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Sludge, treated biowaste and soil – Determination of dry matter – Gravimetric method

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Foreword

This document is a working document.

This document TF WI has been prepared by the Project Horizontal…...

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative Annex A, B, C or D, which is an integral part of this document.

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

[table to be filled and amended by the standards writer]

Material	**Validated for** **(type of sample, e.g. municipal sludge, compost)**	**Reference:**
Sludge	Validated	EN 12880:2000, Annex A
Soil	Validated	ISO 11465:1993
Bio waste, soil improvers and growing media (	Unfertilised peat perlite, coarse bark, composted straw and domestic sewage)	EN 13040:1999, Annex B
Sediment	Not validated yet	
Waste	(on-going - TC292,WG5)	prEN 14346

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Introduction

NOTE: This is a draft version; the introduction will need to be adjusted….

It is the result of a desk study "Desk study on dry matter and loss on ignition". After discussion with all parties concerned in CEN the standard has been developed further as a modular horizontal method and has been validated within in the project ‘Horizontal’.

A horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in a test procedure and not the whole test procedure (from sampling to analyses).

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The other horizontal modules that will be available in due time are to be found in the informative annex [*xxx*] which contains a brief overview of the modules that will be worked out in the project 'Horizontal.'

The texts of the chapters 1 to 12 are normative; annexes are normative or informative, as stated in the top lines of the annexes.

1 Scope

This European Standard specifies a method for the determination of the dry matter on a mass basis of samples of:

- sludges, including liquid, paste-like or solid sludges,
- all types of air-dried soil samples and field moist soil samples,
- sediment, and
- treated biowaste

Note: Determination of water content of a sample using this method is possible provided that other compounds other than water do not contribute significantly to the weight loss by heating to 105 ºC.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN XXXXX:200X – Solid material. Determination of loss on ignition of the dry mass.

EN XXXXX:200X - Solid material. Sample pre-treatment……

EN XXXXX:200X - Solid material. Sampling……..

3 Terms and definitions

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

3.1 Dry matter w_{dm}

Dry residue after drying according to the specified drying process. It is expressed as a percentage or in grams per kilogram.

3.2 Water content w_{wc}

The mass fraction determined as the loss on mass after the specified drying process. It is expressed as a percentage or in grams per kilogram.

3.3 Constant mass

Mass reached when, during the drying process, the difference between two successive weighings of the sample at an interval of minimum 1 h, first heated, then cooled to room temperature, does not exceed 0.5% (m/m) of the last determined mass or 2 mg, whichever is the greater.

Note 1 These definitions do not - for technical reasons - apply to samples containing volatile substances.

Note 2 Usually 16 h to 24 h are sufficient for most soil, sludge, sediment and waste samples, but certain sample types and large samples may require longer drying periods.

4 Safety remarks

Samples of sludge, bio-waste or contaminated soils are liable to ferment and usually contain harmful microorganisms. It is essential to keep them away from any food or drink, and to protect any cuts. Bursting bottles containing e.g. sludge can produce microorganisms-contaminated shrapnel and/or infectious aerosols.

When handling sludge and bio-waste samples, it is necessary to wear gloves, face and eye protection, and sufficient body protection to guard against bottles bursting. Gasses evolved may be flammable.

Special measures must be taken during the drying process to prevent contamination of the laboratory atmosphere by flammable, explosive or toxic gasses.

5 Principle

Samples are dried to constant mass in an oven at (105 $\pm$ 5) ^{o}C. The difference in mass before and after the drying process is used to determine the dry matter and the water content.

6 Interferences and sources of errors

The samples may change chemically during the drying process (e.g. by absorption of carbon dioxide in the case of basic samples or of oxygen caused by reducing substances).

Note 1 When determining the water content, volatile substances (such as organic solvents or substances deriving from the decomposition of organic or inorganic substances) are also included either completely or partially.

Note 2 In case of samples with a high content of solids (e.g. dry matter $w_{dr} \geq 30\%$) there is the risk of water still remaining trapped in the sample after drying.

Note 3 Decomposition of organic matter can, in general, be neglected at this temperature. However, for soil samples with a high content of organic matter (> 10% (m/m)), for example peaty soils, the method of drying should be adapted. In this case, the sample should be dried to constant mass at 50^{o}C. Use of a vacuum will speed up the operation.

Note 4 Some minerals similar to gypsum lose water of crystallisation at a temperature of 105^{o}C.

7 Apparatus

7.1 Drying oven
thermostatically controlled with forced air ventilation, maintaining a temperature of (105 $\pm$ 5)^{o}C.

