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Organic and organo-mineral fertilizers — Determination of the dry matter content

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

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European foreword

This document (CEN/prEN 17773) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This is a working document

This document will supersede CEN/TS 17773:2022. In comparison with the previous edition the following main changes have been made:

Introduction revised according to the recommended wording from FprEN 17704.

Analysis of blends added into the Scope

Normative references to sampling added

Terms and definitions revised and completed

Editorial changes and changes required for FprEN 17704 were included

Test report revised

Bibliography revised

Annex A (informative) Results of the inter-laboratory study, text prepared to be added after the validation

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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

Introduction

The European Committee for Standardization (CEN) was requested by the European Commission (EC) to draft European standards or European standardization deliverables to support the implementation of Regulation (EU) 2019/1009 of 5 June 2019 laying down rules on the making available on the market of EU fertilizing products (“FPR” or “Fertilising Products Regulation”) [1].

Organic and organo-mineral fertilizers are highly valuable tools in modern agriculture. Standardization was identified as having an important role in order to promote the use of organic and organo-mineral fertilizers. The work of CEN/TC 260 seeks to improve the reliability of the supply chain, thereby boosting the confidence of farmers, industry, and consumers in fertilizer products. This initiative will promote and support commercialisation of the European organic and organo-mineral fertilizers industry.

The preparation of this document is based on a standardization request to CEN by the European Commission and the European Free Trade Association (Mandate M/564 and relevant amendments) concerning the modernization of methods of analysis of fertilizers within the framework of Regulation (EU) 2019/1009 of the European Parliament and of the Council. This standardization request, presented as M/564, also contributes to the Communication on “Innovating for Sustainable Growth: A Bio economy for Europe”.

The other standards for determination of dry matter in solid fertilizers and liming materials [2, 3], in soil improvers and growing media [4] and sludge, soil and other solid matrices [5, 6] were studied as a basis of the described method. The sand pan technique suitable for liquid fertilizers where there is a risk of a cake surface formation or foam formation was based on the method of food analysis [7].

WARNING — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this document are carried out by suitably trained staff.

1 Scope

This document is applicable to the fertilizing products blends where a blend is a mix of at least two of the following components [Fertilizers/Liming Materials/Soil Improvers/Growing Media/Inhibitors/Plant Biostimulants] and where the following category [organic fertilizers, organo-mineral fertilizers] is the highest % in the blend by mass or volume, or in the case of liquid form by dry mass. If [organic fertilizers, organo-mineral fertilizers] is not the highest % in the blend, the European Standard for the highest % of the blend applies. In case a fertilizing product blend is composed of components in equal quantity, the user decides which standard to apply.

This document specifies the procedure for the determination and calculation of the dry matter fraction of organic and organo-mineral fertilizers for which the results of the performed analysis are to be calculated to the dry matter basis.

2 Normative references

EN 1482-1:2007 Fertilizers and liming materials - Sampling and sample preparation - Part 1: Sampling

EN 1482-2:2007 Fertilizers and liming materials - Sampling and sample preparation - Part 2: Sample preparation

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

dry residue

remaining mass fraction of a sample after a drying process under specified conditions

3.2

dry matter content

mass fraction of a sample calculated by the determination of dry residue

4 Principle

The samples are dried to a constant mass in an oven at (105 ± 2) °C. The difference in mass before and after the drying process is used to determine dry matter content. This method applies to solid samples and samples which become solid during the drying process. Volatile compounds volatilizing at temperatures up to and including (105 ± 2) °C are expressed as water using this procedure.

5 Sampling and sample preparation

Sampling and sample preparation are not part of this procedure. Sampling method is given in EN 1482-1 and sample preparation method given in EN 1482-2 shall be use.

6 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.

7 Reagents

7.1 Sand, CAS 14808-60-7

NOTE Other heat stable inert materials, e.g., pumice stone, diatomaceous earth, etc. are also suitable.

8 Apparatus

8.1 Drying system, thermostatically controlled and capable of maintaining temperature of (105 ± 2) °C.

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

8.3 Analytical balance, capable of weighing to the nearest 1 mg or better.

8.4 Evaporating dish or crucible. Temperature tolerant laboratory vessel withstanding at least 110 °C. Suitable materials are nickel, platinum, aluminum, ceramic, borosilicate glass or quartz, porcelain, etc.

9 Procedure

CAUTION — Flammable or explosive gases can be released from some samples during the drying process.

