

Sludge, treated biowaste and soil — Determination of loss on ignition

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Foreword

This document CEN/BT TF 151 WI CSS 99023 has been prepared by Technical Committee CEN/BT TF 151 "Horizontal Standards in the fields of sludge, bio-waste and soil", the secretariat of which is held by DS.

This document is currently submitted to the CEN Enquiry.

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 Z.

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

Material	Validated for (type of sample, e.g. municipal sludge, compost)	References:
Sludge	Eight sludge samples Municipal sludge, North Rhine Westphalia	EN 12879:2001 Horizontal Project Interlab comp.
Soil	Muck soil, andosol, garden soil, loess under forest and sandy soil Sludge amended soil, Barcelona	EN 10694:1995 Horizontal Project Interlab comp.
Waste	Contaminated soil, dredged sludge, nickel sludge	EN 15169
Biowaste	Fresh compost, Vienna	Horizontal Project Interlab comp.

Introduction

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'.

After an evaluation study, in which e.g. the ruggedness of the method was studied, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN. The standard is part of a modular horizontal approach in which the standard belongs to the analytical step.

Until now test methods determining properties of materials were often prepared in Technical Committees (TCs) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for every material or product. Consequently this has resulted in duplication of work. To avoid such duplication of work for parts of a testing procedure references to parts of test methods from other TCs were introduced. However the following problems are often encountered while using references in this way: 1) The referenced parts are often not edited in a way that they could easily be referred to, 2) the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

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 assessing a property and not the whole "chain of measurement" (from sampling to analyses). [A beneficial feature of this approach is that "modules" can be replaced by better ones without jeopardizing the standard "chain"](#).

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 modules that relates to this standard are specified in section XX Normative references.

An overview of modules and the manner, in which modules are selected will be worked out later, at which time proper reference in this standard will be provided.

1 Scope

This European Standard specifies a method for the determination of the loss on ignition of dry mass of sludge, sediment, soil, and treated biowaste at 550 °C after the dry matter have been determined in accordance with the method of CEN/BT 151 WI CSS99023.

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;
- treated biowaste.

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. H₂O, CO₂, SO₂, O₂) 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).

CSS99031 Sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan

CSS99058 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions

CSS99057 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques

CSS99032 Sludge, treated biowaste, and soils in the landscape – Sampling - Part 3: Guidance on sub-sampling in the field

CSS99059 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

CSS99060 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan

CSS99035 Soil, sludge and treated biowaste – Pre-treatment for inorganic characterization

CSS99022 Soil, sludge and treated biowaste – Determination of dry matter – Gravimetric method

3 Terms and definitions

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

3.1

loss on ignition (LOI)

change in mass as a result of heating a sample under specified conditions

NOTE The loss on ignition (LOI) is expressed as a weight percentage of the dry mass.

3.2

residue on ignition

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 matter, w_{dm}

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

3.4

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.

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 $(550 \pm 25) ^\circ\text{C}$.

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 mm 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

Suitable for the initial cooling of crucibles.

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 CSS99031-32 and 57-60 (Horizontal standard module(s) for sampling of sludge, soil and biowaste).

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 CSS99034 (Horizontal standard module(s) for pre-treatment of solid materials).

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 CSS 99022 for the initial crucible weighing. If not, the sample is a representative portion of the dry mass obtained according to CSS 99022 Every necessary precaution shall be taken to avoid absorption of atmospheric humidity by the sample until weighed.

Place a crucible (see 7.1) in the furnace (see 7.2) and heat at (550 ± 25) °C for at least 30 minutes. Transfer the crucible from the furnace (see 7.2) after initial cooling on a metal plate (see 7.3) to a desiccator (see 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 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 slowly to 250 °C over a period of 50 minutes (allowing pyrolysis of the sample). Then the temperature is raised slowly to 550 °C and the 550 °C is kept for at least 2 h.

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 (see 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 (see 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 (see 7.2) after initial cooling on a metal plate (see 7.3) to a desiccator (see 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 (see 7.3) for a few minutes. While still warm, transfer the crucible to a desiccator (see 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 (see 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.

10 Quality assurance of the overall procedure

10.1 Quality control

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.

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):

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

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

$$(2) \quad w_{LOI} = 100 - \left(\frac{m(d) - m(a)}{m(b) - m(a)} \cdot 100 \right) \cdot \frac{100}{w_{DM}}$$

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;

w_{DM} is the dry matter of the sample, in percentages

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 un-dried sample in grams;

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

m_d = is the mass of the dish or crucible containing the ignited sample in grams

The results shall be rounded to the nearest 0.1 percent.

12 Precision data

The performance characteristics of the method (Annex A) data have been evaluated. Table 1 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Table A.2 in Annex A by taking the median value and rounding the numbers.

Table 1 — Typical values and observed ranges of the repeatability and reproducibility limits

<p>The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. Conversely, the repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample.</p>		
Results of the validation of the Determination of loss on ignition – Gravimetric method in soil, sludge and treated biowaste	Typical value %	Observed range %
Repeatability limit, r	5.5	3 – 8.5
Reproducibility limit, R	11	4 – 17

NOTE 1. The above results refer to the difference that may be found between two test results performed on two laboratory samples obtained under the same conditions. In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the above typical reproducibility values and observed reproducibility ranges should be divided by $\sqrt{2}$ to obtain the corresponding typical dispersion limit and its observed range. In the example of LOI in Sludge 2 the result and its dispersion limit is 45.4 ± 1.44 % dm ($2 * sR = 3.17$ % of 45.4). This means that with a 95 % statistical confidence, the values reasonably attributable to the measured parameter are larger than 43.93 % dm and lower than 46.81 % dm.

