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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

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

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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.