

**SLOVENSKI STANDARD**  
**oSIST prEN 15934:2011**  
**01-januar-2011**

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**Blato, obdelani biološki odpadki, tla in odpadki - Izračun suhe snovi z določitvijo suhega ostanka in vode**

Sludge, treated biowaste, soil and waste - Calculation of dry matter by determination of dry residue or water content

Schlamm, behandelter Bioabfall, Boden und Abfall - Berechnung der Trockenmasse durch Bestimmung des Trockenrückstands oder des Wassergehalts

Boue, biodéchet traité, sol et déchets - Calcul de la teneur en matière sèche par détermination du résidu sec ou de la teneur en eau

**Ta slovenski standard je istoveten z: prEN 15934**

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## Foreword

This document (prEN 15934:2010) 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 document is currently submitted to the second CEN Enquiry.

This draft European Standard prEN 15934 was completely technically and editorially revised following the comments made during the first CEN-Enquiry in 2009 and the discussions from CEN/TC 400/WG 4 "Inorganic elements and compounds".

This European Standard is part of a modular horizontal approach in which this document belongs to the analytical step.

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.

Until now, test methods determining properties of materials within the environmental area were prepared in Technical Committees (TCs) working on specific products/matrices (e. g. soil, waste, sludge). However, it is understood that many steps within individual test procedures may also be used for the analysis of various other materials. By careful determination of these steps and selection of specific questions within these steps, elements of the test procedure can be described in a way that can be used for a variety of matrices and materials with certain specifications. This optimization is in line with the development among end-users of standards. A majority of routine environmental analyses are carried out by institutions and laboratories working under a scope that is not limited to one single environmental matrix but covers a wide variety of matrices. Availability of standards covering more matrices contributes to the optimization of laboratory procedures and standard maintenance costs, e. g. costs related to accreditation and recognition.

A horizontal modular approach was developed in the project 'Horizontal'. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). A beneficial feature of this approach is that individual "modules" can be replaced by improved ones without jeopardizing the standard "chain".

The results of the desk study as well as the evaluation and validation studies have been subject to discussions with all parties concerned in the CEN structure during the development by project 'Horizontal'. The results of these consultations with interested parties in the CEN structure have been presented to and discussed in CEN/TC 400.

Based on data from interlaboratory studies and consultations with interested parties within CEN member bodies, it has been concluded that this draft standard prEN 15934 is acceptable for its intended use and is ready for CEN enquiry.

It is recognized that standardization in the environmental field in most national standardization bodies is organized in national standardization committees that mirror the vertical structure of technical committees in the environmental field in CEN. The present CEN enquiry therefore asks for special attention by the NSBs to assure that the relevant and interested parties are consulted during the CEN enquiry, i. e. to assure that one single consolidated enquiry reply on this draft standard prEN 15934 can be presented by the NSB that covers the entire scope of this draft standard.

## Introduction

In case of analysis of solids and sludge water is usually not considered as part of the sample and results are generally related to dry matter, which can be calculated from the water content or the dry residue. For this purpose two methods are described in this European Standard. The choice of the method depends on the type of sample and its content of volatile substances excluding water.

As a result of the validation study the determination of water content by azeotropic distillation has been replaced by Karl-Fischer-titration. Nevertheless the distillation may be useful in certain cases. This method is described in Annex B (informative).

This European Standard is (applicable and) validated for several types of matrices as indicated below (see also Annex A for the results of the validation):

**Table 1 — Matrices for which this European Standard is (applicable and) validated**

Matrix		Validated for
Sludge	(only method A)	Municipal sludge
Biowaste	(only method A)	Fresh compost
Soil	(only method A)	Sludge amended soil
Waste	(method A and B)	Contaminated soil, Dredged sludge, Nickel sludge, Filter cake, Distillation residue, Drilling emulsion

**WARNING** — Persons using this European Standard should be familiar with normal 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 methods for the calculation of the dry matter of sludge, treated biowaste, soil and waste for which the results of performed analysis are to be calculated to the dry matter basis. Depending on the nature and origin of the sample, the calculation is based on a determination of the dry residue (Method A) or a determination of the water content (Method B). It applies to samples containing more than 1 % (mass fraction) of dry residue or more than 1 % (mass fraction) of water.

