



# SLOVENSKI STANDARD

## SIST EN 14346:2007

01-april-2007

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### Karakterizacija odpadkov - Izračun suhe snovi z določitvijo suhega ostanka ali vode

Characterization of waste - Calculation of dry matter by determination of dry residue or water content

Charakterisierung von Abfällen - Berechnung der Trockenmasse durch Bestimmung des Trockenrückstandes oder des Wassergehaltes

Caractérisation des déchets - Calcul de la teneur en matière sèche par détermination du résidu sec ou de la teneur en eau

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#### **ICS:**

13.030.01      Odpadki na splošno      Wastes in general

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ICS 13.030.01

English Version

## Characterization of waste - Calculation of dry matter by determination of dry residue or water content

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oder des Wassergehaltes

This European Standard was approved by CEN on 25 November 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.

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## Foreword

This document (EN 14346:2006) has been prepared by Technical Committee CEN/TC 292 "Characterization 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of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.

Anyone dealing with waste and sludge analysis should 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, infectious) substances, which can be liable to biological and/or chemical reaction. Consequently these samples should be handled with special care. Gases which 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.

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.

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## Introduction

In case of waste analysis 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).

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## 1 Scope

This European Standard specifies methods for the calculation of the dry matter of samples for which the results of performed analysis are to be calculated to the dry matter basis. Depending on the nature 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 % (*m/m*) of dry residue or more than 1 % (*m/m*) 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*

## 3 Terms and definitions

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

NOTE These definitions only apply to waste. In standards dealing with other matrices other terms and definitions exist for technical reasons.

### 3.1 dry residue

$w_{dr}$   
remaining mass fraction of a sample after a drying process at 105 °C as specified in this European Standard

### 3.2 water content

$w_w$   
mass fraction of water in a sample determined by Karl-Fischer-titration as specified in this European Standard

### 3.3 dry matter

$w_{dm}$   
mass fraction of a sample excluding water expressed as a percentage by mass calculated by determination of dry residue or water content according to this European Standard

## 4 Principle

### 4.1 General

Depending on 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 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. 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 — Karl-Fischer-titration (water content)

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. 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.

## 5 Sample preparation and storage

Specification of sample preparation is not included in this European Standard. As the dry matter content is calculated in order to convert obtained results for a sample to the dry matter basis, the determination of dry residue or water content shall be carried out on the identical portions of the same test sample. By that the sample preparation is as specified in the actual analysis standard.

Storage of samples may cause changes as e.g. uptake or liberation of water, carbon dioxide and other volatiles. If it is not possible to carry out the determination of dry residue or water content parallel to the analyses, samples shall be stored in air-tight containers. Biologically active samples shall be stored at about 4 °C and analysed within 3 days.

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## 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 Equipment

**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 Analytical 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 waste 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 ( $m_a$ ).

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 ( $m_b$ ).

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, ( $m_c$ ).

The dry residue ( $m_c - m_a$ ) shall be regarded as constant if the mass obtained after a further one hour 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.

NOTE 2 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 should 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 has to be noted in the test report.

Other techniques than oven drying (infrared/halogen) are allowed, provided they are proven to give comparable results.

## 6.6 Calculation of results

The dry residue, expressed as mass fraction in per cent or in grams per kilogram, is calculated according to Equation (1):

$$w_{\text{dr}} = \frac{(m_c - m_a)}{(m_b - m_a)} \times f \quad (1)$$

where

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

$m_a$  is the mass of the empty dish or crucible, expressed in grams [g];

- $m_b$  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 per cent [%] and  $f = 1\,000$  for expression of results in grams per kilogram [g/kg].

The analysis is performed at least in duplicate; the mean value is calculated and reported.

## 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 interfere. 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 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 Equipment

Usual laboratory glassware and the following equipment shall be used.

**7.3.1 Apparatus suitable for Karl-Fischer-titration.**

**7.3.2 Shaking apparatus.**

**7.3.3 Weighing pipette.**

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

### 7.4 Procedure

#### 7.4.1 Determination of the equivalence factor

Fill a clean and dry 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. Add by use of a weighing pipette (7.3.3) approximately 80 mg of water ( $W$ ) with an accuracy of 0,1 %. Titrate to the end point and note the consumption ( $V_1$ ).

The equivalence factor  $F$ , expressed as mass concentration in milligrams per millilitre, is calculated according to Equation (2):

$$F = \frac{W}{V_1} \quad (2)$$

where

$F$  is the equivalence factor, expressed in milligrams water per millilitre [mg/ml];

$W$  is the quantity of water, expressed in milligrams [mg];

$V_1$  is the volume of the titration fluid, expressed in millilitres [ml].

The equivalence factor shall be determined weekly.

Instead of water other suitable standards with defined water content are applicable. In case of coulometric detection no titer adjustment is required.

#### 7.4.2 Analysis of liquid samples

Treat the liquid sample as follows:

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. Add an appropriate amount of the liquid sample  $M$  with an accuracy of 0,1 % (in case of water contents less than 10 % about 1 g of sample is appropriate).

Note the added volume of the titration liquid ( $V$ ) for the sample.

The water content of the liquid sample, expressed as mass fraction in per cent, is calculated according to Equation (3):

$$w_w = \frac{V \cdot F}{M \cdot 1000} \cdot 100 \% \quad (3)$$

where

$w_w$  is the water content, expressed as mass fraction in per cent [%];

$V$  is the added volume of the titration liquid, expressed in millilitres [ml];

$F$  is the equivalence factor (according to Equation (2)), expressed in milligrams water per millilitre [mg/ml];

$M$  is the mass of an aliquot of the sample, expressed in grams [g].

The dry matter is to be calculated by Equation (6).

#### 7.4.3 Analysis of solid samples

Treat the solid phases as follows:

Weigh approximately 10 g solid sample  $M$  to 0,1 % accurately into a conical flask (7.3.4). Pipette 50,0 ml methanol (7.2.2). Shake for 10 min in a shaking machine (7.3.2). Fill the titration vessel with methanol (7.2.2). Stir without splattering. Titrate with Karl-Fischer reagent (7.2.1) to the end point. Pipette an appropriate volume ( $V_E$ ) of the extract into the sample vessel (in case of water contents less than 5 % about 10 ml of extract is appropriate).