9.1 Procedure A

This procedure is suitable for solid or liquid organic and organo-mineral fertilizers. The procedure is not suitable for some liquid or semi-liquid samples where there is a risk of a cake surface formation or foam formation which hinders an even drying. For these samples Procedure B (9.2) is preferable.

Place an evaporating dish or crucible (8.4) in the drying system (8.1) set at (105 ± 2) °C for a minimum of 30 min. After cooling in the desiccator (8.2) to ambient temperature, weigh the dish or crucible (8.4) to the nearest 1 mg and note as m_a .

Weigh into the evaporating dish or crucible (8.4) 5 g to 10 g of solid sample or 10 g to 30 g of liquid sample. The mass should be adjusted to assure that at least 0,2 g of dry residue will remain after the drying process. Spread the sample to an even depth not exceeding 2 cm. Weigh the loaded dish or crucible (8.4) to the nearest 1 mg and note as m_b . Place the evaporating dish or crucible (8.4) containing the sample into the drying system (8.1) set at (105 ± 2) °C until the residue appears dry, typically overnight but not for more than 20 h. Then follow the procedure described in 9.3.

9.2 Procedure B

This procedure is suitable for liquid organic and organo-mineral fertilizers where there is a risk of a cake surface formation which hinders an even drying.

Weigh approximately 10 g of sand (7.1) into an evaporating dish (8.4), spread the sand to an even depth not exceeding 2 cm evenly and place the dish (8.4) into the drying system (8.1) set at (105 ± 2) °C for a minimum of 120 min. After cooling in the desiccator (8.2) to ambient temperature, weigh the evaporating dish (8.4) containing the dried sand to the nearest 1 mg note as m_a .

Depending on the expected dry residue, weigh into the evaporating dish (8.4) containing the dried sand between 10 g to 30 g of sample and weigh the loaded dish (8.4) to the nearest 1 mg and note as m_b . The mass should be adjusted to assure that at least 0,2 g of dry residue will remain after the drying process. Place the evaporating dish (8.4) containing the sample into the drying system (8.1) set at $(105 \pm 2) ^\circ\text{C}$ until the residue appears dry, typically overnight but not for more than 20 h. Then follow the procedure described in 9.3.

The addition of sand prevents a surface crust from forming and disperses the sample. The amount of sand depends on the sample size and sample properties and may be changed accordingly.

Other heat-stable inert materials e.g., pumice stone, diatomaceous earth diatomaceous earth, etc. are also suitable. Analyst shall ascertain that the inert matrix used, does not give erroneous results for the assay because of decomposition or entrapped moisture loss.

9.3 Procedure A and B

The following applies for both procedures (9.1 and 9.2).

After cooling in the desiccator (8.2) weigh the evaporating dish or crucible (8.4) for the first time. The dry residue shall be regarded as constant if the mass obtained after further 1 h of drying does not differ by more than 0,5 % or 2 mg of the previous value, whichever is the greater (m_c). If the difference is greater, repeat the drying process.

In the case of mass inconstancy after three cycles, the drying process may be stopped (after at least 16 h). The result of the last weighing shall be recorded in the test report.

Other techniques than oven drying, e.g. infrared or halogen lamp drying are allowed, provided they are proven to give comparable results. The technique of choice shall be recorded in the test report.

Ensure that the dry matter is determined using samples identical to those used for determination of parameters that relate to dry matter, by weighing at the same time and from the same sub-sample.

20 h of drying and omission of re-drying as well as re-weighing can be applied for sample types with documented evidence that the necessary drying time is less than 20 h.

10 Quality control

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

11 Calculation and expression of results

The dry residue w is calculated according to Formula (1):

$$w = \frac{m_c - m_a}{m_b - m_a} \cdot f \quad (1)$$

where

- w is the dry matter fraction of the sample, expressed as mass fraction in percent (%) or in grams per kilogram (g/kg);
- m_a is the mass of the empty dish or crucible (including sand, if used), expressed in grams (g);
- m_b is the mass of the dish or crucible (including sand, if used), containing the sample, expressed in grams (g);
- m_c is the mass of the dish or crucible containing the dried sample (including sand, if used), expressed in grams (g);;

f is the conversion factor $f = 100$ for expression of results as dry matter fraction in percent (%) and 1 000 for expression of results in grams per kilogram (g/kg).

