

DIN EN 16181:2019-08 (E)

Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC)

Contents

	Page
European foreword	4
Introduction	5
1 Scope	6
2 Normative references	7
3 Terms and definitions	7
4 Principle	8
5 Interferences	9
5.1 Interference with sampling and extraction	9
5.2 Interference with GC-MS	9
5.3 Interferences with the HPLC	9
6 Safety remarks	10
7 Reagents	10
8 Apparatus	15
9 Sample storage and preservation	17
9.1 Sample storage	17
9.2 Sample pretreatment	17
10 Procedure	18
10.1 Blank test	18
10.2 Extraction	18
10.2.1 General	18
10.2.2 Extraction procedure 1: acetone/hexane-like solvent and agitation	19
10.2.3 Extraction procedure 2: Soxhlet extraction (dry samples)	19
10.2.4 Extraction procedure 3: acetone/petroleum ether/sodium chloride and agitation	20
10.3 Concentration or dilution	20
10.3.1 General	20
10.3.2 For HPLC analysis	21
10.4 Clean-up of the extract	21
10.4.1 General	21
10.4.2 Clean-up A - Aluminium oxide	22
10.4.3 Clean-up B - Silica gel	22
10.4.4 Clean-up C - Gel permeation chromatography (styrene divinylbenzene resin)	22
10.4.5 Clean-up D - DMF/cyclohexane partitioning for aliphatic hydrocarbons removal	23
10.5 Addition of the injection standard	23
10.6 Gas chromatographic analysis (GC)	23
10.6.1 Gas chromatographic analysis with mass spectrometric detection	23
10.6.2 Calibration of the method using an internal standard	24
10.6.3 Measurement	25
10.6.4 Identification	25

10.6.5	Check on method performance	25
10.6.6	Calculation	26
10.7	High-performance liquid chromatographic analysis (HPLC)	27
10.7.1	General	27
10.7.2	Chromatographic separation	27
10.7.3	Detection	27
10.7.4	Calibration	28
10.7.5	Measurement of samples	29
10.7.6	Calculation	29
11	Performance characteristics	29
12	Precision	30
13	Test report	30
Annex A (informative) Repeatability and reproducibility data		31
A.1	Materials used in the interlaboratory comparison study	31
A.2	Interlaboratory comparison results	31
Annex B (informative) Examples of instrumental conditions and chromatograms		34
B.1	Measurement of PAH with GC-MS	34
B.2	Measurement of PAH with HPLC fluorescence	39
Bibliography		46