Accelerated Solvent Extraction

Environmental Applications Summary

Pesticides • Chlorinated Compounds • Persistent Organic Pollutants (POPs)

Thermo SCIENTIFIC

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Additional product information available at
www.thermoscientific.com/samplepreparation

Contents

The Accelerated Solvent Extraction System

Better extractions in less time using less solvent

Accelerated solvent extraction is an established technique used for fast, accurate sample preparation of air filters, soils, solids, and wastes. Coupled with rapid solvent reduction systems, the often labor-intensive steps of sample preparation can be automated in your laboratory. Accelerated solvent extraction eliminates many of the manual steps involved in preparing samples for analysis, which helps ensure increased reproducibility and accelerates the process significantly.

The Thermo Scientific™ Dionex™ ASE™ Accelerated Solvent Extractor systems use a combination of elevated temperature and pressure with common solvents to increase the efficiency of the extraction process. The result is faster extraction times and a significant reduction in solvent use. Many of the organic solvents used in extractions boil at relatively low temperatures. This is a limitation to techniques such as Soxhlet or automated Soxhlet as the highest temperatures at which extractions take place in these techniques will be the solvent's boiling point. If sufficient pressure is exerted on the solvent during the extraction, temperatures above the boiling point can be used. Therefore all of the advantages of working at elevated temperature can be realized even with solvents with relatively low boiling points. Operating at elevated pressures also accelerates the extraction process. Pumping solvent through a packed bed is easier at elevated pressures; pressurized solvent is forced into the pores of the sample matrix. Hence, the combination of elevated temperatures and pressures allows extractions to occur rapidly and completely.

When extractions are achieved at elevated temperatures, several factors contribute to improved speed, efficiency, and reduced solvent use: 1) solvent strength is higher, 2) diffusion rates are faster, 3) solvent viscosity is decreased, and 4) solute-matrix interactions (dipole attractions, Van der Waals forces, hydrogen bonding, etc.) are more easily disrupted allowing the analytes to be removed from the matrix.

For environmental applications, accelerated solvent extraction is proven to produce equivalent to or better than traditional methods. Accelerated solvent extraction is accepted for use in EPA Method SW-846 3545 for the extraction of pesticides and herbicides, PAHs and semivolatile compounds, PCBs, dioxins and furans, and explosive compounds. Accelerated solvent extraction is also accepted for use in EPA Method SW-846 6860 for the determination of perchlorate and in CLP OLM 04.2A for semivolatiles and pesticides. The Application Briefs compiled here show conditions for extracting pesticides, chlorinated compounds, and POPs from a variety of environmental samples. To view the complete Application Notes, visit our website at www. thermoscientific.com/samplepreparation.

Thermo Scientific Dionex ASE 150 and ASE 350 Accelerated Solvent Extractor systems.

Extraction of Polychlorinated Dibenzo-*p*-dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) from Environmental Samples Using Accelerated Solvent Extraction

Introduction

Regulatory agencies are concerned with the high toxicity of PCDDs and PCDFs; great efforts are expended to monitor their presence in environmental samples. The low concentrations at which these compounds are regulated and the strength of their binding to certain matrices pose special challenges to analytical chemists. Accelerated solvent extraction has been applied to the extraction of PCDD and PCDFs from ground chimney brick, urban dust, fly ash and sediment samples. Accelerated solvent extraction complies with U.S. EPA Method 3545 for these compounds.

