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English Version

Sludge, treated biowaste and soil - Determination of linear alkylbenzene sulfonates (LAS) by high-performance liquid chromatography (HPLC) with fluorescence detection (FLD) or mass selective detection (MS)

Boues, biodéchets traités et sols - Détermination des alkylbenzènesulfonates linéaires (LAS) par chromatographie liquide à haute performance (CLHP) avec détection par fluorescence (FLD) ou détection sélective de masse (SM)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von linearen Alkylbenzolsulfonaten (LAS) mittels Hochleistungs-Flüssigkeitschromatographie (HPLC) mit Fluoreszenzdetektion (FLD) oder massenselektiver Detektion (MS)

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

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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Contents

Page

Foreword

This document (CEN/TS 16189:2012) 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

The anionic surfactant LAS (Linear Alkylbenzene Sulfonate) is found in the environment due to the use of LAS in detergents. For more than 30 years LAS has been the largest single surfactant used in detergents, and the use continues on a high level.

Although LAS is readily biodegradable during wastewater treatment, considerable amounts may still be found in sludge of municipal origin. By the use of sludge for soil improvement LAS may end up in the agricultural soil, where a rapid biodegradation takes place.

The method describes the determination of LAS in sludge, soil, treated biowaste and neighbouring fields. LAS is the sodium salt of alkylbenzene sulfonic acids, and it consists of a mixture of the homologues C_{10}-LAS, C_{11}-LAS, C_{12}-LAS, C_{13}-LAS and C_{14}-LAS. LAS is determined as the sum of the homologues.

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 sewage sludge
Biowaste	Fresh compost
Soil	Sludge amended 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 method for the determination of linear alkylbenzene sulfonate (LAS) in sludge, treated biowaste and soil using high-performance liquid chromatography (HPLC) with a fluorescence detector (FLD) or a mass selective detector (MS).

This Technical Specification specifies the determination of the sum of LAS. Under the conditions specified in this Technical Specification, typically a limit of detection of 20 mg/kg (expressed as dry matter) for sludge and of 0,2 mg/kg to 0,5 mg/kg for soil and treated biowaste may be achieved.

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

NOTE The single LAS homologues C_{10} to C_{14} can be determined by this Technical Specification.

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 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 Terms and definitions

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

3.1
analyte
mixture of homologues (i. e. C_{10}-LAS, C_{11}-LAS, C_{12}-LAS, C_{13}-LAS and C_{14}-LAS) where each homologue consists of a mixture of four to six isomers depending on the length of the alkyl group

Note 1 to entry: The dominant homologues in detergents and environmental samples are C_{11}-LAS and C_{12}-LAS. C_{10} to C_{14} refers to the chain length of the linear alkyl group.

4 Principle

After pretreatment, the test sample is extracted by shaking with methanol. If necessary, interfering compounds are removed from the extract by a clean-up on a suitable column.

The extract is analysed by high performance liquid chromatography (HPLC) on a C_8- or C_{18}-column and detection by fluorescence (FLD) or mass spectrometry (MS).

The identification is based on the retention times of the homologues and of the isomers of each homologue. Another identification point is the pattern/fingerprint of the homologues, and the isomer fingerprint of each homologue if a C_{18}-column is used for HPLC. By use of MS detection the relative intensities of two diagnostic ions may also be used for the identification (optional).