



Accelerated Solvent Extraction Environmental Applications Summary

Hydrocarbons • Semivolatiles • Organometallics • Munitions Contaminants • Perchlorate

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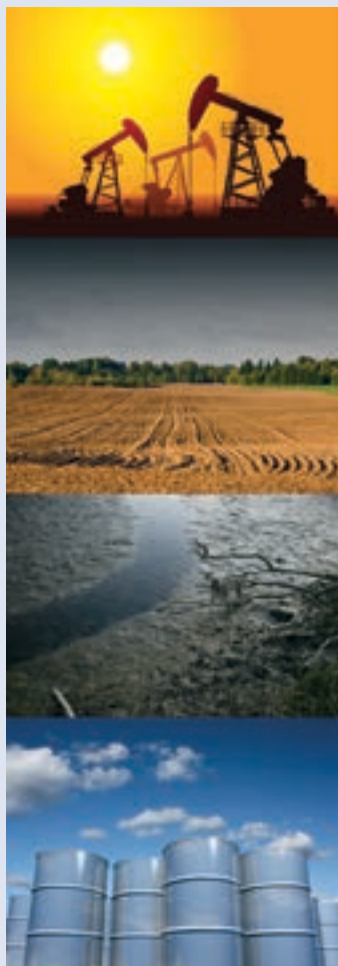
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The Accelerated Solvent Extraction System



Complete extractions in less time, using less solvent

Accelerated solvent extraction is an established technique used for fast and accurate sample preparation of air filters, soils, solids, and semisolids. Accelerated solvent extraction eliminates many of the manual steps involved in preparing samples for analysis, which helps ensure high analyte recovery and increased reproducibility.

The Thermo Scientific™ Dionex™ ASE™ 150 and 350 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. Precise temperature control of the extraction cell contents provides excellent reproducibility, and the high level of automation increases laboratory productivity. 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, since the

highest temperatures at which extractions take place in these techniques will be the boiling point of the solvent. If sufficient pressure is exerted on the solvent during the extractions, temperatures above the boiling point can be used.

Operating at elevated pressures accelerates the extraction process. Pumping solvent through a full extraction cell 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 such as analyte diffusion rates are increased and analyte solubility is increased, solvent viscosity is decreased. The net result means performing

extractions at elevated temperatures uses less time with less solvent.

For environmental applications, accelerated solvent extraction is proven to produce equivalent or better than traditional methods. Accelerated solvent extraction is approved for use in U.S. EPA Method 3545A for the extraction of polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), base/neutral/acids (BNAs), organophosphorus pesticides (OPPs), herbicides, dioxins, and is in compliance with U.S. EPA SW-846. The Application Briefs compiled here show conditions for extracting hydrocarbons, semivolatiles, munitions contaminants, polyphenols, and others from a variety of environmental samples. For additional information about the Dionex ASE systems or to view the complete Application Notes, visit our website at www.thermoscientific.com/samplepreparation.



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

Extraction of PAHs from Environmental Samples by Accelerated Solvent Extraction



Introduction

Previously, the extraction of polycyclic aromatic hydrocarbon (PAH) compounds from environmental materials including soils, sludge, and other solid wastes typically required large amounts of solvents. Soxhlet, for example, can use 250 to 500 mL of solvent for most environmental samples. Accelerated solvent extraction was developed to meet the new requirements for reducing solvent usage in the preparation of solid waste samples. The procedures described in this Application Note meet the requirements for the extraction of PAHs from solid waste as described in U.S. EPA Method 3545A. This method is applicable to solid wastes including soils, sludge's, and sediments.

U.S. EPA SW-846 Method 3545A: PAH Extraction from Soil, Sludge, and Marine Sediment

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or larger stainless steel extraction cells
- LC-PAH Column, 15 cm × 4.6 mm
- Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049465)

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

Solvents and Reagents

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

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%

Purge Time: 60 s

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

Analysis

- HPLC
- GC-MS

Results

Recovery of PAHs from contaminated soil (SRS 103-100)^a.

