SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 16183:2011

01-februar-2011

Blato, obdelani biološki odpadki in tla - Določevanje izbranih ftalatov s kapilarno plinsko kromatografijo z masno selektivno detekcijo (GC-MS)

Sludge, treated biowaste and soil - Determination of selected phthalates using capillary gas chromatography with mass spectrometric detection (GC-MS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung ausgewählter Phthalate mittels kapillarer Gaschromatographie mit massenspektrometrischer Detektion (GC-MS)

Boue, biodéchet traité et sol - Détermination de certains phtalates par chromatographie en phase gazeuse/spectrométrie de masse (GC-MS)

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ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

FINAL DRAFT FprCEN/TS 16183

December 2010

ICS

English Version

Sludge, treated biowaste and soil - Determination of selected phthalates using capillary gas chromatography with mass spectrometric detection (GC-MS)

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This draft Technical Specification is submitted to CEN members for Technical Committee Approval. It has been drawn up by the Technical Committee CEN/TC 400.

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

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Foreword

This document (FprCEN/TS 16183:2010) 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.

This document is currently submitted to the Formal Vote.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This Technical Specification is part of a modular horizontal approach in which this document belongs to the analytical step.

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.

Until now, test methods determining properties of materials within the environmental area were prepared in Technical Committees (TCs) working on specific products/matrices (e.g. soil, waste, sludge). However, it is understood that many steps within individual test procedures may also be used for the analysis of various other materials. By careful determination of these steps and selection of specific questions within these steps, elements of the test procedure can be described in a way that can be used for a variety of matrices and materials with certain specifications. This optimization is in line with the development among end-users of standards. A majority of routine environmental analyses are carried out by institutions and laboratories working under a scope that is not limited to one single environmental matrix but covers a wide variety of matrices. Availability of standards covering more matrices contributes to the optimization of laboratory procedures and standard maintenance costs, e.g. costs related to accreditation and recognition.

A horizontal modular approach was developed in the project "Horizontal". "Modular" means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). A beneficial feature of this approach is that individual "modules" can be replaced by improved ones without jeopardizing the standard "chain".

The results of the desk study as well as the evaluation and validation studies have been subject to discussions with all parties concerned in the CEN structure during the development by project "Horizontal". The results of these consultations with interested parties in the CEN structure have been presented to and discussed in CEN/TC 400.

Based on data from interlaboratory studies and consultations with interested parties within CEN member bodies, it has been concluded that this FprCEN/TS 16183 is acceptable for its intended use and is ready for FV.

It is recognized that standardization in the environmental field in most national standardization bodies is organized in national standardization committees that mirror the vertical structure of technical committees in the environmental field in CEN. The present FV therefore asks for special attention by the NSBs to assure that the relevant and interested parties are consulted during the FV, i.e. to assure that one single consolidated enquiry reply on this FprCEN/TS 16183 can be presented by the NSB that covers the entire scope of this Technical Specification.

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Introduction

This Technical Specification is (applicable and) validated for several types of matrices as indicated below (see also Annex D for the results of the validation).

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

Matrix	Validated for
Sludge	Municipal sludge
Compost	Fresh compost
Soil	Sludge amended soil

WARNING — Persons using this Technical Specification should be familiar with normal 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.

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1 Scope

This Technical Specification specifies a method for the determination of selected phthalates in sludge, treated biowaste and soil, after extraction and gas chromatographic analysis with mass spectrometric detection.

The method is applicable for the determination of phthalates (see Table 2) at the lowest mass content of 0,1 mg/kg to 0,5 mg/kg (expressed as dry matter), depending on the individual substance.

The applicability of the method to other phthalates not specified in Table 2 is not excluded except the isomeric mixtures e.g. DiNP (Di-isononylphthalate), but shall be verified in each case.