U.S. EPA Method 3545A: Dioxins and furans from ground chimney brick, urban dust, and fly ash

Equipment

Dionex ASE 200 Accelerated Solvent Extractor*

Gas Chromatograph (GC) with Mass Spectrometer (MS) (low resolution or high resolution MS-MS)

Low pressure LC system for sample clean-up

Preparative HPLC for sample clean-up

Solvents

Toluene, acetic acid, hydrochloric acid. All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

**Dionex ASE 150 and 350 can be used for equivalent results.*

Extraction Conditions

Solvent:	Toluene or toluene/acetic acid (5%, v/v) if HCl pretreatment currently used
Temperature:	175–200 °C
Pressure:	1500 psi**
Static Time:	5–15 min
Static Cycles:	2 or 3
Flush Volume:	60–70%
Purge Time:	60–100 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-MS

Results

Left table: Average values (ng/kg) from ground chimney brick—comparison of Soxhlet vs. ASE.
Right table: Average values (ng/kg) from urban dust—comparison of Soxhlet vs. ASE.

Group Totals	Soxhlet (n=1)	ASE[a] (n=2)
Total T_4CDD	182	325
Total P_5CDD	175	221
Total H_6CDD	86.7	81.7
Total H_7CDD	221	217
Total O_8CDD	445	314
Total T_4CDF	333	419
Total P_5CDF	146	179
Total H_6CDF	65.9	122
Total H_7CDF	13.2	29.4
Total O_8CDF	n.d. (10)	n.d. (10)
Congeners	**Soxhlet (n=1)**	**ASE[a] (n=2)**
2,3,7,8-T_4CDD	3.3	3.2
1,2,3,7,8-P_5CDD	11.8	13.1
1,2,3,4,7,8-H_6CDD	9.8	8.0
1,2,3,6,7,8-H_6CDD	11.5	9.5
1,2,3,7,8,9-H_6CDD	n.d. (8)	n.d. (8)
1,2,3,4,6,7,8-H_7CDD	113	107
2,3,7,8-T_4CDF	12.5	18.6
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	9.9	12.0
2,3,4,7,8-P_5CDF	13.9	18.1
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	18.7	23.7
1,2,3,6,7,8-H_6CDF	10.7	15.8
2,3,4,6,7,8-H_6CDF	3.3	8.7
1,2,3,7,8,9-H_6CDF	n.d. (2)	n.d. (2)
1,2,3,4,6,7,8-H_7CDF	13.2	29.4
1,2,3,4,7,8,9-H_7CDF	n.d. (3)	n.d. (3)
Toxicity equivalent (NATO)	25.0	28.7
Toxicity equivalent (BgVV)	24.2	29.2

Values are corrected for recovery of ^{13}C-labeled surrogates.
n.d.=not detected. Detection limit, in ppt, given in parentheses.
[a]Sum of two extractions of each sample.

Group Totals	Soxhlet (n=1)	ASE[a] (n=2)
Total T_4CDD	440	530
Total P_5CDD	900	940
Total H_6CDD	1800	2000
Total H_7CDD	2000	2100
Total O_8CDD	2900	2600
Total T_4CDF	2300	2600
Total P_5CDF	4100	4300
Total H_6CDF	4700	4700
Total H_7CDF	2800	2600
Total O_8CDF	2000	2000
Congeners	**Soxhlet (n=1)**	**ASE[a] (n=2)**
2,3,7,8-T_4CDD	6.0	6.0
1,2,3,7,8-P_5CDD	52	57
1,2,3,4,7,8-H_6CDD	46	52
1,2,3,6,7,8-H_6CDD	120	130
1,2,3,7,8,9-H_6CDD	97	1000
1,2,3,4,6,7,8-H_7CDD	1000	820
2,3,7,8-T_4CDF	160	180
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	430	470
2,3,4,7,8-P_5CDF	390	390
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	1100	1100
1,2,3,6,7,8-H_6CDF	540	570
2,3,4,6,7,8-H_6CDF	400	360
1,2,3,7,8,9-H_6CDF	42	42
1,2,3,4,6,7,8-H_7CDF	2100	2000
1,2,3,4,7,8,9-H_7CDF	140	120
Toxicity equivalent (NATO)	540	540
Toxicity equivalent (BgVV)	490	510

Values are corrected for recovery of ^{13}C-labeled surrogates.
[a]Sum of two extractions of each sample.

