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BSI Group Headquarters

389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000

Fax: +44 (0)20 8996 7400

www.bsigroup.com

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Responsible Committee Secretary: Ms Lachean Humphreys (BSI)

Direct tel:

E-mail: lachean.humphreys@bsigroup.com

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

For national standards, comments will be reviewed by the relevant UK national committee and the resulting standards published as a British Standard.

UK Vote

Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
- All comments should be submitted online at <https://standardsdevelopment.bsigroup.com/>. You will need to register in order to comment.

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

English Version

Soil improvers and growing media - Digestion by aqua regia for subsequent determination of elements

Amendements du sol et supports de culture - Digestion
à l'eau régale pour le dosage ultérieur des éléments

Bodenverbesserungsmittel und Kultursubstrate -
Aufschluss mit Königswasser zur anschließenden
Bestimmung der Elemente

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 223.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 13650:2025) has been prepared by Technical Committee CEN/TC 223 “Soil improvers and growing media”, the secretariat of which is held by NEN.

This document is currently submitted to CEN Enquiry.

This document will supersede EN 13650:2001.

prEN 13650:2025 includes the following significant technical changes with respect to EN 13650:2001:

- the heating block procedure has been added to the reflux digestion procedure;
- the scope has been expanded including liming materials and preformed materials.

This document has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

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 digestion of soil, treated biowaste, sludge and waste was prepared by CEN/TC 444 “Environmental characterization of solid matrices”. CEN/TC 260 “Fertilizers and liming materials” published a standard with a similar procedure for organic and organo-mineral fertilizers.

It is known that the digestion of soil improvers or growing media with aqua regia will not necessarily lead to complete element recoveries and that the extract from a test sample may not reflect the total concentrations of the target analytes. However, for most applications the result obtained based upon digestion methods specified in this document are considered to be fit for the intended purpose.

1 Scope

This document specifies a method for the digestion of soil improvers and growing media using an aqua regia digestion.

Materials containing more than about 28 % (m/m) organic matter will require treatment with additional nitric acid. With high solute concentrations in extract solutions, spectral interferences and background enhancement can be expected.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TS 17732:2022, *Soil improvers and growing media — Terminology*

EN 12579:2024, *Soil improvers and growing media — Sampling*

prEN 13039:2025, *Soil improvers and growing media — Determination of organic content and ash*

prEN 13040-1:2025, *Soil improvers and growing media — Sample preparation — Part 1: Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory bulk density*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 17732:2022 and the following apply.

3.1

aqua regia

digestion solution obtained by mixing 1 volume of nitric acid (mass fraction of 65 % to 70 %) and 3 volumes of hydrochloric acid (mass fraction of 35 % to 37 %)

3.2

digestion

mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with a reagent mixture

4 Principle

A test portion is digested with aqua regia according to one of the following heating procedures:

- Method A: reflux for 120 min $\pm$ 10 min, followed by filtration/centrifugation;
- Method B: heating block at 105 °C $\pm$ 5 °C for 120 min + 10 min, followed by filtration/centrifugation.

5 Reagents

5.1 General

Use only acids and reagents of recognized analytical grade to avoid high blank values for subsequent analytical measurements. Use a test blank solution throughout the procedure applying all steps with the same amount of acids, but without a sample.

5.2 Water, having an electrical conductivity not higher than 0,2 mS/cm at 25 °C, free of the elements to be determined.

5.3 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$; $\rho \approx 1,18 \text{ g/ml}$; 37 % mass/volume.

5.4 Nitric acid, $c(\text{HNO}_3) = 15 \text{ mol/l}$; $\rho \approx 1,42 \text{ g/ml}$; not less than 65 % mass/volume.

5.5 Nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$. Dilute 35 ml nitric acid (5.4) to 1 l with water (5.2).

5.6 Antifoaming agent, e.g. *n*-dodecane ($\text{C}_{12}\text{H}_{26}$) or *n*-octanol ($\text{C}_8\text{H}_{18}\text{O}$) are suitable.

6 Apparatus

6.1 General

Usual laboratory apparatus. All glassware and plastics ware shall be adequately cleaned and stored to avoid any contamination.

Depending upon the concentration of the element of interest, particular care should be exercised with respect to the effective cleaning of the vessels.

6.2 Method A — Thermal heating under reflux conditions

6.2.1 Digestion vessel, temperature-resistant and capable of containing the mixture of sample and digest solution, for example a quartz vessel. The digestion vessel shall have a volume of at least 5 times of the volume of the aqua regia used. The inner wall of the vessel shall be inert and shall not release substances to the digest in excess of the purity requirements of the subsequent analysis.