Table 2 — Phthalates which can be determined according to CEN/TS 16183

No	Name	Formula	Abbreviation	Molar mass g/mol	CAS-RN[a]
1	Dimethylphthalate	$C_{10}H_{10}O_4$	DMP	194,2	00131-11-3
2	Diethylphthalate	$C_{12}H_{14}O_4$	DEP	222,2	00084-66-2
3	Dipropylphthalate	$C_{14}H_{18}O_4$	DPP	250,3	00131-16-8
4	Di-(2-methyl-propyl)phthalate	$C_{16}H_{22}O_4$	DiBP	278,4	00084-69-5
5	Dibutylphthalate	$C_{16}H_{22}O_4$	DBP	278,4	00084-74-2
6	Butylbenzylphthalate	$C_{19}H_{20}O_4$	BBzP	312,4	00085-68-7
7	Dicyclohexylphthalate	$C_{20}H_{26}O_4$	DCHP	330,4	00084-61-7
8	Di-(2-ethylhexyl)phthalate	$C_{24}H_{38}O_4$	DEHP	390,6	00117-81-7
9	Dioctylphthalate	$C_{24}H_{38}O_4$	DOP	390,6	00117-84-0
10	Didecylphthalate	$C_{28}H_{46}O_4$	DDcP	446,7	00084-77-5
11	Diundecylphthalate	$C_{30}H_{50}O_4$	DUP	474,4	03648-20-2
a Chemical Abstracts Service Registry Number.					

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2 Normative references

The following referenced documents are indispensable for the application 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.

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

EN ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges from sewage and water treatment works (ISO 5667-13:1997)*

EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15:2009)*

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ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

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

3 Principle

The dried sample, dried by freeze-drying or with sodium sulfate is extracted with ethyl acetate on the shaking device. An aliquot of the extract is cleaned with aluminium oxide (if necessary) followed by gas chromatographic separation using capillary columns and identification and quantification of the phthalates by mass spectrometry.

4 Interferences

4.1 General

Due to their use as plasticizer agents, phthalates are ubiquitous. The sources of phthalates are multiple and shall be checked and reduced by every laboratory itself. Therefore, special attention shall be paid to avoid contaminations.

4.2 Interferences during sampling

In order to avoid interferences and cross contaminations, do not use plastic materials (pipes, etc.).

4.3 Cross contamination

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Chemicals and analytical equipment can be of various quality. Cross contamination is likely to occur with laboratory air. Therefore, remove, as far as possible, plastic materials from the laboratory. Cleaning agents often contain phthalates and may severely contaminate the laboratory air if in use regularly. Therefore, refrain from using these agents during application of this procedure.

Using plastic gloves during pretreatment may increase the contamination.

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4.4 Interferences in gas chromatography

Phthalates may bleed from the septa of the injector into the gas chromatograph, therefore use septa that are not likely to contaminate the system.

Fittings, e.g. of syringes, or equipment and septa of the sampling bottles (see 6.5) may also contain phthalates.

5 Reagents

5.1 General

All reagents shall be of recognized analytical grade.

Use only reagents with negligibly low concentration of phthalates and verify by blank determinations and, if necessary, apply additional cleaning steps.

5.2 Nitrogen, N_2, of high purity, at least a volume fraction of 99,9 % for drying and, if necessary, for concentration by evaporation.

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5.3 Helium, He, of high purity, at least a volume fraction of 99,999 %.

5.4 Ethyl acetate, $C_4H_8O_2$, phthalate-free, high purity.

5.5 Methanol, CH_3OH.

5.6 Isooctane, C_8H_{18} (2,2,4-trimethylpentane).

5.7 Quartz wool, heated to 400 °C for at least 4 h.

5.8 Aluminium oxide, Al_2O_3, neutral, 50 µm to 200 µm particle size, heated to 400 °C for at least 4 h.

Store in covered flask or desiccator. Use within five days after heat-treatment.

NOTE Alternative materials, like Florisil[1)] or silica may be used, provided their properties and capacity to separate are similar to aluminium oxide and their properties are checked according to 5.3.