Extraction of Chlorinated Herbicides Using Accelerated Solvent Extraction

Introduction

The use of accelerated solvent extraction in the extraction of chlorinated herbicides from solid wastes is more convenient, faster, and less solvent-intensive than previous methods. The procedures described in this Application Note meets the requirements for sample extraction as prescribed by U.S. EPA Method 3545. This method is applicable to the extraction of water-insoluble or slightly water-soluble chlorinated herbicides in preparation for gas chromatographic measurements.

U.S. EPA Method 8150A with 3545A: Chlorinated herbicides from soil, sludge, and sediments

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells

Gas Chromatograph (GC) with Electron Capture Detector (ECD)

Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE)
Dispersant for ASE™, 1 kg Bottle (P/N 062819)

Dichloromethane

Acetone

Phosphoric acid

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent:	Dichloromethane/acetone (1:2, v/v), with 4% (v/v) H_3PO_4/H_2O (1:1)
Temperature:	100 °C
Pressure:	1500 psi**
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60% of extraction cell volume
Purge Time:	60 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-ECD

Results

See tables below.

Average recovery of chlorinated herbicides from three soil types*—ASE compared to wrist-shaking.

Chlorinated Herbicide Target Compound	Average Recovery (% of Shaking Method)
2,4-D	116.2
2,4-DB	112.9
2,4,5-T	106.6
2,4,5-TP	117.4
Dalapon	101.8
Dicamba	108.1
Dichlorprop	107.7
Dinoseb	118.4

[a] Averages from extraction of sand, loam, and clay soils.

Average recovery and precision for extraction of chlorinated herbicides from three soil types by ASE method.

Matrix[a]	ASE (% of Spike)	ASE[b] (RSD,%)	Shaking (% of Spike)	Shaking[b] (RSD,%)	ASE as % of Shaking
Clay (low)	36.1	54.5	42.2	25.2	89.8
Clay (high)	71.1	15.0	61.6	21.6	112.8
Loam (low)	56.7	11.2	36.9	78.7	126.6
Loam (high)	59.9	14.1	43.9	14.7	132.8
Sand (low)	51.1	12.6	49.7	13.3	111.2
Sand (high)	69.2	39.5	66.3	35.5	104.4

[a] Low spiking levels ranged from 50 to 500 µg/kg. High spiking levels ranged from 500 to 5000 µg/kg.

[b] Each precision (RSD,%) value is the average of seven replicate measurements for each compound, then averaged for all compounds.

Extraction of Chlorinated Pesticides Using Accelerated Solvent Extraction

Introduction

Accelerated solvent extraction provides a more convenient, faster, and less solvent intensive method than previously available for the extraction of chlorinated pesticides from environmentally important samples. Recoveries of these analytes by accelerated solvent extraction are equivalent to or better than other more solvent intense methods such as Soxhlet. Accelerated solvent extraction also avoids the problem of multiple washing procedures associated with sonication.

The procedure described in this Application Note meets the requirement for sample extraction as prescribed by EPA Method 3545. This method is applicable to the extraction of water-insoluble or slightly water-soluble volatiles and semivolatiles in preparation for gas chromatographic or GC/MS measurement. The method is applicable to the extraction of chlorinated pesticides from soils, clays, wastes, and sediments containing from 5 to 250 µg/kg of the target compounds.

U.S. EPA Method 3545A: Organochlorinated Pesticides (OCPs) from soil, clay, sludge, and sediments

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells

Gas Chromatograph (GC) with Mass Spectrometer (MS) or GC

Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE)

Dispersant for ASE, 1 kg Bottlet (P/N 062819)

Hexane

Acetone

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent:	Acetone/hexane (1:1 v/v)
Temperature:	100 °C
Pressure:	1500 psi**
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60%
Purge Time:	60 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-MS

Results

Average recovery of pesticides from three soil types*—ASE compared to automated Soxhlet.