Compound	Average Recovery, n = 8 (as % of Soxhlet)	RSD (%)
Fluorene	83.4	1.6
Phenanthrene	119.2	1.9
Anthracene	88.0	6.6
Fluoranthene	101.2	14
Pyrene	104.8	18
Benz[a]anthracene	93.6	10
Chrysene	121.8	15
Benzo[b,k]fluoranthene ^b	142.3	8.1
Benzo[a]pyrene	100.3	15

^a Analyte concentration range: 20–1400 mg/kg per component

^b Sum of benzo[*b*]fluoranthene and benzo[*k*]fluoranthene

PAHs from marine sediment HS-3 (mg/kg).

Compound	Average Recovery (n = 4)	Standard Deviation	Certified Value (n = 4)	90% CI ^a
Naphthalene	8.87	1.00	9.0	0.7
Acenaphthalene	ND ^b	NA ^c	0.3	0.1
Acenaphthene	4.89	0.51	4.5	1.5
Fluorene	10.09	1.26	13.6	3.1
Phenanthrene	68.80	6.44	85.0	20
Anthracene	7.73	0.57	13.4	0.5
Fluoranthene	54.73	4.82	60.0	9
Pyrene	33.70	2.83	39.0	9
Benz[a]anthracene	12.40	1.07	14.6	2
Chrysene	14.95	1.52	14.1	2
Benzo[a]pyrene	6.27	0.65	7.4	3.6
Benzo[b]fluoranthene	11.46	1.27	7.7	1.2
Benzo[k]fluoranthene	10.16	1.28	2.8	2
Benzo[ghi]perylene	4.14	0.69	5.0	2
Dibenz[ah]anthracene	2.58	0.33	1.3	0.5
Indeno[1,2,3- <i>cd</i>]pyrene	4.30	0.77	5.4	1.3

^a CI = Confidence interval of the mean

^b ND = Not detectable at the level of detection (1.5 mg/kg)

^c NA = Not applicable

Simultaneous Extraction of PAHs and PCBs from Environmental Samples Using Accelerated Solvent Extraction



Introduction

Polycyclic aromatic hydrocarbons (PAHs) and Polychlorinated Biphenyls (PCBs) are considered toxic and carcinogenic. They are classified as persistent organic pollutants (POPs). Typically, PAHs and PCBs are extracted separately, using different solvent combinations. The purpose of this Application Note is to report on the development of a single extraction method for PAHs and PCBs from mussel tissue and soil using accelerated solvent extraction. Accelerated solvent extraction has been approved for use in U.S. EPA Method 3545A.

Equipment

- Dionex ASE 350 Accelerated Solvent Extractor*, equipped with 66 mL stainless steel extraction cells
- 250 mL clear collection bottles (P/N 056284)
- Filters, Glass Fiber, 34, 66, or 100 mL cell (P/N 056781)
- Analytical balance
- GC with Electron Capture Detector (ECD)
- GC with MS
- Nitrogen evaporator

*ASE 150 and 200 can be used for equivalent results.

U.S. EPA SW-846 Method 3545A: PAH and PCB Extraction from Soil and Mussel Tissue

Solvents and Reagents

- Alumina, Acidic, 60-325 Mesh, Fisher Chemical (P/N A948-500)
- Decachlorobiphenyl
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Dichloromethane
- Fisherbrand™ Robotic Screw Top Autosampler; Amber with Write-on Patch (P/N 03-391-9)
- Hexane

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

Extraction Conditions

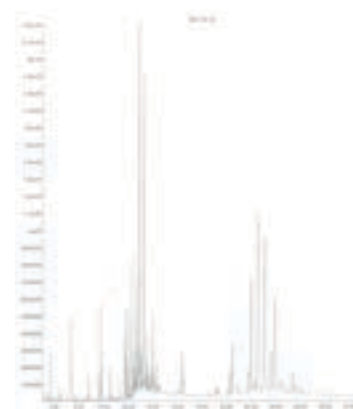
Method 1:	Extraction of PAHs and PCBs from spiked mussel tissue at two different temperatures
Solvent:	Dichloromethane
Temperature:	125 °C
Pressure:	1500 psi*
Static Time:	6 min
Static Cycles:	4
Flush Volume:	60%
Purge Time:	300 s

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

Analysis

- GC-ECD
- GC/MS

Results



GC/MS analysis of mussel sample extracted by Method 1.

Data for mussel and soil samples extracted by Method 1.