Silica or borosilicate glass vessels may be used instead of quartz vessels.

It may be necessary to periodically clean the digestion vessels with a suitable surfactant to remove persistent deposits.

6.2.2 Reflux condenser, adaptable to the digestion vessel (6.2.1).

6.2.3 Absorption vessel, volatile species trap, in an open digestion system capable of trapping one or more volatile measurement species, adaptable to the reflux condenser (6.2.2).

6.2.4 Heating device, for example a heating mantle, thermostatic controlled, or an aluminium block thermostat.

6.3 Method B — Thermal heating with a heating block with containers

6.3.1 Digestion tube, 50 ml plastic tube, temperature-resistant and capable of containing the mixture of sample and digest solution, with a screw cap from e.g. polypropylene.

The part of the tube not being heated and the screw cap function as a condenser but are not really a reflux system. The material of the tube and screw cap need to be tested to be sure that release of elements of

interest does not take place. Other materials and vessels with other volumes than mentioned above are allowed to be used if suitability has been proven.

6.3.2 Temperature controlled heating block, heating block able to heat the tube(s) to a temperature of $105\text{ °C} \pm 5\text{ °C}$.

6.4 Sample containers, plastics and glass containers are both suitable.

6.5 Filter paper, usually with a pore size of $0,45\text{ }\mu\text{m}$ and resistant to the diluted aqua regia final digestion solution.

6.6 Volumetric flasks, usually of nominal capacity of 50 ml or 100 ml.

6.7 Analytical balance, with an accuracy of 1 mg or better.

6.8 Boiling aids, anti-bumping granules or glass beads, diameter 2 mm to 3 mm, acid washed.

7 Sample pre-treatment

Prepare the solid sample by reducing the particle size below 0,5 mm in accordance with Clause 10 of prEN 13040-1:2025. The residual moisture content in the analysed sample is determined in accordance with prEN 13040-1:2025, Clause 11, using 10 g of sample.

Prepare the liquid samples in accordance with Clause 9 of prEN 13040-1:2025. Liquid samples may be digested as such or after drying at 70 °C . The (residual) moisture content in the analysed sample is determined in accordance with prEN 13040-1:2025, Clause 11, using 10 g of sample.

8 Sampling

Sampling is not part of the method specified in this document. Follow EN 12579:2024 dealing with soil improvers and growing media. It is important that the laboratory receives a sample that is representative of the product under consideration. The sample should not have been damaged or changed during transport or storage.

9 Procedure

9.1 Safety

WARNING — Persons using this document should be familiar with usual laboratory practice. Some of the reagents used in this document are highly corrosive and very toxic. Safety precautions are absolutely necessary.

All procedures should be performed in a fume hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high organic content. Avoid contact with the chemicals and the gaseous reaction products.

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

9.2 Blank test

Carry out a reagent blank test digestion in parallel with the determination, using the same procedure and the same quantities of all the reagents as in the determination, but omitting the test portion. The laboratory shall define acceptable limits.

NOTE The measurement of a blank is introduced to determine the contribution of the extracting solution, glassware, digestion tube and filter paper used to the measured value.

9.3 Method A — Thermal heating under reflux conditions

This procedure may be used for all samples of soil improvers and growing media if a sufficiently sensitive method for determination is used. For some liquid samples with a low dry matter content, a higher intake volume or a pre-concentration step may be necessary (see Annex A). Pre-concentration may be carried out by heating an amount of liquid sample to approximately 70 °C to evaporate excessive water. The pre-concentration factor shall be taken into account in the final calculations.

Weigh approximately 1 g to 3 g, to the nearest 0,001 g of the test sample, and transfer to the digestion vessel (6.2.1). In case of dry samples moisten the test portion with about 0,5 ml to 1,0 ml water (5.2). Add while mixing, 21,0 ml $\pm$ 0,1 ml of hydrochloric acid (5.3) followed by 7,0 ml $\pm$ 0,1 ml of nitric acid (5.4), drop by drop if necessary to reduce foaming. Connect the reflux condenser (6.2.2) to the digestion vessel (6.2.1). Fill the absorption vessel (6.2.3) with approximately 15 ml nitric acid (5.5). Connect the absorption vessel to the reflux condenser and let stand at room temperature until any effervescence almost ceases to allow for slow oxidation of the organic matter in the sample.

The time of standing at room temperature can have an influence on the digestion rate of aqua regia. For consistency, it is recommended to start heating as soon as possible after the first strong reaction has ceased.