Pesticide	Average Recovery (% of Soxhlet)
Alpha BHC	93.3
Gamma BHC-Lindane	95.6
Beta BHC	98.6
Heptachlor	88.0
Delta BHC	99.5
Aldrin	94.9
Heptachlor Epoxide	100.7
Gamma Chlordane	99.5
Alpha Chlordane	102.0
Endosulfan 1	100.3
p,p'-DDE	98.6
Dieldrin	101.2
Endrin	97.2
p,p-DDD	104.6
Endosulfan II	105.6
p,p'-DDT	74.9
Endrin Aldehyde	104.0
Endosulfan Sulfate	105.2
Methocychlor	79.6
Endrin Ketone	102.9

[a] Averages from extraction of sand, loam, and clay soils.

Extraction of Organophosphorus Pesticides (OPPs) Using Accelerated Solvent Extraction

Introduction

The use of accelerated solvent extraction in the extraction of OPP compounds from solid wastes is a more convenient, faster, and less solvent-intensive method than previously available. OPP recoveries by accelerated solvent extraction are equivalent to recoveries from Soxhlet and other methods. Accelerated solvent extraction also avoids the problem of multiple washing procedures associated with sonication.

The procedures described in this Application Note meet the requirements for sample extraction as prescribed by U.S. EPA Method 3545A. This method is applicable to the extraction of water-insoluble or slightly water-soluble OPPs in preparation for gas chromatographic measurements. This method is applicable to soils, solid wastes, and sediments containing 250–2500 µg/kg of OPPs.

U.S. EPA Method 3545A: OPPs from soil, sludge, and sediments

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells

Gas Chromatograph (GC) with Nitrogen Phosphorous Detector (NPD)

Vials for collection of extracts (40 mL, P/N 049465, 60 mL, P/N 049466)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE)
Dispersant for ASE, 1 kg Bottle (P/N 062819)

Dichloromethane

Acetone

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent:	Dichloromethane/acetone (1:1 v/v)
Temperature:	100 °C
Pressure:	1500 psi**
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60% of extraction cell volume
Purge Time:	60 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-NPD

Results

Average recovery and precision for OPP extractions from three soil types.

Matrix[a]	ASE (% of Spike)	ASE[b] (RSD,%)	Shaking (% of Spike)	Shaking[b] (RSD,%)	ASE as % of Shaking
Clay (low)	55.0	6.2	56.4	7.6	98.8
Clay (high)	69.2	5.2	72.3	16.3	96.3
Loam (low)	61.3	11.6	60.4	8.6	103.6
Loam (high)	61.4	7.8	64.2	6.3	96.5
Sand (low)	59.0	13.0	63.3	6.7	95.0
Sand (high)	64.1	11.9	63.2	4.8	101.2

[a] Low spiking level is approximately 250 µg/kg. High spiking level is approximately 2500 µg/kg.

[b] Each recovery and precision (%RSD) value is the average of seven replicate measurements for each compound, then averaged for all compounds.

Rapid Determination of Persistent Organic Pollutants (POPs) Using Accelerated Solvent Extraction

Introduction

The United Nations Environmental Program (UNEP) has been implemented in an effort to combat the release of selected POPs. POPs are found in environmental samples such as soils, sludges, solid and semi-solid waste, and sediments. POPs are also found in biological samples such as human breast milk, and fish tissue. UNEP is interested in eliminating POPs from the environment because these compounds are considered toxic, carcinogenic, and mutagenic, and degrade slowly in the environment, posing a threat to the global environment.

Accelerated solvent extraction is equivalent to U.S. EPA Methods 3540, 3541, 3550, and 8150 for the extraction of organochlorine pesticides (OCPs), organophosphorous pesticides (OPPs), semivolatiles or base neutral acids (BNAs), chlorinated herbicides, polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyls (PCBs). Accelerated solvent extraction complies with U.S. EPA Method 3545A for these compounds.