5.9 Internal standards.

For example:

— deuterated di-n-butylphthalate, "D4-ring-DBP";

— deuterated D4-$C_{16}H_{22}O_4$;

— deuterated di-(2-ethylhexyl)phthalate "D4-ring-DEHP";

— deuterated D4-$C_{24}H_{38}O_4$; di-n-octylphthalate, "D4-ring-DOP";

— D4- $C_{24}H_{38}O_4$;

— ^{13}C-labelled standards can also be used, if available.

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5.10 Reference substances.

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Table 2 gives a list of phthalates, with defined mass concentrations, for the preparation of reference solutions for the gas chromatographic procedure.

5.11 Solutions of the single substances.

In a 10 ml volumetric flask (6.13), transfer e.g. 10 mg of each of the reference substances (5.10) in ethyl acetate (5.4) and bring to volume with ethyl acetate (5.4) (concentration: 1 g/l).

Store the solutions in glass bottles at – 18 °C, protected from light, and check the concentration at least every three months.

5.12 Stock solution.

In a 10 ml volumetric flask (6.13), dissolve between 100 µl and 500 µl of the single substance solutions (5.11) and bring to volume with ethyl acetate (5.4) (concentration: 10 mg/l to 50 mg/l).

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1) Florisil is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by CEN of this product.

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Store the solution in a glass bottle at – 18 °C, protected from light, and check the concentration at least every three months.

5.13 Reference solutions for multipoint calibration (see Annex A).

Prepare solutions by adequate dilution of the stock solution (5.12) and internal standards (5.9) with ethyl acetate (5.4).

Store the solutions in a glass bottle at – 18 °C, protected from light, and check the concentration at least every three weeks (see Annex A).

5.14 Solution of the internal standards (see Annex A)

5.14.1 Internal standard solution of D4-phthalates.

Weigh e.g. 0,1 g of an internal standard phthalate (D4) (5.9) in a 10 ml volumetric flask (6.13) filled with about 5 ml of ethyl acetate (5.4) and bring to volume with ethyl acetate (5.4). Store the solutions in glass bottles at – 18 °C.

5.14.2 Solution I internal standard mix.

Combine the solutions of the single internal standard phthalates (5.9) e.g. by dilution 1:100 as follows: Transfer with a syringe 0,1 ml (6.15) of each solution into a 10 ml volumetric flask (6.13) filled with about 5 ml of ethyl acetate (5.4). Bring to volume with ethyl acetate. The final concentration of di-n-octylphthalate (D4) di-n-butylphthalate (D4) and di-(2-ethylhexylphthalate) will be 100 mg/l in ethyl acetate (5.4).

5.14.3 Solution II internal standard mix

Take from this 1:100 dilution (5.14.2) e.g. 250 µl, transfer into a volumetric flask, 250 ml (6.13), filled with 250 ml of ethyl acetate (5.4).

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The final concentration of di-n-octylphthalate (D4), di-n-butylphthalate (D4) and di-(2-ethylhexylphthalate) is 0,1 mg/l in ethyl acetate (5.4).

5.14.4 Solution III internal standard mix.

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Dilute the solution I internal standard (5.14.2.) 1:10: Pipette 1 ml of the solution (5.14.2.) in a 10 ml volumetric flask (6.13) filled with about 5 ml of ethyl acetate (5.4). Bring to volume with ethyl acetate. The final concentration of di-n-octylphthalate (D4), di-n-butylphthalate (D4) and di-(2-ethylhexylphthalate) is 10 mg/l in ethyl acetate.

5.15 Sodium sulfate, Na_2SO_4, heated to 400 °C for at least 4 h.

6 Apparatus

6.1 General

Equipment or parts of it which are likely to come into contact with the sample or its extract shall be free from phthalates. This may be achieved by thorough cleaning of all glass apparatus and checked by the blank determination.

6.2 Wide-neck flat bottomed flasks with glass stoppers, preferably brown glass, volume 500 ml and 1 000 ml.

6.3 Drying oven, capable of being maintained at a temperature of (105 ± 5) °C.

6.4 Muffle furnace, adjustable, up to temperatures of (400 ± 10) °C, with capacity of e.g. at least 60 l.