PAH Recoveries – Mussel (N = 6)				PAH Recoveries – Soil (N = 6)			
Compound	% Recovery	SD	% RSD	Compound	% Recovery	SD	% RSD
Nitrobenzene-d5**	83.3	0.54	13.05	Nitrobenzene-d5**	94.6	0.81	17.20
2-Fluorobiphenyl**	95.1	0.43	9.13	2-Fluorobiphenyl**	101.2	0.25	4.87
p-Terphenyl-d4**	91.4	0.27	5.92	p-Terphenyl-d4**	102.1	0.10	1.94
Naphthalene	89.1	0.28	6.33	Naphthalene	79.0	0.47	6.29
Acenaphthylene	101.2	0.30	5.91	Acenaphthylene	76.3	0.21	5.44
Acenaphthene	98.3	0.28	5.65	Acenaphthene	102.9	0.33	6.40
Fluorene	107.5	0.46	8.65	Fluorene	80.3	0.21	5.31
Phenanthrene	104.6	0.30	5.70	Phenanthrene	114.8	0.37	6.39
Anthracene	100.1	0.29	5.77	Anthracene	91.4	0.51	11.19
Fluoranthene	97.1	0.30	6.24	Fluoranthene	103.6	0.12	2.23
Pyrene	88.9	0.24	5.31	Pyrene	97.4	0.14	2.90
Benzo(a)anthracene	85.4	0.21	4.85	Benzo(a)anthracene	99.0	0.17	3.35
Chrysene	95.5	0.27	5.66	Chrysene	91.2	0.09	1.90
Benzo(b)fluoranthene	91.7	0.31	6.72	Benzo(b)fluoranthene	96.3	0.14	2.82
Benzo(k)fluoranthene	88.3	0.20	4.43	Benzo(k)fluoranthene	92.8	0.13	2.70
Benzo(a)pyrene	89.9	0.28	6.29	Benzo(a)pyrene	83.0	0.23	5.52
Benzo(ghi)perylene	94.1	0.31	6.60	Benzo(ghi)perylene	82.4	0.13	3.22
Dibenzo(a,h)anthracene	92.3	0.28	6.06	Dibenzo(a,h)anthracene	78.9	0.15	3.68
Indeno(1,2,3-cd)pyrene	91.1	0.31	6.72	Indeno(1,2,3-cd)pyrene	84.6	0.11	2.65
PCB Recoveries – Mussel (N = 6)				PCB Recoveries – Soil (N = 6)			
Compound	% Recovery	SD	% RSD	Compound	% Recovery	SD	% RSD
2,4,5,6-tetrachloro-m-xylene**	93.1	0.48	5.21	2,4,5,6-tetrachloro-m-xylene**	86.7	1.2	4.72
Aroclor 1254	95.9	0.06	3.26	Aroclor 1254	101.6	0.19	3.15

**Surrogate Spike

Extraction of Total Petroleum Hydrocarbon Contaminants (Diesel and Waste Oils) in Soils by Accelerated Solvent Extraction



Introduction

Underground storage tanks containing hydrocarbon based fuels are found worldwide. Many of these tanks have leaks, allowing gasoline, diesel, oil, or other hydrocarbon materials to contaminate the surrounding soil. In 1992, only two million of the five million tanks in the U.S. were monitored for leaks. However, underground storage tanks represent only one of many sources of hydrocarbon contamination in soils. Accelerated solvent extraction has been demonstrated to be equivalent to existing extraction methodologies, such as Soxhlet and automated Soxhlet, for most RCRA (Resource Conservation and Recovery Act) analytes from solid and semisolid samples. This Application Note reports on the use of accelerated solvent extraction for the extraction of diesel range organics (DRO), waste oil products (WOO), and total petroleum hydrocarbons (TPH, the sum of DRO and WOO) from soils.

U.S. EPA Method 3545A: Total Petroleum Hydrocarbons from Soil, Sludge, and Sediments

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or larger stainless steel extraction cells
 - GC with Flame Ionization Detector (FID)
 - Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049465)
 - Analytical balance
- *ASE 150 and 350 can be used for equivalent results.

