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Sludge, treated biowaste and soil - Determination of pharmaceutical products

Boues, bio-déchets traités et sols - Détermination des produits pharmaceutiques

Schlamm, behandelter Bioabfall und Boden - Bestimmung pharmazeutischer Produkte

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (CEN/TS 16178:2012) 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

Drugs absorbed by the organism after intake are subject to metabolic reactions, such as hydroxylation or cleavage. However, a significant amount of the original or metabolised substance leaves the organism via urine or faeces. The contamination concerns ground and surface water, but also waste water and the solid matrices like sludge or soils.

Due to their polarity, persistence and water solubility, some drugs and metabolites are able to pass through the wastewater treatment plants. Their low adsorption on sludge and soils may cause the contamination of surface and ground water. It is therefore necessary to analyse these molecules through an analytical SPELC-MS/MS method as presented in this document. The method allows the identification of twelve molecules belonging to the four predominant therapeutic classes in France: analgesics/anti-inflammatories, lipid regulators, beta-blocker and anti-epileptics.

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 to analyse pharmaceutical compounds in sludge, treated biowaste and soil. Pharmaceuticals analysis has been carried out on a LC/MS-MS quantum. The main difficulty for analysis is the lack of sample certified for target analytes. Even with spiked solid matrices it is still delicate to correctly verify the impact of extraction step, because it is not commutable to a real sample.

This document provides a final protocol on the extraction and purification tested on sludge, soils and sediments spiked with the pharmaceutical compounds listed in Table 1.

Table 1 — Details to the pharmaceutical compounds of interest

Steroid hormones	CAS-RN[a]	Purity %	Temperature preservation	Formula	Molar mass g/mol	Abbreviation
Estrone	53-16-7	> 99	25°C	$C_{18}H_{22}O_2$	270,4	E1
17*β*-Estradiol	50-28-2	> 98	25°C	$C_{18}H_{24}O_2$	272,4	E2
17*α*-Estradiol	57-9	99	25°C	$C_{18}H_{24}O_2$	272,4	α-E2
Ethinylestradiol	57-63-6	> 98	25°C	$C_{20}H_{24}O_2$	296,4	EE2
Estrone d4 Estrone-2,4,16,16-d4	53866-34-5	> 95	25°C	$C_{18}H_{18}O_2D_4$	274,4	E1 d4
Estradiol d5 17b-Estradiol-2,4,16,16,17-d5	221093-45-4	> 98	25°C	$C_{18}H_{19}O_2D_5$	277,4	E2 d5
Ethinylestradiol d4 17*α*-Ethinylestradiol-2,4,16,16-d4	350820-06	> 98	25°C	$C_{20}H_{20}O_2D_4$	300,4	EE2 d4

[a] Chemical Abstracts Service Registry Number

2 Principle

After pretreatment the freeze-dried sample is extracted by ultrasonication with an appropriate solvent. Then the extract is purified on a suitable cartridge. The extract is analysed by high-performance liquid chromatography (HPLC) on a C_{18} column and detected by mass spectrometry.

The identification is based on the retention times of the analytes and on the mass-spectrometric detection. The detection is conducted in the MS/MS-mode in order to avoid interferences and over-quantification.