U.S. EPA Methods 3540, 3541, 3550, and 8150: POPs from various environmental samples

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* with Solvent Controller (P/N 048765)

Use either:

- 22 mL stainless steel extraction cells (P/N 048764)
- 11 mL stainless steel extraction cells (P/N 048765)
- 33 mL stainless steel extraction cells (P/N 048766)

Cellulose filters (P/N 0469458)

Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

Analytical balance (to read to nearest 0.0001 g or better)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle) (P/N 062819)

Hexane

Dichloromethane

Acetone

Toluene

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Pesticides and PCBs (8081/8082)

Solvent: Hexane/acetone (1:1), (v/v)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1–2
Flush Volume: 60%
Purge Time: 60–120s

Hexachlorobenzene (8270)

Solvent: Dichloromethane/acetone (1:1), (v/v)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1–2
Flush Volume: 60%
Purge Time: 60–120 s

Dioxins (PCDD and PCDF) (8290)

Solvent: Toluene (100%) or toluene/acetic acid (5%, v/v) if HCl pretreatment currently used
Temperature: 75–200 °C
Pressure: 1500 psi**
Static Time: 5–15 min
Static Cycles: 2–3
Flush Volume: 60–70%
Purge Time: 60–120 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-MS

GC-MS/MS

Results

Average recovery of pesticides from three soil types[a]—ASE compared to automated Soxhlet.

Pesticide	Average Recovery (% of Soxhlet)
Heptachlor	88.0
Aldrin	94.9
Gamma Chlordane	99.5
Alpha Chlordane	102.0
Dieldrin	101.2
Endrin	97.2
p,p'-DDT	74.9

[a] Averages from extraction of sand, loan, and clay soils.

Analyzing PBDEs in House Dust Samples with the Thermo Scientific TSQ Quantum XLS Ultra GC-MS/MS in EI-SRM Mode

Introduction

Polybrominated diphenyl ethers (PBDEs) were introduced in the 1960s as flame retardants and are used today in a wide variety of household apparatuses, consumer electronics, furniture and more. Environmental levels of PBDEs have been continuously increasing due to their special persistence. Certain congeners have been banned completely and are currently in the list of the Stockholm convention's POPs. Sources of human intake are typically via ingestion (nutritional) and the inhalation of indoor and house dust.

Due to growing concerns over the health risks from constant exposure to this class of compounds and the accumulation effects in the food chain, suitable analytical methods are required to quantify flame retardant compounds at low levels in a variety of matrices.

U.S. EPA Method 1614: PBDEs from House Dust

Equipment

Dionex ASE 350 Accelerated Solvent Extractor equipped with 22 mL or larger stainless steel extraction cells

Gas Chromatograph (GC) with Mass Spectrometer (MS)

Solvents

Methylene chloride

Silica gel

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

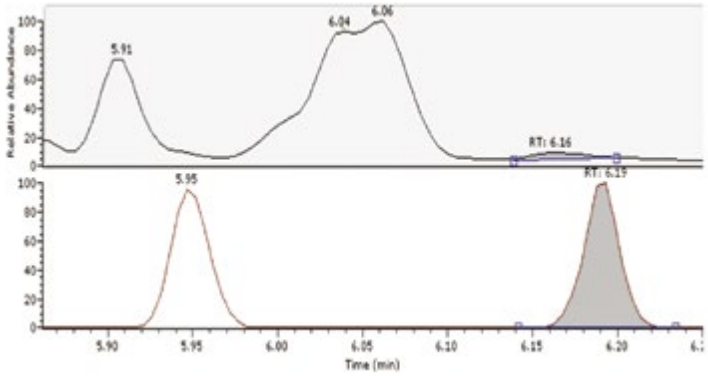
Solvent:	Methylene chloride (100%)
Temperature:	100 °C
Pressure:	1500 psi*
Static Time:	5 min
Static Cycles:	3
Flush Volume:	60%
Purge Time:	120 s

**Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

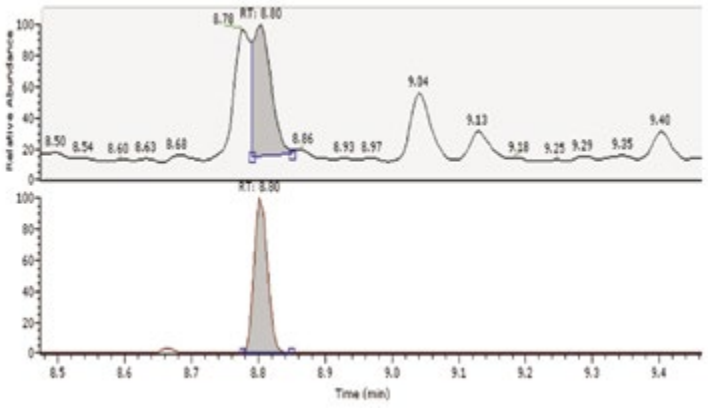
Analysis

GC-MS

Results



House dust sample in NCI (top) and EI-SRM (bottom) showing BDE47 at a level of 0.07 ppb.



House dust sample in NCI (top) and EI-SRM (bottom) showing BDE 183 at a level of 0.17 ppb.

Extraction of Polybrominated Biphenyls (PBDEs) from Sediments Using Accelerated Solvent Extraction

Introduction

Polybrominated diphenyl ethers (PBDEs) were developed in the early 1970s and are used today as flame retardants for various consumer products, including clothing, furniture, and plastics. Many of these consumer products are disposed in municipal landfills where the PBDEs then leach into groundwater and accumulate in certain biosystems. Recent studies indicate that PBDE concentrations in these biosystems are on the rise. In the 1980s, PBDEs were discovered in European waterways, which led the European Union to ban their production and use. Although the toxicity of PBDEs is still under investigation, evidence suggests that PBDEs may compromise endocrine or hepatic functions.

In this Application Note, PBDEs were extracted from human breast milk (freeze-dried), sediments, fish tissues, and polymers.

U.S. EPA Method 1614: PBDEs from sediments

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* with Solvent Controller (P/N 048765)*

22 mL stainless steel extraction cells (P/N 048764)

Gas Chromatograph (GC) with Mass Spectrometer (MS)

Cellulose filters (P/N 0469458)

Vials for collection of extracts, 60 mL (P/N 049466)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE)

Dispersant for ASE, 1 kg Bottle (P/N 062819)

Methylene chloride

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Sediments

Solvent:	Methylene chloride (100%)
Temperature:	100 °C
Pressure:	1500 psi**
Static Time:	5 min
Static Cycles:	2
Flush Volume:	60%
Purge Time:	120 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-MS

Results

With a detection limit of 0.5 µg/kg, the GC/MS analysis found BDE-47 in 22% of the 332 sediment sample extracts. BDE-47 is one of two major constituents of Penta, which is a commercial product used to flame retard polyurethane foam.

Accelerated Solvent Extraction of Polychlorinated Biphenyls from Polyurethane Foam Absorbent Cartridges

Introduction

U.S. EPA Method TO-41 has been used for a number of years for the determination of polychlorinated biphenyls (PCBs) in air. The traditional extraction method for PCBs has been Soxhlet, as typified by U.S. EPA Method 3540. This method requires 12–14 hours of extraction time and 350 mL of solvent per sample to perform. The size and volume requirements of the PUF cartridges have made alternative extraction methods difficult to employ. Accelerated solvent extraction has been found to be a successful alternative method with several advantages over traditional methods.

U.S. EPA Method TO-41: PCBs from air sampling PUF cartridges

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* equipped with either:

22 mL stainless steel extraction cells (P/N 048764)

11 mL stainless steel extraction cells (P/N 048765)

Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

Gas Chromatograph (GC)

Kimwipes® (Kimberly-Clark Corporation)

2.5 inch diam. × 3.0 inch PUF cartridge

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents

Hexane

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent:	Hexane
Temperature:	100 °C
Pressure:	1500 psi**
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60%
Purge Time:	100 s

**Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC

Results

See tables below.