Method A applies to sludge, treated biowaste, soil and solid waste, method B applies to liquid waste and to samples which are suspected or known to contain volatiles except of water.

## 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.

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

prEN WI 00400022, *Sludge, treated biowaste and soil — Guidance for sample pre-treatment*

## 3 Terms and definitions

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

### 3.1

#### dry residue

$w_{dr}$

remaining mass fraction of a sample after a drying process at 105 °C under specified conditions

### 3.2

#### water content

$w_w$

mass fraction of water in a sample determined by Karl-Fischer-titration under specified conditions

### 3.3

#### dry matter

$w_{dm}$

mass fraction of a sample excluding water expressed as mass fraction calculated by determination of dry residue or water content

## 4 Principle

### 4.1 General

Depending on the origin (sludge, biowaste, soil, waste) and the nature of the sample (liquid, solid or multiphase) either the water content or the dry residue is to be determined. The results from the determination of water content or dry residue are used to calculate the dry matter. In case of multiphase (waste) samples these samples shall be homogenized. If homogenization is not possible, a phase separation according to EN 15002 shall be applicable and the phases are analysed separately.

### 4.2 Principle of Method A — Drying at 105 °C (dry residue)

The sample is dried to a constant mass in an oven at  $(105 \pm 3)$  °C. This method applies to solid samples and samples which become solid during the drying process. The method generally applies to soil, sludge and treated bio waste. Solid samples containing amounts of volatiles which are expected to affect the results shall be analysed by Method B.

### 4.3 Principle of Method B — Direct Karl-Fischer-titration (water content)

The method usually applies to liquid samples and homogenised multiphase samples. It could also be used for solid samples in case the “water content” is of interest and in case volatiles are expected to interfere with the determination according to Method A. The water content of a sample is determined by direct Karl-Fischer-titration and either volumetric or coulometric detection. Liquid samples are directly added to the cell, while solid samples are extracted by use of methanol.

## 5 Sample preparation

Samples should be pretreated according to prEN WI 00400022, if necessary.

## 6 Method A — Drying at 105 °C

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### 6.1 General

When carrying out one or more analysis on a sample, the dry matter shall be determined on an identical test portion in parallel in order to recalculate the results of the other analysis to the dry matter basis.

### 6.2 Interferences

The samples can change during the drying process, e.g. by absorption of carbon dioxide in the case of alkaline samples, or of oxygen by reducing substances. Volatile compounds evaporating at 105 °C are measured as water using this procedure.

NOTE For alkaline samples or samples susceptible to oxidation the drying could be carried out in a nitrogen atmosphere.

### 6.3 Hazards

Flammable or explosive gases may be released in the drying process.

### 6.4 Apparatus

**6.4.1 Drying system** thermostatically controlled and capable of maintaining a temperature of  $(105 \pm 3)$  °C; e.g. drying oven, infrared system, halogen lamp system.



**6.4.2 Desiccator** with an active drying agent such as silica gel.

**6.4.3 Precision balance** with an accuracy of 1 mg or better.

**6.4.4 Evaporating dish or crucible.** Temperature tolerant laboratory vessel withstanding 105 °C. Suitable materials are metallic or ceramic.

NOTE 1 If the sample is to be ashed following this procedure, it should be ensured that the material will withstand the elevated temperatures.

NOTE 2 The material of the evaporating dish or crucible should be compatible with the nature of the sample to avoid interferences upon weighing due to possible oxidations.

## 6.5 Procedure

Place an evaporating dish or crucible (6.4.4) in the drying system (6.4.1) set at  $(105 \pm 3)$  °C for a minimum of 30 min. After cooling in the desiccator (6.4.2) to ambient temperature, weigh the basin to the nearest 1 mg.

Depending on the expected dry residue, weigh into the evaporating dish or crucible a suitable amount of material so that the residue obtained has a weight of at least 0,5 g. Weigh the loaded basin to the nearest 1 mg.