Solvents and Reagents

- Acetone
 - Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
 - Methylene chloride
 - Ottawa sand
 - Silica gel
 - Sodium sulfate (anhydrous) used after extraction
 - n-Tricosane (C23) as surrogate
- All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions

Solvent: Methylene chloride/acetone (1:1), (v/v)

Temperature: 175 °C

Pressure: 1500 psi*

Static Time: 5 min

Static Cycles: 1

Flush Volume: 75%

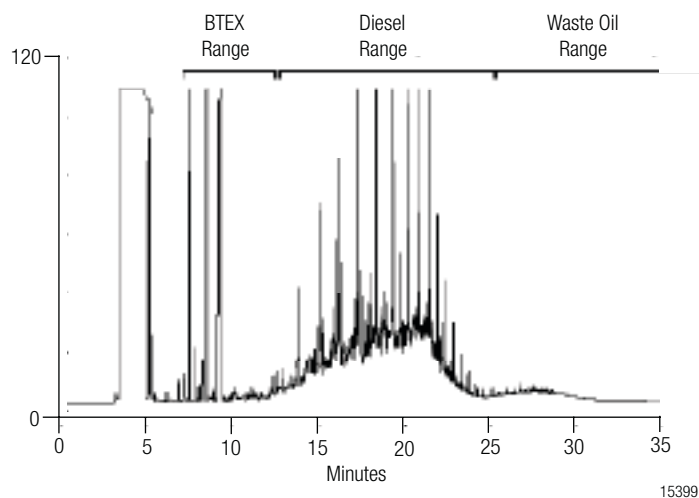
Purge Time: 60 s

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

Analysis

- GC-FID

Results



Typical gas chromatogram from a soil extract.

Results for all hydrocarbon groups (average of TPH, DRO, WOO).

Matrix and Concentration	Bias ¹	Precision ²	MDL (mg/kg)	RQL
Clay, low	123.8	19.7	5.0	20.0
Clay, high	111.0	18.5	NA ³	NA
Loam, low	124.6	21.6	5.6	22.2
Loam, high	100.8	7.9	NA	NA
Sand, low	123.6	21.5	5.2	21.0
Sand, high	108.6	12.2	NA	NA
Average	115.4	16.9	5.3	21.1

¹ Bias was calculated as recovery of ASE as a percent of the spike.

² Precision was calculated as the RSD (%) of ASE recovery from the spiked soils.

³ NA = not applicable.

Accelerated Solvent Extraction of Hydrocarbon Contaminants (BTEX, Diesel, and TPH) in Soils



Introduction

Benzene, Toluene, Ethylbenzene and Xylene (BTEX) are a group of compounds found in petroleum products. The main source of BTEX contamination is the leakage of gasoline from faulty and poorly maintained storage tanks but can also occur from pipeline leakage and accidental spills.

Accelerated solvent extraction has been demonstrated to be equivalent to existing extraction methodologies such as Soxhlet and automated Soxhlet for most RCRA (Resource Conservation and Recovery Act) analytes from solid and semisolid samples. This Application Note reports the use of accelerated solvent extraction of diesel fuel, gasoline and total petroleum hydrocarbons (TPH) from soils.

U.S. EPA Method 3545A: BTEX and TPH from Soils

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or larger stainless steel extraction cells
 - GC with Flame Ionization Detector (FID)
 - Infrared Spectrophotometer (IR), single wavelength or FTIR
 - Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049465)
 - Analytical balance
- *ASE 150 and 350 can be used for equivalent results.

Solvents and Reagents

- Acetone
- Hexane
- Methylene chloride
- Pentane
- Perchloroethylene (PCE)

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

Extraction Conditions for Dry Samples (less than 40% [wt] water)

Solvent: Perchloroethylene (IR determination); Pentane or hexane (GC)

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.

Extraction Conditions for Wet Samples (greater than 40% [wt] water)

Solvent: Perchloroethylene (IR determination); Hexane/Acetone, 1:1 volume (GC)

