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Solid materials – Determination of loss on ignition

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Foreword

This document is a working document.

This document XX WI has been prepared by 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)**	**Document**
Sludge	Validated	EN 12879:2001
Soil	Validated	EN 10694:1995
Bio waste, soil improvers and growing media		
Sediment		
Waste		

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Introduction

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

This document is developed in the project 'Horizontal'. 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 loss on ignition of dry mass of sludge, sediment, soil, and waste at 550ºC after the dry matter have been determined in accordance with the method of EN XXXXX.

This method applies to the determination of loss on ignition of:

- sludges, including liquid, paste-like or solid sludges
- all types of soil samples
- sediments
- bio waste (matrices to be defined during validation), and
- waste

NOTE The loss on ignition is often used as an estimate for the content of non-volatile organic matter in the sample. It should be noted that inorganic substances or decomposition products (e.g. H_2O, CO_2, SO_2, O_2) are released or absorbed and some inorganic substances are volatile under the reaction conditions.

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 dry matter– gravimetric method

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 Loss on ignition (LOI)

The change in mass as a result of heating a sample under specified conditions. The loss on ignition (LOI) is expressed as a weight percentage of the dry mass.

3.2 Residue on ignition

The mass remaining after heating a sample under specified conditions. The residue on ignition is expressed as a weight percentage of the dry mass.

3.3 Dry mass

The mass of sample obtained after the specified drying process. It is expressed in grams or kilograms.

3.4 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.

3.5 Constant mass

Constant mass is reached when the change in dry mass during a further period of heating of 1 hour is within 0,5 % (m/m) or 2 mg, whichever is the greater.

4 Safety remarks

Samples of sludge, biowaste or contaminated soil are liable to ferment and may contain harmful microorganisms. Consequently it is recommended that these samples should be handled with special care. The gases, which may be produced by microbiological activity, are potentially inflammable and will pressurise sealed bottles. Exploding bottles are likely to result in infectious shrapnel and/or pathogenic 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 ignition process to prevent contamination of the laboratory atmosphere by flammable, explosive or toxic gasses.

5 Principle

A dried test sample is heated in a furnace to constant mass at $(550 \pm 25)\,^{o}C$.

The difference in mass before and after the ignition process is used to calculate the loss on ignition.

The determination is performed on a dried sample or directly on the un-dried sample including a drying step or by referring to the dry matter.

6 Interferences and sources of errors

LOI is an empirical parameter, thus in principle there is no interference connected to the determination. However, for some purposes the determination of LOI is used for the assessment of the content of organic matter in the sample. It should be noted that elementary carbon in the sample will be included in the loss on ignition value. Furthermore, any volatilisation or chemical reactions of inorganic compounds will also be included in the loss on ignition value.

Note 1 Chemically bound water could be released during heating, thereby contributing to the loss on ignition.

Note 2 Iron or other metals present in the sample in metallic state could be oxidised during heating, thereby producing lower results.

Note 3 Sulphides present in the sample could be oxidised to sulphate during heating, thereby producing lower results

Note 4 Explosive ignition is likely to result in loss of residue from the crucible, thereby contributing to the loss on ignition.

Note 5 Calcium hydroxide or calcium oxide present in large amounts (e.g. sludge conditioned with lime) may combine with sulphuric oxides liberated during ignition or with carbon dioxide formed during ignition producing lower results.

7 Apparatus

7.1 Crucible
typically 50 to 70 mm in diameter, suitable for ignition at 550°C, e.g. made of nickel, platinum, porcelain, or silica.

7.2 Muffle furnace
or equivalent equipment, capable of maintaining a temperature of (550 ± 25) °C.

7.3 Metal plate

7.4 Desiccator
with an active drying agent, such as silica gel.

7.5 Analytical balance
with an accuracy of 1 mg or greater.

8 Sampling and sample pre-treatment

8.1 Sampling

Sampling should be carried out in accordance with EN yyyy (Horizontal standard module(s) for sampling of sludge, soil and biowaste) (and other relevant standards for sampling – to be defined).

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 the results. Biological 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 in a well-ventilated place.

8.2 Sample pre-treatment

Samples shall be homogenized according to EN wwww: (Horizontal standard module(s) for pre-treatment of solid materials). – (+ other, if relevant - to be adjusted

9 Procedure

9.1 Sludge, sediment, soil, and waste with low content of volatiles

If the determination of dry matter and the determination of loss on ignition are carried out in successive operations in the same crucible, refer to EN XXXXX for the initial crucible weighing. If not, the sample is a representative portion of the dry mass obtained according to EN XXXXX: Every necessary precaution shall be taken to avoid absorption of atmospheric humidity by the sample until weighed.

Place a crucible (7.1) in the furnace (7.2) and heat at (550 ± 25) °C for at least 30 minutes. Transfer the crucible from the furnace (7.2) after initial cooling on a metal plate (7.3) to a desiccator (7.4) and finish cooling to ambient temperature. Weigh the empty crucible to the nearest 1 mg, (m_a).

Weigh into the crucible 0.5 g to 5 g of the dried sample to the nearest 1 mg, (m_b), and raise the furnace temperature to (550 ± 25) °C and hold this temperature for at least 1 hour.

Note 1 If the dry mass has a high organic matter content, losses may occur as a result of rapid ignition or deflagration of the sample. In this case heat the sample slowly until ignition. For certain wastes (e.g. paper wastes and demolition wood) a step-wise heating process can be used: the crucible is inserted in a cold furnace; the temperature is raised to 250 ºC over a period of 50 minutes (allowing pyrolysis of the sample). Then the temperature is raised to 550 ºC over a period of 60 min and the 550 ºC is kept for at least 60 min.