12 Test report

The test report shall contain at least the following information:

- a) all information necessary for the complete identification of the sample;
- b) test result;
- b) test method used with reference to this document;
- c) date of sampling and sampling procedure (if known);
- d) date when the determination was finished;
- e) all operating details not specified in this document, or regarded as optional, together with details of any incidents that occurred when performing the method, which might have influenced the test result(s).

Annex A (informative)

Results of the inter-laboratory study

To be included after validation

A.1 Inter-laboratory tests

The precision of the method has been determined in an inter-laboratory study (ILS) with xx participating laboratories from xx EU countries using xx different samples. Further details regarding the outcome of the inter-laboratory study are available in the final validation report [x].

The samples were chosen to represent all typical organic and organo-mineral fertilizers available on the market. The selected samples covered the whole range of dry matter content in organic and organo-mineral fertilizers.

xx different sample materials (x solid and x liquid, x of them blends) were included in the ILS. A detailed description of the materials is given in Table A.1.

Table B.1 — Samples selected for the inter-laboratory study

Sample ID	Sample type

A.2 Statistical results for the determination of dry matter

Statistical evaluation was carried out using a validated software for ILS based on the mathematical algorithms prescribed by [ISO 5725-2](#), *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*. [8] Both methods – direct drying and sand-pan technique were tested. The results are summarized in Table A.2 and Table A.3.

Table A.2 — Results of the statistical evaluation- determinatio n of dry matter by direct dryingSampl e	DM-S- 1	DM-S- 2	DM-S- 3	DM-S- 4	DM-S- 5	DM-S- 6	DM-S- 7	DM-S- 8	DM-S- 9	DM-S- 10
<i>L</i>										
<i>L_A</i>										
<i>N</i>										
<i>N_A</i>										
<i>O</i>										
\bar{x}										
<i>S_R</i>										
<i>S_r</i>										
<i>RSD_R</i>										
<i>RSD_r</i>										
<i>R</i>										
<i>r</i>										
<i>Horrat</i>										
<i>L</i>	Number of participating laboratories									
<i>L_A</i>	Number of laboratories after elimination of outliers									
<i>N</i>	Number of all analytical values									
<i>N_A</i>	Number of analytical values after rejection of outliers									
<i>O</i>	Percentage of outliers (%)									
\bar{x}	Total mean of results (without outliers) (%)									
<i>S_R</i>	Reproducibility standard deviation (%)									
<i>S_r</i>	Repeatability standard deviation (%)									
<i>RSD_R</i>	Relative reproducibility standard deviation (%)									
<i>RSD_r</i>	Relative repeatability standard deviation (%)									
<i>R</i>	Reproducibility limit (2,77 <i>S_R</i>) (%)									
<i>r</i>	Repeatability limit (2,77 <i>S_r</i>) (%)									
<i>Horrat</i>	Horrat index									

Table A.3 — Results of the statistical evaluation- determination of dry matter by sand-pan technique

Sample	DM-S-5	DM-S-6	DM-S-7	DM-S-8	DM-S-9	DM-S-10
L						
L_A						
N						
N_A						
O						
$\bar{\bar{x}}$						
s_R						
s_r						
RSD_R						
RSD_r						
R						
r						
$Horrat$						
L	Number of participating laboratories					
L_A	Number of laboratories after elimination of outliers					
N	Number of all analytical values					
N_A	Number of analytical values after rejection of outliers					
O	Percentage of outliers (%)					
$\bar{\bar{x}}$	Total mean of results (without outliers) (%)					
s_R	Reproducibility standard deviation (%)					
s_r	Repeatability standard deviation (%)					
RSD_R	Relative reproducibility standard deviation (%)					
RSD_r	Relative repeatability standard deviation (%)					
R	Reproducibility limit ($2,77 s_R$) (%)					
r	Repeatability limit ($2,77 s_r$) (%)					
$Horrat$	Horrat index					

Bibliography

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- [1] Regulation (EU) 1009/2019 of the European Parliament and of the Council of 5 June 2019 laying down rules on the making available on the market of EU fertilising products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009 and repealing Regulation (EC) No 2003/2003. Official Journal of the EU, L 170
- [2] EN 12048, *Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at (105 +/- 2)°C (ISO 8190, modified)*
- [3] EN 14787, *Fertilizers and liming materials — Determination of water content — Guidelines and recommendations*
- [4] EN 13040, *Soil improvers and growing media — Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*
- [5] EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*
- [6] ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*
- [7] Bradley R.L. Jr. Moisture and total solid analysis. In: *S. Suzanne Nielsen: Food Analysis*. Springer, Fourth Edition, 2010
- [8] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*