Temperature: 200 °C

Pressure: 1500 psi

Static Time: 5 min

Static Cycles: 1

Flush Volume: 60%

Purge Time: 60 s

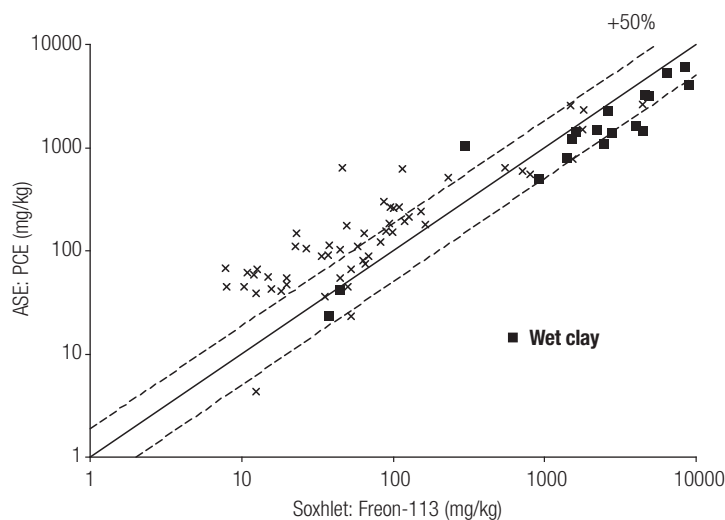
Analysis

GC-FID or IR or FTIR

Results

Recovery by ASE of volatile compounds spiked on sand.

Compound	Avg. (%), n = 4	RSD (%)
Benzene	99.7	2.5
Toluene	99.5	2.7
Ethylbenzene	100.0	3.7
<i>o</i> -Xylene	99.6	1.2
<i>n</i> -Nonane	97.1	2.6
<i>n</i> -Decane	98.1	1.0
<i>n</i> -Undecane	99.5	0.9
Naphthalene	99.9	1.7
2-Methylnaphthalene	99.4	2.3
<i>n</i> -Tetradecane	99.0	2.2
<i>n</i> -Pentadecane	97.2	3.0



Extraction of total petroleum hydrocarbons: ASE (using PCE) compared against Soxhlet (using Freon-113); analysis by IR. Plotted points represent a variety of soil samples. Wet clay soils are identified by filled boxes.

Extraction of BNAs (Base, Neutrals and Acids) Using Accelerated Solvent Extraction



Introduction

Previously, the extraction of environmental materials such as soils, sludge, and other solid wastes required large amounts of solvents. Recent and anticipated changes in environmental regulations will severely restrict the amount of solvent usage in laboratories worldwide. Accelerated solvent extraction was developed to meet the new requirements for reducing solvent usage in the preparation of solid waste samples. The procedures described in this Application Note meets the requirements for sample extraction as determined by U.S. EPA Method 3545A. This method is applicable to the extraction of water-insoluble or slightly water-soluble volatile and semivolatile compounds in preparation for GC or GC/MS measurement. This method is applicable to soils, clays, wastes, and sediments containing 250–12,500 µg/kg of BNA compounds.

U.S. EPA Method 3545A: BNAs from Soil, Clay, and Loam

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or 22 mL stainless steel extraction cells
 - GC with MS
 - Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049465)
- *ASE 150 and 350 can be used for equivalent results.

Solvents and Reagents

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

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%

Purge Time: 60 s

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

Analysis

- GC or GC/MS

Results

Average recovery of BNAs from three soil types^a—ASE compared to automated soxhlet.

BNA Target Compound	Average Recovery (% of Soxhlet)	BNA Target Compound	Average Recovery (% of Soxhlet)
Phenol	102.0	2,6-Dinitrotoluene	90.3
bis(2-Chloroethyl)ether	101.9	Acenaphthalene	101.7
2-Chlorophenol	101.6	3-Nitroaniline	92.9
1,3-Dichlorobenzene	120.6	Acenaphthene	98.4
1,4-Dichlorobenzene	119.2	4-Nitrophenol	75.6
1,2-Dichlorobenzene	112.5	2,4-Dinitrotoluene	90.7
2-Methylphenol	104.7	Dibenzofuran	98.8
bis(2-Chloroisopropyl)ether	100.2	4-Chlorophenylphenylether	94.4
<i>o</i> -Toluidine	110.3	Fluorene	95.4
<i>N</i> -Nitroso-di- <i>n</i> -propylamine	98.1	4-Nitroaniline	99.1
Hexachloroethane	118.6	<i>N</i> -Nitrosodiphenylamine	96.8
Nitrobenzene	100.2	4-Bromophenylphenylether	95.8
Isophorone	101.7	Hexachlorobenzene	93.7
2,4-Dimethylphenol	109.8	Pentachlorophenol	81.2
2-Nitrophenol	96.3	Phenanthrene	100.6
bis(Chloroethoxy)methane	97.2	Anthracene	101.2
2,4-Dichlorophenol	98.6	Carbazole	99.1
1, 2,4-Trichlorobenzene	104.2	Fluoranthene	102.7
Naphthalene	106.1	Pyrene	105.2
4-Chloroaniline	108.1	3,3'-Dichlorobenzidine	116.5
Hexachlorobutadiene	104.7	Benz[<i>a</i>]anthracene	101.5
4-Chloro-3-methylphenol	99.7	Chrysene	102.9
2-Methylnaphthalene	102.1	Benzo[<i>b</i>]fluoranthene	99.0
Hexachlorocyclopentadiene	75.8	Benzo[<i>k</i>]fluoranthene	103.1
2,4,6-Trichlorophenol	95.9	Benzo[<i>a</i>]pyrene	99.9
2,4,5-Trichlorophenol	94.1	Indeno[1,2,3- <i>cd</i>]pyrene	102.7
2-Chloronaphthalene	96.2	Dibenz[<i>a,h</i>]anthracene	98.6
2-Nitroaniline	92.6	Benzo[<i>g,h,i</i>]perylene	66.4