Note 2 If the sample contains higher amounts of moisture, insert the crucible in a cold furnace and raise the furnace temperature evenly to (550 ± 25) °C over a period of 1 hour and hold this temperature for at least 1 hour.

Place the hot crucible containing the residue on ignition on a metal plate (7.3) for a few minutes. While still warm, transfer the crucible to a desiccator (7.4) and leave to cool to ambient temperature.

As soon as ambient temperature is reached, weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

The crucible is weighed immediately after removal from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and thus the loss on ignition shall be regarded as constant if the mass obtained after a further half-hour period of ignition at (550 ± 25) °C in the preheated furnace, (m_c - m_a), differs max. 0.5% of the previous value or 2 mg, whichever is the greater (3.5).

Note 3 In cases when even after the third ignition period constant mass is not obtained, record the value determined in the last of the three measurements. The lack of constant mass should be reported together with the result.

Note 4 If black carbon particles are still present (some organic substances burn slowly at 550 °C), wet the residue using a few drops of an ammonium nitrate solution. After repeated drying insert the crucible into the furnace and slowly heat to avoid losses by deflagration and continue heating the residue at (550 ± 25) °C. Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled water. Both the value of loss on ignition obtained after the third ignition period and the value of loss on ignition obtained after addition of ammonium nitrate shall be given in the test report.

9.2 Waste samples containing volatile substances

For samples containing significant amounts of volatile substances the dry matter cannot be determined as dry residue. In this case the dry matter shall be calculated from the water content. In this case the loss on ignition is always performed directly on the un-dried sample.

Place a crucible (7.1) in the furnace (7.2) and heat at (550 ± 25) °C for at least 30 min. Transfer the crucible from the furnace (7.2) after initial cooling on a metal plate (7.3) to a desiccator (7.4) and finish cooling to ambient temperature. Weigh the empty crucible to the nearest 1 mg, (m_a).

Weigh into the crucible 0.5 g to 5 g of the sample to the nearest 1 mg, (m_b). Larger masses may be taken if complete combustion can be assured. All necessary precautions should be taken to avoid loss of volatiles from the samples until it has been weighed.

Note 1 To avoid splashing from escaping vapours or sudden fire it is recommended to remove most of the volatile components from the sample at ambient temperature in a fume hood prior to ignition.

Note 2 Samples containing highly flammable components e.g. solvents or waste oil should be ignited and allowed to burn in a fume hood before being inserted into the furnace.

When ready the crucible is then inserted into a cold furnace and the temperature of the furnace is raised to (550 ± 25) °C and hold for at least 1 hour.

Place the hot crucible containing the residue on ignition on a metal plate (7.3) for a few minutes. While still warm, transfer the crucible to a desiccator (7.4) and leave to cool to ambient temperature. As soon as ambient temperature is reached, weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

Weighing is carried out immediately after removal of the crucible from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition - and therefore the loss on ignition - shall be regarded as constant, if the mass obtained after a further half-hour period of ignition at 550°C in the pre-heated furnace, (m_c - m_a), does not differ by more than 0.5% of the previous value or 2 mg, whichever is the greater (3.5).

Note 3 In cases when even after the third ignition period constant mass is not obtained, record the value determined in the last of the three measurements. The lack of constant mass should be reported together with the result.

Note 4 If black carbon particles are still present (some organic substances burn slowly at 550 ^{o}C), wet the residue using a few drops of an ammonium nitrate solution. After repeated drying insert the crucible into the furnace and slowly heat to avoid losses by deflagration and continue heating the residue at (550 ± 25) ^{o}C. Ammonium nitrate solution is prepared by dissolving 10 g of reagent grade ammonium nitrate, NH_4NO_3, in 100 ml distilled water. Both the value of loss on ignition obtained after the third ignition period and the value of loss on ignition obtained after addition of ammonium nitrate shall be given in the test report.

10 Quality assurance of the overall procedure

10.1 Quality control

At least one duplicate analysis of a control sample of a relevant sample type 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 of the samples be carried out in duplicate.

10.2 Precision data

To be inserted in Annex

11 Calculation and expression of results

The loss on ignition of the dry mass of a solid sample is expressed in percent of the dry mass.

If the loss on ignition is performed on a dried sample the result shall be calculated from equation (1):

(1) $w_{LOI} = \frac{(m_b - m_c)}{(m_b - m_a)} \times 100$

If the loss on ignition is performed directly on the un-dried sample the result shall be calculated from equation (2):

(2) $W_{LOI} = \left(\frac{m(d) - m(c)}{m(d) - m(a)} \cdot 100 - w_w \right) \cdot \frac{100}{100 - w_w}$

The residue on ignition of the dry mass of a solid sample expressed in percentages shall be calculated from equation (2):

$W_R = 100 - w_{LOI}$

where

w_{LOI} is the loss on ignition of the dry mass of a solid sample, in percentages;

W_R is the residue on ignition of the dry mass of a solid sample, in percentages;

m_a is the mass of the empty crucible, in grams;

m_b is the mass of the crucible containing the dry mass, in grams;

m_c is the mass of the crucible containing the ignited dry mass, in grams;

m_d is the mass of the crucible containing the un-dried sample in grams

The results shall be rounded to the nearest 0.1percent.

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) In case of addition of ammonium nitrate results according to clause 9

g) 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