NOTE It is common practice to add hydrochloric acid directly to the reaction vessel, followed by the nitric acid, and to assume that the aqua regia forms in the reaction vessel. However, problems have been encountered with materials rich in carbonates, where a substantial portion of the hydrochloric acid has been consumed before the nitric acid can be added. Under such circumstances, the correct formation of aqua regia is doubtful.

The amount of aqua regia is sufficient only for oxidation of about 0,85 g of organic matter (corresponding to about 0,5 g of organic carbon). If there is any doubt about the amount of organic matter present, carry out a determination according to prEN 13039:2025. If there is more than 0,85 g of organic matter in the test sample reduce the quantity of sample analysed or proceed as follows.

Allow the first reaction with aqua regia to subside. Then add an extra 1 ml of nitric acid (5.4) only to every 0,17 g of organic matter above 0,85 g. Do not add more than 10 ml of nitric acid at any time and allow any reaction to subside before proceeding further.

Add a few roughened glass beads (6.8) and connect the digestion vessel (6.2.1) to the heating device (6.2.4). Raise the temperature of the reaction mixture slowly until reflux conditions are reached 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. Add the content of the absorption vessel to the digestion vessel via the reflux condenser, rinsing both the absorption vessel and condenser with further 10 ml of diluted nitric acid (5.5).

Transfer quantitatively the solution content of the digestion vessel into a suitable sized volumetric flask and add water (5.2) to the volume mark.

Alternatively, another procedure may be applied, such that the adjustment to volume with the solid residue still present shall be carried out immediately after digestion.

If the measurement solution contains particles due to precipitation which may clog nebulizers or interfere with an injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered (6.5).

The extracts are suitable for analysis using prEN 18259:2025 and prEN 18258:2025.

9.4 Method B — Thermal heating with a heating block with containers

This procedure may be used for all samples of soil improvers and growing media if a sufficiently sensitive method for determination is used. For some liquid samples with a low dry matter content, a higher intake volume or a pre-concentration step may be necessary (see Annex A). Pre-concentration may be carried out by heating an amount of liquid sample to approximately 70 °C to evaporate excessive water. The pre-concentration factor shall be taken into account in the final calculations.

Weigh an amount of not more than 2 g of the test portion (typically 0,5 g to 1 g of dry sample), to the nearest 0,001 g, and transfer to the digestion vessel (6.3.1). In case of dry samples moisten the test portion with a few drops of water (5.2). Add 12,0 ml + 0,1 ml hydrochloric acid (5.3) followed by 4,0 ml + 0,1 ml nitric acid (5.4). Let stand at room temperature until any effervescence almost ceases to allow for slow oxidation of the organic matter in the sample.

The amount of aqua regia is sufficient only for oxidation of about 0,50 g of organic matter (corresponding to about 0,3 g of organic carbon). If there is any doubt about the amount of organic matter present, carry out a determination according to prEN 13039:2025. If there is more than 0,50 g of organic matter in the test sample reduce the quantity of sample analysed or proceed as follows.

Allow the first reaction with aqua regia to subside. Then add an extra 1 ml of nitric acid (5.4) only to every 0,17 g of organic matter above 0,50 g. Do not add more than 10 ml of nitric acid at any time and allow any reaction to subside before proceeding further.

NOTE For some elements, e.g. chromium, the additional volume of HNO_3 is essential in order to have a sufficient recovery upon digestion.

The time of standing at room temperature can have an influence on the digestion rate of aqua regia. For consistency, it is recommended to start heating as soon as possible after the first strong reaction has ceased.

Loosely screw on the tube cap (not very tight!) and place the digestion vessel on the heating block (6.3.2) and slowly increase the temperature to the boiling point. Keep the temperature on the boiling point during 120 min + 10 min.

Let the vessel cool down to room temperature and fill up with water (5.2) to the volume mark.

If a non-graduated digestion tube is used, transfer quantitatively the solution into a suitable sized volumetric flask and add water (5.2) to the volume mark.

Alternatively, another procedure may be applied, such that the adjustment to volume with the solid residue still present shall be carried out immediately after digestion.

If the measurement solution contains particles due to precipitation which may clog nebulizers or interfere with an injection of the sample into the instrument, the sample may be centrifuged or filtered (6.5).

The extracts are suitable for analysis using prEN 18259:2025 and prEN 18258:2025.

10 Test report

The test report may be prepared separately or in conjunction with the test report of the subsequent analytical method.

The test report shall include the following information:

- a) the digestion method used, together with a reference to this document (including its year of publication);
- b) a complete identification of the sample;