Place the evaporating dish or crucible (6.4.4) containing the sample in the drying system (6.4.1) set at  $(105 \pm 3)$  °C until the residue appears dry, typically overnight.

NOTE 1 For some samples e.g. sludge, there is a risk of formation of a cake surface, which hinders an even drying. To avoid this, a glass rod can be weighed along with the dish. If cake formation occurs during drying, the glass rod is used to stir the sample, to break up the cake. This should be repeated as necessary. The rod should be reweighed afterwards.

After cooling in the desiccator (6.4.2) weigh the evaporating dish or crucible and contents for the first time,.

The dry residue shall be regarded as constant if the mass obtained after further 1 h of drying does not differ by more than 0,5 % of the previous value or 2 mg whichever is the greater. Otherwise repeat the drying process.

In case of weight inconstancy after three cycles the drying process may be stopped (after at least 12 h). The result of the last weighing shall be recorded in the test report.

In case of infrared or halogen lamp drying systems the corresponding manufacturer's instructions apply. The technique of choice shall be recorded in the test report.

Other techniques than oven drying, infrared or halogen lamp drying are allowed, provided they are proven to give comparable results.

## 6.6 Calculation of results

The dry residue is calculated according to Equation (1):

$$w_{\text{dr}} = \frac{m_{\text{c}} - m_{\text{a}}}{m_{\text{b}} - m_{\text{a}}} \cdot f \quad (1)$$

where

$w_{\text{dr}}$  is the dry residue of the sample, expressed as mass fraction in percent (%) or in grams per kilogram (g/kg);

$m_{\text{a}}$  is the mass of the empty dish or crucible, expressed in grams (g);

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$m_b$  is the mass of the dish or crucible containing the sample, expressed in grams (g);

$m_c$  is the mass of the dish or crucible containing the dried sample, expressed in grams (g);

$f$  is the conversion factor  $f = 100$  for expression of results as mass fraction in percent (%) and  $f = 1\,000$  for expression of results in grams per kilogram (g/kg).

Perform the analysis at least in duplicate; determine the mean value and calculate the dry matter according to equation (5).

## **7 Method B — Direct Karl-Fischer-titration (volumetric/coulometric detection)**

### **7.1 Interferences**

Substances like peroxides, oxides, hydroxides, ammonia, carbonates, hydrogen carbonates, disulfites, nitrite, sulfite, thiosulfate, hydrazine and derivatives, iron (II), copper (I), tin (II), silanols, arsenite, arsenate, selenite, tellurite, phenols, boric compounds, aldehydes, acetone and other reactive ketones may cause interferences. Especially all reductive or oxidative reagents are potentially interfering. In case of concern about interferences results can be verified by azeotropic distillation (Annex B).

Most of the interferences are reduced in case of extraction with methanol before analysis.

### **7.2 Reagents**

All reagents used shall be at least of analytical grade and suitable for their specific purposes. Hygroscopic substances shall be stored in a desiccator.

**7.2.1 Karl-Fischer reagent** as recommended by provider of KF-system.

**7.2.2 Methanol.** The methanol used shall not contain more than a mass fraction of 0,005 % water.

**7.2.3 Toluene** if necessary to solve a sample.

### **7.3 Apparatus**

Usual laboratory glassware and the following equipment shall be used.

**7.3.1 Karl-Fischer-titrator.**

**7.3.2 Shaking device.**

**7.3.3 Weighing pipette.**

**7.3.4 Conical flasks,** 250 ml with ground glass neck and stopper.

### **7.4 Procedure**

#### **7.4.1 Determination of the equivalence factor**

Fill the titration vessel (7.3.1) with methanol (7.2.2). Stir without splattering. Titrate with Karl-Fischer reagent (7.2.1) to the end point. Using a weighing pipette (7.3.3) add approximately 80 mg of water ( $W$ ) with an accuracy of 0,1 %. Titrate to the end point and record the consumption ( $V_1$ ).

The equivalence factor  $F$  is calculated according to Equation (2):