^aAverages from extraction of sand, loam, and clay soils.

Extraction of Base/Neutral and Acids (BNAs) from Large-Volume Samples Using Accelerated Solvent Extraction



Introduction

The procedures described in this Application Note meet the requirements for sample extraction as determined by U. S. EPA Method 3545A. This method is applicable to the extraction of water-insoluble or slightly water-soluble volatile and semivolatile compounds in preparation for GC/MS measurement. This method is applicable to soils, clays, wastes, and sediments containing from 250-12500 µg/kg of BNA compounds. Accelerated solvent extraction of BNA compounds from soils, sludge's and solid wastes is more convenient, faster, and less solvent-intensive than traditional methods such as sonication and Soxhlet.

U.S. EPA Method 3545A: BNAs from Soil, Sludge, and Sediments

Equipment

- Dionex ASE 300 Accelerated Solvent Extractor*, equipped with 66 mL or 100 mL stainless steel extraction cells
- GC with MS
- 250 mL clear collection bottles (P/N 056284)
- Cellulose filters (P/N 056780)

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

Solvents and Reagents

- Acetone
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Dichloromethane
- Ottawa sand

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%

Purge Time: 90-180 s

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

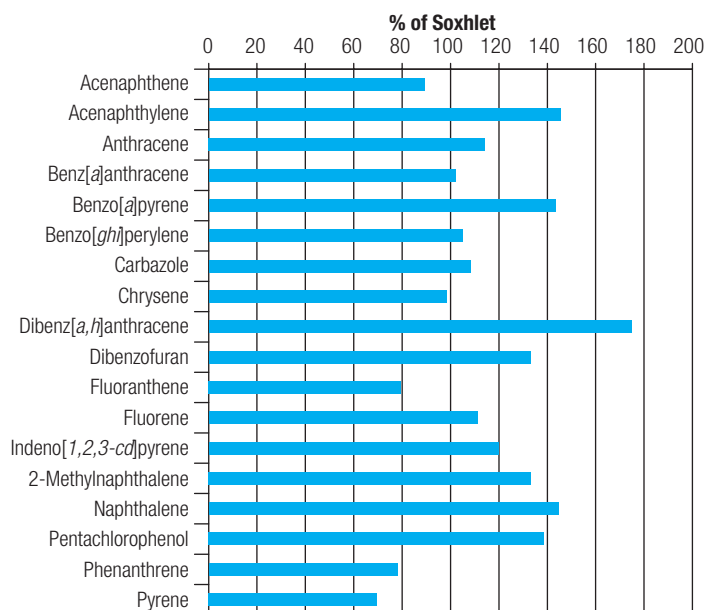
Analysis

- GC/MS

Results

Average ASE recovery of BNAs from CRM 105-100.

