination of samples (e.g. plastic materials for inorganic elemental analysis, quartz or glass for volatile and organic analytes). Care shall be taken to ensure a good cleaning, in order to avoid cross-contamination of samples.

An informative list of appropriate equipment for the sample treatment procedures is given in Annex C.

5 Interferences and sources of error

The (sub)-sample shall be re-homogenised after any operation that may have resulted in segregation of different sized particles.

Care should be taken to avoid loss of material and contamination of the sample via the air, by dust, by the use of the apparatus (e.g. from the ambient laboratory atmosphere or between samples stored or processed close to one another).

Three types of contamination could occur from the apparatus:

- abrasion;
- cross-contamination;
- chemical release.

It is recommended to perform treatment of waste material in a separate room used only for this purpose, especially crushing or sieving.

If the sample has a dust-like consistency or contains (semi)-volatile compounds, part of it may be lost and this may alter its physical-chemical properties.

6 Procedure

6.1 Key concepts

Preparation of the test portion can be a complex process, because of a number of factors: sample type and its physical state, amount of laboratory sample, type and number of determinations to be carried out etc. The prepared test portions shall satisfy the following requirements at the same time:

- each test portion shall be as representative as possible of the laboratory sample;
- the amount and the physical state (e.g. particle size) of each test portion have to comply with the requirements of the respective analytical technique;
- for each test portion, no losses of and no contamination with respective analytes of interest should occur.

The preparation of the test portions from the laboratory sample, that has been taken according to the sampling plan, is related to the requested analytical determinations. This means that, if needed, contact has to be established among all involved parties such as the sampler, the customer and the analytical laboratory to achieve the requirements of the standards to be used for the requested determinations.

The preparation of test portions in the laboratory will frequently involve a sequence of operations such as homogenisation, phase separation, drying, reducing particle size and sub sampling. Specific forms of these operations are described in A.2 to A.6, respectively. A number of decisions on the specific order of these operations for a particular laboratory sample have to be made. In some cases, the sequence of operations to be applied is rather straightforward, but in more complicated cases (e.g. when several determinations with different requirements have to be performed) it can be critical to choose the right sequence of such operations. For soil samples more specific procedures are described in ISO 11464 for inorganic parameters or in ISO 14507 for organic parameters.

In order to define the operations to be applied to a laboratory sample to produce one or more representative test portions, three main steps have to be considered:

— **Definition of analytical requirements**

  First, the requirements of analytical procedures of interest shall be defined:

  — what methods shall be used;

  — how many test portions are necessary;

  — quantity and properties of the test portions necessary for each analytical procedure;

  — preservation requirements (e.g. time frame, temperature, addition of reagents).

  NOTE 1 Indicative amounts of test portions and specific requirements of the analytical methods involved are given in Annex D. It is recommended to prepare at least five times the amounts needed as test portions for the tests.

— **Definition of sequence of operations**

  Then, the sequence of operations shall be defined according to the flow sheet (Figure 1), based on the properties of the laboratory sample and the requirements of the analytical procedures: each single operation of this sequence has to be considered like an independent module; available modules are:

  — phase/fraction separation;

  — drying;

  — particle size reduction;

  — homogenisation;

  — sub-sampling.

  NOTE 2 For practical reasons it is recommended to group the parameters in a way that test samples with similar requirements can be prepared for several parameters. The same test sample may be used for different parameters if it fulfils the necessary requirements.

  Frequently, different determinations have to be performed on the laboratory samples. In those cases, modules have to be combined and/or repeated to obtain sub-samples, finally resulting in different test portions. In order to define the actual sequence of operations to be applied to a given sample, the flow sheet (Figure 1) shall be used.

— **Choice of appropriate procedures**

According to the requirements of the respective analytical techniques and the properties of the sample the appropriate sample treatment technique has to be chosen within each module by following the instructions of Annex A. Instructions are given in this annex in which case a particular operation is appropriate to use.