NOTE 2. The repeatability limit (r) and the reproducibility limit (R) as given in Table A.2 (Annex A) and in this table are indicative values of the attainable precision if the Determination of loss on ignition – Gravimetric method is performed in accordance with this standard [CSS99023].

NOTE 3. A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the the Determination of loss on ignition – Gravimetric method in soil, sludge and treated biowaste.

NOTE 4. In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table A.2 (Annex A) and this table.

13 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)

Repeatability and reproducibility data

A.1 Performance characteristics

A.1.1 Objective of the interlaboratory comparison

In a European wide interlaboratory comparison study according to ISO 5725-2, the performance characteristics of the standard “Sludge, soil and treated biowaste - Determination of loss on ignition” were established.

A.1.2 Materials used in the interlaboratory comparison study

The interlaboratory comparison of “Sludge, soil and treated biowaste - Determination of loss on ignition” was carried out with 25 - 29 European laboratories on 6 materials. The materials selected for the interlaboratory comparison were chosen to represent soil, sludge and biowaste as broad as possible, because the standard will find general application across different types of soil and soil related materials. (Detailed information can be found in the final report on the Interlaboratory comparison study mentioned in the Bibliography).

In the interlaboratory comparison study the following starting points were used:

The laboratory samples were all taken from one large batch of the different materials according to the normal practice. The normal size reduction and the normal repeated mixing were carried out as needed to obtain representative laboratory samples from the large batch sample (ref JRC).

The experimental plan was designed by project HORIZONTAL on the basis of each laboratory being given two laboratory samples of each material to be tested. This is in accordance with ISO 5725-2.

The materials examined cover all the grain size classes to which the the “Sludge, soil and treated biowaste - Determination of loss on ignition” applies: very fine grained materials (like sludge: 0 µm to about 125 µm) and fine-grained materials (soil and compost: 0 mm to 4 mm).

Table A.1 provides a list of the types of materials chosen for testing and the selected components.

Table A.1 — Material types tested and components analysed in the interlaboratory comparison of the method for the Determination of loss on ignition – Gravimetric method in soil, sludge and treated biowaste.

Grain size class	Sample code	Material type tested	Parameters/congeners
Sludge (<0.5 mm)	Sludge 1	Mix 1 of municipal WWTP sludges from North Rhine Westphalia, Germany	LOI
	Sludge 2	Mix 2 of municipal WWTP sludges from North Rhine Westphalia, Germany	LOI
Fine grained	Compost 1	Fresh compost from Vienna, Austria	LOI

(< 2 mm)	Compost 2	Compost from Germany	LOI
	Soil 4	A sludge amended soil from Hohenheim, Germany	LOI
	Soil 5	An agricultural soil from Reading, UK	LOI

A.1.3 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table A.2).

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The repeatability limit was calculated using the relationship : $r_{\text{test}} = f \cdot \sqrt{2} \cdot s_{r,\text{test}}$ with the critical range factor $f = 2$.

For instance, the repeatability limit around a measurement result of 50 % dm LOI is $\pm 2.54\%$ dm (i.e $\pm 5\%$ of 50).

NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value $f = 2$ used in the factor $f \cdot \sqrt{2}$ corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value $f = 2$ corresponds to the usual value $k = 2$ of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). However it may be necessary to use a larger value for f in situation as described in clause 12.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The reproducibility limit was calculated using the relationship: $R = f \cdot \sqrt{2} \cdot s_R$ with the critical range factor $f = 2$.

For instance, the reproducibility limit around a measurement result 50 % dm LOI is $\pm 6.05\%$ dm (i.e $\pm 12\%$ of 50)

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NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value $f = 2$ used in the factor $f \cdot \sqrt{2}$ corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value $f = 2$ corresponds to the usual value $k = 2$ of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the dispersion limit is equal to $k \cdot s_R$ with the usual value $k = 2$, resulting in a dispersion limit lower than the reproducibility limit (i.e. a ratio of $\sqrt{2}$). However it may be necessary to use a larger value $f \cdot \sqrt{2}$ (or k) in situation as described in clause 12 .

In case of relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Tables A.2 (this means that the value chosen for the critical range factor f is larger than 2 as well as for the coverage factor k for dispersion). This is because the extreme results may have been obtained in accordance with the present standard and/or be caused by the variability within, or in between, the laboratory samples.

Table A.2 — Results of the interlaboratory comparison studies of the determination of loss on ignition – Gravimetric method in soil, sludge and treated biowaste. All concentrations in % dm.

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Total number of data	No of LOD
Sludge 1	LOI	53.37	1.25%	4.20%	1.87	6.28	12	3	93	0
Sludge 2	LOI	45.37	1.12%	1.59%	1.43	2.02	9	5	67	0
Compost 1	LOI	44.83	1.44%	1.87%	1.81	2.35	11	3	84	0
Compost 2	LOI	27.98	3.30%	6.48%	2.59	5.07	11	3	86	0
Soil 4	LOI	4.84	2.18%	5.45%	0.30	0.74	12	4	86	0
Soil 5	LOI	4.99	3.03%	4.44%	0.42	0.62	11	4	81	0

Abbreviations: sr Repeatability standard deviation; SR Reproducibility standard deviation; r Repeatability limit (comparing two measurements); R Reproducibility limit (comparing two measurements); p Number of labs.