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Organska in organsko-mineralna gnojila - Razklop z zlatotopko za določevanje elementov

Organic and organo-mineral fertilizers - Digestion by aqua regia for subsequent determination of elements

Organische und organisch-mineralische Düngemittel - Aufschluss durch Königswasser zur anschließenden Bestimmung der Elemente

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Organic and organo-mineral fertilizers - Digestion by aqua regia for subsequent determination of elements

Organische und organisch-mineralische Düngemittel - Aufschluss durch Königswasser zur anschließenden Bestimmung der Elemente

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (FprCEN/TS 17768:2021) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document is currently submitted to the Vote on TS.

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Introduction

Aqua regia is applied to digest different matrices for the subsequent determination of many elements. For example, a multi-matrix standard for aqua regia extraction of soils, sludges and biowaste was prepared by CEN/TC 444 “Environmental characterization”. A similar procedure was applied for determination of aqua regia extractable elements according to FprCEN/TS 17769 and FprCEN/TS 17770. CEN/TC 223 “Soil improvers and growing media” published a standard for a similar procedure for soil improvers and growing media. Wide use of the aqua regia digestion, availability of the instruments and the possibility to merge the standards for different matrices in the future, were the main reasons for also applying this method of digestion for organic and organo-mineral fertilizers.

1 Scope

This document specifies the procedure for the digestion of different organic fertilizers and organo-mineral fertilizers with aqua regia to enable a subsequent determination of elements.

The extracts are suitable for analysis using FprCEN/TS 17770 and FprCEN/TS 17769.

NOTE Alternatively, inductively coupled plasma mass spectrometry (ICP-MS) can be used for the measurement if the user proves that the method gives the same results.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

4 Principle

The samples are digested by boiling in aqua regia.

5 Sampling and sample preparation

Sampling should be performed carefully, following the principles described in EN 1482 (all parts) with appropriate adaptations, required to account for specificities of organic and organo-mineral fertilizers.

6 Reagents

All reagents should be of recognized analytical grade. The reagents and water used shall have negligible concentrations of the elements to be determined if compared to the lowest concentrations of these elements in the sample solution.

6.1 Hydrochloric acid 37 % HCl, $c(HCl) \approx 12$ mol/l, $\rho \approx 1{,}18$ g/ml.

6.2 Nitric acid 65 %, $c(HNO_3) \approx 14{,}3$ mol/l, $\rho \approx 1{,}4$ g/ml.

6.3 Antifoaming agent, e.g. *n*-dodecane ($C_{12}H_{26}$) or octanol ($C_8H_{18}O$) are suitable.

7 Apparatus

7.1 Common laboratory glassware

All glassware and plastic ware shall be adequately cleaned and stored to avoid any contamination.

7.2 Apparatus for thermal heating digestion,

Temperature is controlled with reaction vessel and reflux condenser. The capacity of the reaction vessel should be at least 5 times of the volume of the aqua regia used.

7.3 Filter paper,

Paper ash free and of recognized and tested quality.

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

8 Procedure

CAUTION – Suitable precautions shall be taken to avoid any contact of laboratory staff with acid fumes. The digestion procedure shall be carried out in a well-ventilated fume cupboard.

8.1 Sample digestion

This procedure can be used for all samples of organic fertilizers and organo-mineral fertilizers if a sufficiently sensitive method for determination is used. For some liquid samples with a low dry matter content (FprCEN/TS 00260236 shall be used), a pre-concentration step (8.2) may be necessary.

Weigh 2,5 g to 5,0 g of the solid, or liquid sample representing approximately 1,5 g to 3,0 g of dry sample, to the nearest 0,001 g. Transfer the sample quantitatively to the reaction vessel (7.2). Liquid samples are preferably weighed directly into the reaction vessel (7.2). Moisten the solid sample with about 0,5 ml to 1,0 ml of water and add, while gently mixing, 21 ml of hydrochloric acid (6.1) followed by 7 ml of nitric acid (6.2) drop wise if necessary. Add one drop of the antifoaming agent (6.3) in the case of excessive foaming. Connect the condenser to the reaction vessel and let the mixture stand at laboratory temperature until any effervescence almost ceases. Transfer to the heating device and slowly raise the temperature of the reaction mixture to reflux conditions and maintain for 2 h ensuring that the condensation zone is lower than 1/3 of the height of the condenser. Then allow to cool and rinse the condenser with 10 ml of water.