6.2 Sequence of treatment techniques

The flow sheet in Figure 1 describes the procedure to enable decisions on the specific order of treatment operations for a particular laboratory sample in order to yield in representative test portions. It shall be applied on the starting laboratory sample and repeated on all sample fractions or sub-samples subsequently obtained during the preparation, in an iterative cycle until all analytical requirements are fulfilled.

If volatile compounds or moderately volatile compounds are parameters of interest this should be considered in the sampling plan and may result in separate samples. In case of a single laboratory sample special care has to be taken in order to avoid losses of the volatile compounds during homogenisation and/or reduction of particle size. A preliminary sub-sampling without any homogenisation step may be necessary (see A.2.4, A.6.4) if the representativity of the remaining sample is not substantially altered.

NOTE In special cases sub-sampling without a drying step will not lead to representative sub-samples.

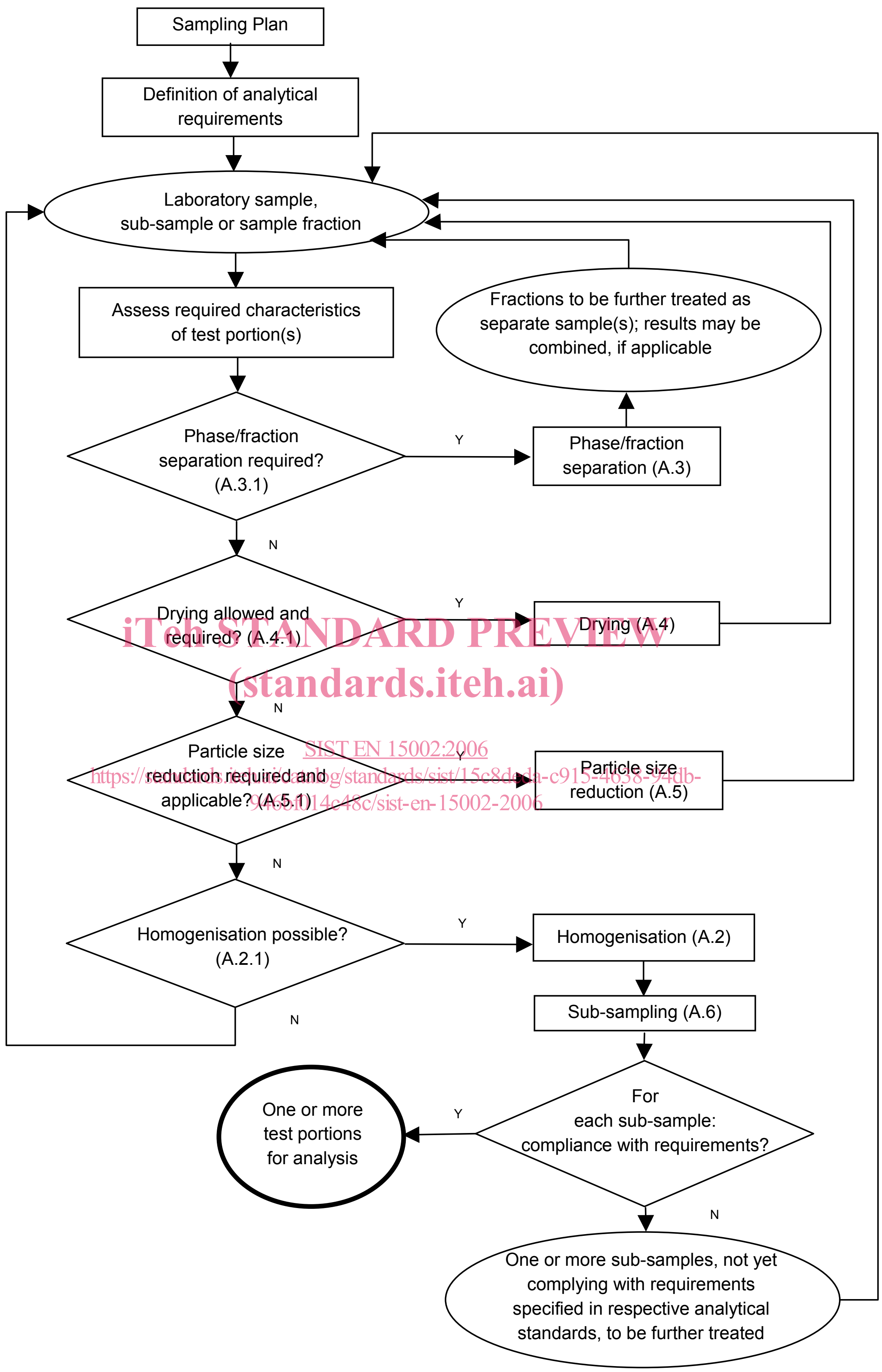
Sampling Plan
Definition of analytical requirements
Laboratory sample, sub-sample or sample fraction
Assess required characteristics of test portion(s)
Fractions to be further treated as separate sample(s); results may be combined, if applicable
Phase/fraction separation required? (A.3.1)
Y
Phase/fraction separation (A.3)
N
Drying allowed and required? (A.4.1)
Y
Drying (A.4)
N
Particle size reduction required and applicable? (A.5.1)
Y
Particle size reduction (A.5)
N
Homogenisation possible? (A.2.1)
Y
Homogenisation (A.2)
N
Sub-sampling (A.6)
For each sub-sample: compliance with requirements?
Y
One or more test portions for analysis
N
One or more sub-samples, not yet complying with requirements specified in respective analytical standards, to be further treated

