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English Version

Sludge treated biowaste and soil - Determination of
nonylphenols (NP) and nonylphenol-mono- and diethoxylates
using gas chromatography with mass selective detection (GC-
MS)

Boues, biodéchets traités et sols - Détermination des
nonylphénols et nonylphénol-mono- et di-éthoxylates par
chromatographie en phase gazeuse avec détection
sélective de masse (GC-MS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung
von Nonylphenolen (NP) und Nonylphenol-Mono- und
Diethoxylaten mittels Gaschromatographie mit
massenselektiver Detektion (GC-MS)

This Technical Specification (CEN/TS) was approved by CEN on 24 April 2011 for provisional application.

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Foreword

This document (CEN/TS 16182:2012) 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

Nonylphenols (NP) are mainly found in the environment as degradation products of nonylphenol polyethoxylates (NPEO). NPEO have many applications as non-ionic detergents in washing and cleaning agents.

After use NPEO are degraded by de-ethoxylation, resulting in polyethoxylates with fewer ethoxy-groups. Nonylphenol-diethoxylates (NP2EO), nonylphenol-monoethoxylates (NP1EO) and nonylphenols (NP) are the last three products in the degradation chain. Due to their significant presence in sewage sludge, all three components are included in this Technical Specification.

This Technical Specification is applicable for several types of matrices and validated for municipal sewage sludge (see also Annex A for the results of the validation).

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 method for the determination of nonylphenols (NP), nonylphenol-monoethoxylates (NP1EO) and nonylphenol-diethoxylates (NP2EO) in sludge, treated biowaste and soil using GC-MS.

For sludge a limit of detection of 0,1 mg/kg and for soil and treated biowaste 0,02 mg/kg (expressed as dry matter) may be achieved.

Lower limits of detection may be achieved by concentrating the extract by solvent evaporation.

NOTE 4-tert-octylphenol can also be analysed with this method.

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 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

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

EN ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis (ISO 16720)*

EN ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry (ISO 22892)*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

3 Principle

After pretreatment, the test sample is extracted by shaking with a mixture of acetone and petroleum ether (1:1). If necessary, interfering compounds are removed from the extract by a clean-up on a suitable column.

The extract is treated with *N*-methyl-*N*-(trimethylsilyl)-trifluoroacetamide (MSTFA) reagent for the derivatization (silylation) of the analytes, and subsequently analysed by gas chromatography and mass selective detection (GC-MS).

Nonylphenols and nonylphenol-mono- and diethoxylates are identified from the GC fingerprint, the relative retention times and the relative intensities of two diagnostic ions. The quantification is based on an internal standard procedure. The internal standards (¹³C-labelled 4-n-NP and ¹³C-labelled 4-n-NP2EO) are taken through the whole analytical procedure.

4 Interferences

4.1 Interferences from sampling

Use sampling containers of materials (preferably glass or steel) that do not significantly affect the sample during the contact through sampling and storage. Plastic containers may be used if it has been proven that they do not significantly affect the sample.