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Chemical Analyses – Determination of dry matter and water content on a mass basis in sediment, sludge, soil, and waste – Gravimetric method

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Introduction

This document is developed in the project 'Horizontal'. It is a result of a desk study on dry matter, loss on ignition and total organic carbon in the project that aims at describing horizontal standards to be used for sludge, soil and biowaste. After discussion with all parties concerned in CEN the standard will be developed further as a modular horizontal method and validated in the project 'Horizontal'.

Until now test methods determining properties of materials were often prepared in Technical Committees (TC's) working on specific products or specific sectors. In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis 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 performing 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, together forming the basis for the test procedure.

This standard is a module for determination of dry matter and water content in sludge, soil, sediment and waste.

The texts of the chapters 1 to 11 are normative, except notes, which are informative. Annexes are informative.

1 Scope

This European Standard specifies a method for the determination on a mass basis of the dry matter and water content of samples of sludge, soil, sediment and waste – excluding samples containing volatile substances.

This method applies to the determination of dry matter and water content of:

- sludges, including liquid, paste-like or solid sludges,
- all types of soil samples,
- sediment, and
- waste

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 – Determination of loss on ignition of the dry mass in sediment, sludge, soil, and waste.

EN XXXXX:200X - Sample preparation

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 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.

5 Interferences

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 $105^{o}C$.

6 Storage

During storage, samples may be subject to changes (e.g. uptake or liberation of water, carbon dioxide and other volatiles) which are liable to falsify results. Biologically active samples should be analysed within 3 days. If analysed within this period, the samples should be stored at about 4°C, or otherwise stored directly at maximum - 18°C. Other samples may be stored in a closed container between 0°C and 4°C.

7 Hazards

Samples of sludge, biowaste or contaminated soil 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 microorganism-contaminated shrapnel and/or infectious aerosols.

When handling sludge and biowaste 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.

8 Apparatus

8.1 Drying oven
thermostatically controlled with forced air ventilation, maintaining a temperature of (105 ± 5)°C.

8.2 Desiccator
with active drying agent such as silica gel.

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

8.4 Analytical balance
with an accuracy of 1 mg or better.

9 Procedure

Place an evaporating dish or crucible (8.3) in the drying oven (8.1) set at (105 ± 5)°C for a minimum of 30 minutes and then cool to ambient temperature in a desiccator (8.2), 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:200X), it shall be pre-ignited at 550°C for a minimum of 30 min.

Depending on the expected water content, weigh into the evaporating dish or crucible (8.3) a suitable amount of sludge, m_b, so that the dry matter obtained has a mass of not less than 0.5 g.

Place the evaporating dish or crucible (8.3) containing the sample in the drying oven (8.1) set at 105°C until the residue appears dry, typically overnight.

Note 1 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 2 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 (3.4).

Otherwise, repeat drying until constant mass is reached.

Note 3 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.

10 Expression of results

10.1 Calculation

Calculate the dry matter (w_{dm}) or the water content (w_w) 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.

10.2 Precision data

See annex A.

The repeatability of separate duplicate determinations shall satisfy the conditions given in Annex A for similar sample types.

11 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 10;

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

Annex A

Performance data of interlaboratory comparisons

(Informative)

Sludge: performance data from EN 12880 (8 sludges).
(May be taken, as EN 12880 is technically identically to EN XXXXX).

Soil: Information to be obtained.

Waste (excluding samples containing significant amount of volatile substances): information to be obtained.

Bibliography

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

EN 12880:2000; Characterization of sludges – Determination of dry residue and water content.

EN 13040:1999; Soil improvers and growing media – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density.

DS 204:1980 (in compliance with SFS 3008, NS 4764, and SS 028113); Determination of total residue and total fixed residue in water, sludge and sediment.