Target Compound	Certified Value	Average Value	Average Recovery	RSD
Acenaphthene	387088	345833	89.4	2.20
Acenaphthylene	10093	14683	145.5	3.54
Anthracene	250928	286500	114.3	38.3
Benz[a]anthracene	151693	154833	102.1	4.96
Benzo[a]pyrene	50766	72833	143.5	3.60
Benzo[ghi]perylene	13658	14333	105.0	13.3
Carbazole	23812	25783	108.3	2.70
Chrysene	191278	188333	98.4	3.93
Dibenz[a,h]anthracene	6587	11517	174.9	3.90
Dibenzofuran	154956	206167	133.1	3.56
Fluoranthene	852017	677667	79.6	21.3
Fluorene	222583	247667	111.3	2.25
Indeno[1,2,3-cd]pyrene	12994	15550	119.8	9.26
2-Methylnaphthalene	16318	21750	133.3	2.50
Naphthalene	9488	13717	144.6	4.55
Pentachlorophenol	726795	1007000	138.6	5.38
Phenanthrene	697061	543667	78.0	3.43
Pyrene	640373	446333	69.7	3.62



ASE recoveries of BNA from a certified reference material [CRM 105-100].

Determination of Organotin Compounds in Sediments Using Accelerated Solvent Extraction



Introduction

Organotin compounds are a highly versatile group of organometallic chemicals used in industrial and agricultural applications. Organotin compounds with alkyl or phenyl substituents exhibit considerable toxicity toward both aquatic organisms and mammals. Analysis of sediments is of particular importance because they are ultimate depository of these compounds. These accumulated materials can then be released into the environment, creating an environment hazard long after initial deposition has ceased. Trace level determinations of organotin compounds in environmental samples are complicated by the fact that compounds with 1–3 substituents are non-volatile due to their polar and ionic character. Accelerated solvent extraction has been shown to provide better or equivalent recoveries compared to traditional techniques for compound classes covering a wide range of polarities and exhibiting both high and low levels of bonding to the matrix.

Organotin from Aquatic Sediments

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL stainless steel extraction cells
- GC with MS
- Vials for collection of extracts (40 mL, P/N 049465)

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

Solvents and Reagents

- Acetic acid
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Methanol
- Water (HPLC grade)
- Sodium tetraethylborate
- Sodium acetate

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

Extraction Conditions

Solvent: 1 M sodium acetate,
1 M acetic acid in methanol
(1:1)

Temperature: 100 °C

Pressure: 1500 psi*

Static Time: 5 min

Static Cycles: 3–5

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/MS

Results

Absolute and relative recoveries, RSDs, and MDLs determined from lake sediments.

Compound ^a	1000 ng/g spike (n=3)				10 ng/g spike (n=5)		
	Absolute Recovery ^b		Relative Recovery ^c		Relative Recovery ^c		MDL ^d (ng/g)
	%	RSD	%	RSD	%	RSD	
MBT	97	4	100	1	105	3	0.9
DBT	100	2	102	3	101	2	0.5
TBT	98	6	102	2	100	4	1.1
MPT	80	7	87	3	104	7	2.0
DPT	95	6	97	2	98	1	0.4
TPT	94	(5)	100	(2)	101	(2)	0.5
TCyT	73	(4)	72	(2)	51	(4)	1.0

^aCompound abbreviations: monobutyltin (MBT), tributyltin (TBT), dibutyltin (DBT), monophenyltin (MPT), diphenyltin (DPT), triphenyltin (TPT), tricyclohexyltin (TCyT)

^bAbsolute recoveries determined by addition of internal standard to sediment extracts

^cRelative extraction recoveries determined by addition of internal standard to sediments prior to extraction

^dMDL = three times the standard deviation of the low spike concentration

Extraction of Explosives from Soils by Accelerated Solvent Extraction



Introduction

Compounds used in high explosives may be toxic, carcinogenic, and are found on the Resource Conservation and Recovery Act (RCRA) list. These compounds and their metabolites are monitored in soils and ground waters to ensure compliance with U.S. EPA regulations. Accelerated solvent extraction has been demonstrated to be equivalent to existing extraction methodologies for most RCRA analytes from soil and semisolid samples, and meets the requirements of U.S. EPA Method 3545A. Accelerated solvent extraction can be used for the extraction of explosive compounds such as HMX, RDX, 2,4,6-trinitrotoluene (TNT), and some of their known metabolites from soils. Compound recovery was determined for spiked soil samples. Sample-to-sample precision was determined for soil samples obtained from the sites of munitions plants in Germany.