Figure 1 — Flow sheet – sequence of operations

7 Report

The work carried out by the testing laboratory shall be covered by a report which accurately, clearly and unambiguously presents all relevant information.

Each report shall include at least the following information:

a) name, address and location of any laboratory involved in the preparation of the test portions;

b) unique identification of report (such as serial number) and of each page and total number of pages of the report;

c) description and identification of the laboratory sample;

d) date of receipt of laboratory sample;

e) reference to this European Standard, i.e. EN 15002;

f) reference to the sampling report; if a sampling report is not available, precise reference shall be made to the company or persons responsible for the sampling;

g) whole sequence and operating conditions (procedures and apparatuses) actually applied to the laboratory sample for preparation of test portions;

h) any details not specified in this European Standard or which are optional, and any other factors which may have affected the results.

For the subsequent performance of the analyses the dates of the preparation of the test portions shall be available.

Annex A
(normative)

Guideline for choosing sample treatment techniques

A.1 General

The preparation of test samples from a laboratory sample will frequently involve a sequence of operations such as homogenisation, phase separation, drying, particle size reduction and sub-sampling. Specific forms of these operations are described in this annex.

The sample treatment techniques prescribed in the analytical standards have to be fulfilled in any case.

A.2 Homogenisation

A.2.1 General information

Before each operation that implies sub-sampling, a homogenisation step is required, in order to guarantee that all sub-samples or sample fractions have the same properties and composition. The homogenisation technique to be used is chosen depending on the properties of the sample.

In many cases before homogenisation particle size reduction may be necessary.

If homogenisation of a sample is too difficult or even practically impossible (e.g. if the sample contains pieces of plastic or metal), its phases shall be separated and treated as they were different samples (see A.3 phase/fraction separation).

A.2.2 Solid samples

A.2.2.1 Manual homogenisation

When to use it

— Generally usable;

— in cases when mechanical homogenisation could lead to loss of volatile compounds of interest.

When not to use it

— For samples that form layers because of the presence of particles of different density;

— for samples with particles of such a large size that homogenisation can not be reached by manual mixing.

Procedure

Mix the sample with appropriate tool (e.g. shovel, scoop, pestle and mortar). If there is a risk of losses of volatile substances the manual homogenisation has to be done very carefully.