SLOVENSKI STANDARD
SIST-TS CEN/TS 16177:2012

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Blato, obdelani biološki odpadki in tla - Določevanje amonija, nitrata in nitrita, sposobnih ekstrakcije

Sludge, treated biowaste and soil - Extraction for the determination of extractable ammonia, nitrate and nitrite

Schlamm, behandelter Bioabfall und Boden - Bestimmung von extrahierbarem Ammoniumstickstoff, Nitrat- und Nitritstickstoff

Boues, bio-déchets traités et sols - Extraction pour la détermination de l'azote nitrique et ammoniacal extractible

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TECHNICAL SPECIFICATION

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English Version

Sludge, treated biowaste and soil - Extraction for the determination of extractable ammonia, nitrate and nitrite

Boues, biodéchets traités et sols - Extraction pour la détermination de l'azote nitrique et ammoniacal extractible

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Foreword

This document (CEN/TS 16177:2012) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

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

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This Technical Specification is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

Table 1 — Matrices for which this Technical Specification is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Fresh compost Compost
Soil	Sludge amended soil Agricultural soil

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

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

1 Scope

This Technical Specification specifies a procedure for the determination of ammonium nitrogen and nitrate nitrogen in sludge, treated biowaste and soil after extraction with a 1 mol/l potassium chloride solution. The extraction method is suitable for fresh samples.

The determination of nitrogen fractions can be done manually or by automated methods.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 11732, *Water quality — Determination of ammonium nitrogen — Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732)*

ISO 7150-1, *Water quality — Determination of ammonium — Part 1: Manual spectrometric method*

ISO 14256-2, *Soil quality — Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution — Part 2: Automated method with segmented flow analysis*

3 Terms and definitions

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

3.1
nitrogen fractions
mass of ammonium nitrogen and nitrate nitrogen (mineral nitrogen) that is released after a single or repeated extraction of the sample using 1 mol/l potassium chloride solution

4 Principle

An aliquot of the homogenised fresh material is shaken for 1 h with 1 mol/l potassium chloride solution at room temperature. The ratio of extractant to material varies according to the material tested. The extraction solution is centrifuged or filtered and an aliquot of the filtrate is analysed by flow injection analysis (FIA) or continuous flow analysis (CFA) or by manual methods as distillation and titration or spectrophotometric method.

5 Interferences and sources of errors

The samples can change composition due to biological and/or chemical activity. The fresh or the deep frozen homogenised test sample is directly transferred to the extraction bottle, which is filled with the potassium chloride solution, if a change in the content of the nitrogen fractions can be expected. Drying of the material, even rapid microwave drying will result in a change of the nitrogen content especially of ammonium. Take care to use purified glassware and equipment and filter papers free of contaminations with nitrate and ammonium. Cleaning of glassware with water shall be performed after each use, especially to avoid cross contaminations from samples with high contents of nitrogen fractions, like sludge or biowaste. Use separate equipment for the analysis of soil samples, as contents of nitrogen fractions can be near the detection limit. A blank test shall be carried out to assure purity of reagents and equipment.

6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

6.1 Water, grade 2 according to EN ISO 3696.

6.2 Potassium chloride, c(KCl) = 1 mol/l.

Dissolve 373 g of KCl, dried at 105 °C, in approximately 3 l water (6.1) and dilute to 5 l with water (6.1).

7 Apparatus

Usual laboratory apparatus, and in particular the following:

7.1 Analytical balance with an accuracy of at least 0,01 g.

7.2 Wide necked glass or plastic bottles with secure stopper or caps, nominal volume 250 ml or 500 ml or other.

The material shall not adsorb ammonium, nitrate or nitrite and shall not be contaminated with these compounds.

7.3 Shaking apparatus, end-over-end shaker, frequency 30 min^{-1} to 40 min^{-1} or other appropriate shakers.

7.4 Filter paper, free of nitrogen fractions, pore size 8 µm to 12 µm.

8 Procedure

8.1 Preparation

Pretreat the samples according to EN 16179, if not otherwise specified.

When the content of mineral nitrogen is determined in deep-frozen samples, the temperature and the duration of the thawing process shall be controlled. The samples can be thawed at room temperature, if they are homogenised and extracted within 4 h after beginning of thawing. Thawing at 4 °C is also possible, but the thawing period should not exceed 48 h. The fresh test sample can be homogenised by manual methods which shall be performed in a way that avoids contamination of the test sample with nitrogen fractions.

NOTE The use of gloves is recommended when homogenising test samples.

8.2 Extraction

Transfer a known amount of the homogenised test sample (equal to 1,0 g to 10,0 g dry mass) into an extraction bottle (7.2), add potassium chloride solution (6.2) in a mass (dry mass of the sample, m) to volume (of the extracting solution, V) ratio of (m/V):

— 1 to 5 (m/V) for field moist fresh soil (< 10 mm); or

— 1 to 10 (m/V) for dry soil samples (< 2 mm); and

— 1 to 20 up to 1 to 80 (m/V) for treated biowaste, sludge or sludge amended soils.

Close the bottle cap and place the extraction bottle in the shaking apparatus (7.3). Shake it for 1 h at room temperature. A minimum of one repetition of the extraction process after filtration is necessary for dry soil samples < 250 µm, dried biowaste and sludge samples. Drying leads to changes in the original composition of

nitrogen fractions and shall be avoided. The amount of test sample is related to the homogenising procedure. Take care, that the test sample is a homogeneous part of the collected sample and the laboratory sample.

NOTE The amount of test sample can be adapted to macromethods – the ratio of sample to extractant should be the same as stated above. The used extraction temperature should be noted in the test report, as temperature affects the effectiveness of the extraction.

8.3 Filtration

Filter the extraction solution through a filter (7.4). Discard the first 10 ml and collect an aliquot from the subsequent filtrate for determination of the nitrogen fractions.

NOTE Centrifugation is recommended for samples, which are subjected to repeated extractions.

The analysis of the nitrogen fractions shall be done as soon as possible. Because the high concentration of potassium chloride avoids biological activity, the filtrates can be stored at 4 °C for at least seven days.

8.4 Determination

Determine the ammonium nitrogen and ammonium concentration according to EN ISO 11732, ISO 14256-2 or ISO 7150-1.

NOTE Other methods of determination may be used provided equivalence of results can be shown.

8.5 Calibration

Calibrate the analytical part according to the standard used for the determination, using ammonium and nitrate in inorganic salts, e. g. ammonium chloride and potassium nitrate.

8.6 Blank determination

Carry out at least two blank determinations in each series and use the average blank value for subsequent calculations. Blank determinations are carried out by using 1 mol/l potassium chloride (6.2) without sample addition throughout the whole procedure.

9 Calculation and expression of results

9.1 Method of calculation

Calculate the ammonium nitrogen and nitrate nitrogen according to the standard used for the determination.

9.2 Expression of results

The results of extractable ammonium nitrogen and nitrate nitrogen are expressed in milligrams per kilogram dry matter. The dry matter is determined according to the standard used for the determination.

10 Precision

The performance characteristics of the method data have been evaluated (see Annex A).