U.S. EPA Method 3545A: RCRA Compounds from Soil

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or larger stainless steel extraction cells
- Thermo Scientific™ Dionex™ DX-500 HPLC system with AD20 (UV-vis Absorbance Detector)
- GC with Electron Capture Detector (ECD)

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

Solvents and Reagents

- Acetone
- Methanol

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

Extraction Conditions

Solvent: Acetone or Methanol

Temperature: 100 °C

Pressure: 1500 psi*

Static Time: 5 min

Static Cycles: 1

Flush Volume: 60%

Purge Time: 200 s

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

Analysis

- HPLC
- GC-ECD

Results

Recovery from spiked soil at the 3 mg/kg level.

Sample	HMX mg/kg	RDX mg/kg	TNT mg/kg	DNT mg/kg
1	3.07	3.06	3.03	3.14
2	2.99	2.89	3.02	3.07
3	2.99	2.86	2.92	3.06
4	2.84	2.74	2.81	2.97
Average	2.97	2.89	2.95	3.06
Recovery (%)	99.0	96.3	98.3	102
RSD (%)	3.2	4.6	3.5	2.3

Determination of Perchlorate in Vegetation Samples Using Accelerated Solvent Extraction and Ion Chromatography



Introduction

Perchlorate (ClO_4^-) is an environmental contaminant that has been found in drinking, ground, and surface waters in several states within the U.S. Perchlorate has been shown to present a health-based risk to humans. Exposure to perchlorate disrupts uptake of iodide by the thyroid gland. For this reason, the EPA has placed this anion on its Contaminant Candidate List for drinking water. This Application Brief provides the details of using accelerated solvent extraction for the determination of perchlorate in soil, milk, and several plant matrices. The method provides a rapid means of extracting perchlorate from all of the aforementioned matrices using only water as an extraction solvent. The benefits of this method are simplicity, speed of analysis and automation.

U.S. EPA Method 314.1: Perchlorate from Soil, Milk and Plant Matrices

Equipment

- Dionex ASE 200 Accelerated Solvent Extractor*, equipped with 11 mL or larger stainless steel extraction cells
- Thermo Scientific™ Dionex™ ICS-2500 Ion Chromatography system
- Thermo Scientific™ Dionex™ OnGuard™ II Cartridge
- Vials for collection of extracts (60 mL, P/N 049465)

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

Solvents and Reagents

- Deionized water ($\text{DI H}_2\text{O}$), Type I reagent grade, 18 Ω -cm resistance or better
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Sodium perchlorate, 98%
- Sodium salts
- Sodium hydroxide (NaOH) 50% w/w

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

Chromatographic Conditions

Columns:	Thermo Scientific™ Dionex™ IonPac™ AS16 Analytical (2 x 250 mm), Dionex IonPac AG16 Guard (2 x 50 mm), Dionex IonPac Cryptand C1 Concentrator (4 x 25 mm)
Eluent:	0.50, 65, and 100 mm NaOH
Flow Rate:	0.25 mL/min
Temperature:	35 °C
Backpressure:	2300 psi
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS ULTRA II Anion Self-Regenerating Suppressor™, external water mode, 100 mA current
Run Time:	46 min

Accelerated Solvent Extraction Conditions

Solvent:	Water
Temperature:	80 °C
Pressure:	1500 psi*
Static Time:	5 min
Extraction Cycles:	3
Flush Volume :	30%
Purge Time:	120 s

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

Analysis

IC

Results

Recovery data for accelerated solvent extraction of perchlorate.

Analyte	Perchlorate (ppb)	% Recovery*	% RSD
Soil	50	106	7.89
Alfalfa	50	94.2	8.24
Corn	50	88.7	8.86
Milk	25	118.7	1.57

* Analysis was performed using EPA method 314.1 with a Dionex ICS-2500 ion chromatography system.

Recovery and reproducibility data for accelerated solvent extraction of perchlorate.

Analyte	Perchlorate (ppb)	% Recovery*	% RSD
Melon	10	110	2.48
	50	96.8	2.54
	200	103	5.51
Corn	10	102	5.36
	50	88.7	8.86
	200	95.7	6.80
Spinach	10	106	5.40
	50	101	7.17
	200	97.9	6.53

* Analysis was performed using EPA method 314.1 with a Dionex ICS-2500 ion chromatography system.

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Notes

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