Initial demonstration of laboratory performance for ASE and Soxhlet extraction of PUF cartridges*.

Sample #	ASE (µg)	Soxhlet (µg)	US EPA Method 608 Acceptance Criteria
LCS 1	4.63	4.87	
LCS 2	4.44	4.93	
LCS 3	4.72	5.16	
LCS 4	4.10	4.26	
Average (µg)	4.47	4.80	3.42 - 8.40 µg
Average Recovery	90%	96%	38 - 158%
Standard Deviation	0.274	0.384	< 1.23

*Recoveries are based on a spiked value of 5.00 µg Aroclor 1248.

Method detection limit study for ASE and Soxhlet.

Sample #	ASE (µg)	Soxhlet (µg)
MDL 1	0.92	0.79
MDL 2	0.97	0.90
MDL 3	0.91	0.95
MDL 4	0.95	0.78
MDL 5	0.80	0.79
MDL 6	1.11	0.88
MDL 7	0.95	0.90
MDL 8	1.21	0.82
Average	0.98	0.85
Standard Deviation	0.13	0.06
MDL (3δ)	0.38	0.19

*Recoveries are based on a spiked value of 1.00 µg Aroclor 1248.

Extraction of PCBs from Environmental Samples Using Accelerated Solvent Extraction

Introduction

Polychlorinated biphenyls (PCBs) are found in many solid waste materials worldwide. This Application Note describes the application of accelerated solvent extraction to the extraction of PCBs from sewage sludge, river sediments, marine sediments, and marine tissue (oyster). The procedures described in this Application Note meet the requirements for sample extraction as determined by U.S. EPA Method 3545 for solid samples.

Accelerated solvent extraction is a direct replacement for solvent-intensive techniques such as Soxhlet and sonication. For the preparation of solid waste samples, containing PCBs, accelerated solvent extraction provides more convenient, faster extractions with significantly less solvent usage than these other methods.

U.S. EPA Method 3545A: PCBs from soil, sludge, and sediments

Equipment

Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells

Gas Chromatograph (GC) with Electron Capture Detector (ECD)

Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

**Dionex ASE 150 and 350 can be used for equivalent results.*

Solvents and Reagents

Diatomaceous Earth (DE)

Dispersant for ASE, 1 kg Bottle (P/N 062819)

Acetone

Hexane

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent:	Hexane/acetone (1:1), (v/v)
Temperature:	100 °C
Pressure:	1500** psi
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60% of extraction cell volume
Purge Time:	60 s

***Pressure studies show that 1500 psi is the optimum extraction pressure for all accelerated solvent extraction applications.*

Analysis

GC-ECD

Results

PCB recoveries from sewage sludge*.

PCB Congener	Average Recovery, n = 6 (as % of Soxhlet)	RSD (%)
PCB 28	118.1	2.5
PCB 52	114.0	4.7
PCB 101	142.9	7.4
PCB 153	109.5	5.8
PCB 138	109.6	3.9
PCB 180	160.4	7.5

*Analyte concentration range: 160–200 µg/kg per component

PCB recovery from river sediment (SRM 1939)*.

PCB Congener	Average, n = 6 (as % of Soxhlet)	RSD (%)
PCB 101	89.2	3.7
PCB 153	62.3	4.1
PCB 138	122.1	2.3
PCB 180	111.5	5.9

*Analyte concentration range: 170–800 µg/kg per component

Recovery of Aroclor 1254 from soil (CRM911-050).

Run Number	Aroclor Found (µg/kg)
1	1290.0
2	1365.8
3	1283.4
4	1368.6
Average	1327.0 (99.0%)
RSD	3.51%

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