Transfer the content of the reaction vessel quantitatively into a 150 ml volumetric flask, dilute to the mark with water and mix well.

Test solutions can be filtered or centrifuged if necessary. Use ash-free filter paper of recognized and tested quality (7.3) and discard the first portion of the filtrate (approximately 20 ml).

Carry out the measurement immediately or store the extracts in tightly closed vessels for up to 15 days.

The procedure can be modified for the use of 100 ml or 50 ml volumetric flasks. In this case the mass of the sample and volumes of the acids should be changed accordingly. For more information, see Annex A.

NOTE 1 Weigh the samples for dry matter determination according to FprCEN/TS 17773 at the same time and from the same sub-sample to ensure that the dry matter is determined on the samples identical to those used for determination of parameters that relates to dry matter.

NOTE 2 If excessive foaming occurs despite the addition of an anti-foaming reagent, it is possible to leave the samples overnight after the addition of the acids to allow a slow oxidation or to use a digestion block with two independent heating zones.

NOTE 3 The presence of small quantities of organic matter after digestion will not affect determination of the elements by ICP-AES and it is not necessary to apply additional organic matter removal.

NOTE 4 Alternatively, digestion by the use of microwave technique is possible.

8.2 Sample pre-concentration

This procedure is necessary for liquid samples with low dry matter content in the case that the measurement method is not sufficiently sensitive. See Annex A for calculation of the initial sample mass to be taken for the pre-concentration step to achieve concentrations of the individual elements above the limit of quantification.

Weigh approximately 10 g to 50 g of the liquid sample, representing 1 g to 3 g of dry sample, to the nearest 0,001 g directly into the reaction vessel (7.2) and heat the vessel to approx. 75 °C to evaporate excessive water. After evaporation to approx. 5 ml and cooling, follow procedure 8.1. Adjustment of the pre-concentration step for different final volumes is given in Annex A.

NOTE Evaporation of a large volume of sample usually takes several hours. Higher temperature (up to 95 °C) for faster evaporation can be used if the final digestate is not used for the determination of mercury. The fastest evaporation can be achieved by using a 250 ml beaker placed on a hot plate for evaporation and consequently also for digestion under a watch glass.

8.3 Blank

Prepare a blank test solution following the same procedure as for samples. The measurement of a blank is necessary to determine the contribution of the extracting solution, glassware and filter paper used.

Annex A
(informative)

Adjustment to different digestion procedures and measurement apparatuses

A.1 Adjustment of the procedure to different final volumes

Volumetric flasks or graduated plastic tubes for 150 ml, 100 ml, or 50 ml may be used for the final volume adjustment. In Table A.1 there are given recalculated parameters for the different final volumes.

Table A.1 — Recalculated parameters for different final volumes

VF [ml]	HNO_3 [ml]	HCl [ml]	*m* [g]	VM [ml]
150	7	21	1,5 - 3	6
100	4,7	14	1 to 2	4
50	2,3	7	0,5 to 1	2
VF – final volume of a volumetric flask or a graduated plastic tube *m* – mass of the sample recalculated to dry matter VM – maximum volume after pre-concentration step (approx.)				

A.2 Calculation of the sample mass

A.2.1 General

According to the given legislative limits and the limits of quantification for the individual elements and the measurement apparatus, the mass of the sample should be adjusted to ensure that the concentration of the element in the final digestate is above the limit of quantification (see Table A.2). The calculation of the mass of the sample is necessary only for the elements with a low legislation limit and/or very high LOQ (limit of quantification). Usually only for cadmium, in some cases also for arsenic and nickel. The highest volume calculated for different elements will be applied.

Table A.2 — Legislative limits and typical limits of the quantitative determination

Element	Cd	Pb	Hg	As	Cr	Cu	Ni	Zn
Legislative limit [mg/kg]	1,5	120	1	40	200	600	50	1500
Legislative limit [µg/l]	30	2400	20	800	4000	12000	1000	30000
LOQ [µg/l]	7,5	100	1,0	75	75	75	75	250
Legislative limits are from Regulation (EU) 2019/1009 of the European Parliament and of the Council Legislative limit in [µg/l] was calculated for 1 g sample (dry matter) in 50 ml LOQ – example of typical values for an ICP-AES instrument, radial view LOQ – example of a limit for Hg is given for a direct amalgamation technique								