7.2 Desiccator
with active drying agent such as silica gel.

7.3 Temperature tolerant evaporating dish or crucible
withstanding at least 105^{o}C for dry matter analyses or 550^{o}C for further analyses of loss on ignition is required (See EN xxxxx). Suitable materials are nickel, porcelain, silica, and platinum.

7.4 Analytical balance

with an accuracy of 1 mg or better. For samples with dry residue of 10 g or higher, an analytical balance with an accuracy of 10 mg may be used.

8 Sampling and sample pre-treatment

Sampling and sample pre-treatment shall be carried out in accordance with the method specified in EN XXXX for …….. and in EN XXXX for

9 Procedure

Place an evaporating dish or crucible (7.3) in the drying oven (7.1) set at (105 ± 5) ^{o}C for a minimum of 30 minutes and then cool to ambient temperature in a desiccator (7.2), with the lid closed. After cooling, weigh the dish or crucible to the nearest 1 mg, m_a.

If the same crucible is to be used for the subsequent loss on ignition measurement (Se EN XXXXX), it shall be pre-ignited at 550^{o}C for a minimum of 30 min.

Depending on the expected water content, weigh into the evaporating dish or crucible (7.3) a suitable amount of sample, m_b, so that the dry matter obtained has a mass of not less than 0.5 g.

Note 1 Sample amounts of 30 – 50 g are suitable for field-moist soil samples, paste-like and solid sludge and solid waste. A larger test portion may be needed to assure a representative sample for e.g. composted bark samples. For air-dried soil samples 10 – 15 g are suitable.

Note 2 Determination of dry matter shall be determined on samples identical to those used for determination of parameters that relates to dry matter.

Place the evaporating dish or crucible (7.3) containing the sample in the drying oven (7.1) set at 105^{o}C until the residue appears dry, typically overnight.

Note 3 There is a risk of a cake surface forming. The formation of such cake surface impedes even drying. To avoid this, a glass rod can be weighed along with the crucible. If cake formation occurs during drying, the glass rod is used to stir the sludge to break up the cake and bring the liquid surface into contact with hot air. This is repeated as necessary.

Note 4 In the case of samples containing considerable amounts of water careful evaporation of the major part of the water is preferred (e.g. in a water bath) in order to avoid loss of substances by splashing. Alternatively freeze-drying may be used as a first step for the determination of dry matter.

After cooling in the desiccator (7.2), weigh the evaporating dish or crucible and contents for the first time, m_c.

The mass (m_c – m_a) shall be regarded as constant if the mass obtained after another hour of drying does not differ by more than 0.5% of the previous value or 2 mg, whichever is the greater (3.3).

Otherwise, repeat drying until constant mass is reached.

Note 5 In cases when even after the third drying process it is not possible to obtain a constant value, record the value determined after at further 2 h together with a remark on the unfinished process.

Note 6 20 hours of drying and omission of re-drying/re-weighing can be applied for sample types with documented evidence that the necessary drying time is less than 20 hours.

10 Quality assurance of the overall procedure

10.1 Quality control

At least one duplicate analysis should be carried out in each batch of analyses. Where uncertainty exists about the homogeneity or behaviour of the sample it is recommended that the analysis be carried out in duplicate.

10.2 Precision data

To be inserted in Annex

11 Calculation and expression of results

Calculate the dry matter (w_{dm}) or the water content (w_{wc}) expressed as a percentage of mass or grams per kilogram using the following equations:

$$w_{dm} = \frac{m_c - m_a}{m_b - m_a} \times f$$

$$w_{wc} = \frac{m_b - m_c}{m_b - m_a} \times f$$

where:

w_{dm} = is the dry matter of the sample, in percentages or grams per kilogram;

w_{wc} = is the water content of the sample, in percentages or grams per kilogram;

m_a = is the mass of the empty dish or crucible in grams;

m_b = is the mass of the dish or crucible containing the sample in grams;

m_c = is the mass of the dish or crucible containing the dry matter in grams;

f is a conversion factor, f = 100 for expression of results as a percentage and factor f = 1 000 for expression in grams per kilogram.

Values should be rounded to the nearest 0.1% (w/w) or alternatively to the nearest 1 g/kg.

12 Test report

The test report shall contain the following information:

a) Reference to this European Standard;

b) All information necessary for the complete identification of the sample;

c) Details of sample pre-treatment, if carried out;

d) Particular characteristics of the sample;

e) Results of the determination according to clause 11;

f) Any detail not specified in this European Standard and any other factor that may have affected the results.

Annex A
(informative)

Validation of methods

Annex B
(informative)

The modular horizontal system

Annex C
(informative)

Information on WP xx and the project Horizontal

Bibliography