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Accelerated Solvent Extraction

Environmental Technical Resource Guide

- PAHs and PCBs • Dioxins/Furans • Pesticides
- Flame Retardants • Trivalent and Hexavalent Chromium

Accelerated Solvent Extraction for Environmental Sample Preparation



Uncomplicated Operation for Efficient Extractions

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Accelerated solvent extraction is a sample preparation technique that improves extraction efficiency for solid and semi-solid sample matrices through the use of elevated temperature and pressure with organic and water-based solvents. The technique reduces both extraction times and solvent usage in addition to providing more consistent and reliable data when compared to methods such as Soxhlet.

Many of the organic solvents used in these extractions boil at relatively low temperatures at atmospheric pressure. If sufficient pressure is exerted on the solvent, the boiling point of the solvent is elevated. As a result, temperatures above the boiling point can be used for extraction. When extractions are achieved at elevated temperatures, several factors contribute to improved speed, efficiency, and reduced solvent use:

1. Solvating power is higher
2. Diffusion rates are faster
3. Solvent viscosity is decreased
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.



Thermo Scientific™ Dionex™ ASE™ 150/350 Accelerated Solvent Extractor systems.

[Click on the image to learn more](#)

The accelerated solvent extraction technique is accepted for use in U.S. EPA Method SW-846 3545A for the extraction of semivolatile organic compounds, organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, PCBs, PAHs, Dioxins/Furans, and diesel range organics. 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 semi-volatiles and pesticides.

[To view the complete Application Notes, visit our website at \[thermoscientific.com/samplepreparation\]\(https://thermoscientific.com/samplepreparation\)](https://thermoscientific.com/samplepreparation)



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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. Accelerated solvent extraction provides a more convenient, faster, and less solvent intensive method than previously available for the extraction of PAHs from solid wastes.

Results

When using the accelerated solvent extraction technique to extract PAHs from soil, the spike recovery of 16 PAH compounds is between 86.7% and 116.2%, showing that this technique is suitable for extracting PAHs from soil. Extracting a sample using accelerated solvent extraction technique takes only 20 min and requires only 40 mL of solvent.

Analyte	% Recovery (50 ng/g)	% Recovery (200 ng/g)	%RSD
Naphthalene	113.8	92.6	1.2
Acenaphthylene	97.2	106.7	1.7
Acenaphthene	103.1	100.5	2.1
Fluorene	115.6	89.5	3.6
Phenanthrene	112.1	100.2	2.2
Anthracene	98.3	100.7	0.7
Fluoranthene	86.7	91.6	3.5
Pyrene	115.3	88.7	3.1
Benzo(a)anthracene	110.1	95.0	1.8
Chrysene	109.5	93.4	4.2
Benzo(a)fluoranthene	103.4	85.0	0.9
Benzo(k)fluoranthene	101.2	95.8	3.0
Benzo(a)pyrene	116.2	102.1	1.9
Indeno(1,2,3-cd)pyrene	97.0	101.1	3.3
Dibenzo(a,h)anthracene	110.7	106.5	4.8
Benzo(g,h,i)perylene	112.3	99.0	5.3

Spiked recovery data

Accelerated Solvent Extraction Conditions

Solvent:	Methylene chloride/acetone (1:1 v/v)
Temperature:	100 °C
Static Extraction Time	5 min
Number of Static Cycles:	2
Purge Volume:	60%
Purge Time:	90 sec
Extraction Cell Size:	34 mL stainless steel
Filters:	Cellulose (30 mm)
Total Extraction Time per Sample:	20 min
Total Solvent Volume per Sample:	40 mL
Sample Size:	10 g



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Typically, PAHs and PCBs are extracted separately using different extraction methods. The purpose of this study is to report on the development of a single extraction method for PAHs and PCBs from mussel tissue and soil using accelerated solvent extraction technique. Our study consists of two parts:

1. Extraction of PAHs and PCBs from spiked mussel tissue at two different temperatures
2. Extraction of PAHs and PCBs from soil as per standard reference materials (SRMs) provided by Resource Technology Corporation

Results

In this application note, the extraction of PAHs and PCBs are combined into one method in mussel tissue and soil with excellent percent recoveries using the accelerated solvent extraction technique. Another benefit derived from this work, is the use of alumina in the extraction cell to retain lipids, bypassing a post extraction cleanup step.

PAH Recoveries - Mussel (N = 6)			
Compound	% Recovery	SD	% RSD
Nitrobenzene-d5**	84.8	0.11	12.46
2-Fluorobiphenyl**	112.3	0.06	5.12
p-Terphenyl-d4**	105.8	0.10	9.09
Naphthalene	72.5	0.08	10.85
Acenaphthylene	82.3	0.09	10.50
Acenaphthene	81.2	0.07	9.20
Fluorene	79.5	0.06	7.41
Phenanthrene	95.3	0.06	6.49
Anthracene	85.2	0.07	8.01
Fluoranthene	90.8	0.08	8.43
Pyrene	86.2	0.07	7.82
Benzo(a)anthracene	84.7	0.09	10.48
Chrysene	114.0	0.11	9.99
Benzo(b)fluoranthene	89.2	0.07	7.97
Benzo(k)fluoranthene	84.7	0.05	5.33
Benzo(a)pyrene	77.7	0.08	10.39
Benzo(ghi)perylene	87.5	0.14	16.46
Dibenzo(a,h)anthracene	77.7	0.08	10.85
Indeno(1,2,3-cd) pyrene	83.5	0.07	7.97
PCB Recoveries - Mussel (N = 6)			
Compound	% Recovery	SD	% RSD
2,4,5,6-tetrachloro-m-xylene**	94.67	3.75	3.96
Aroclor 1254	85.68	1.87	2.18

**Surrogate Spike

Data for mussel samples extracted by Method 2.





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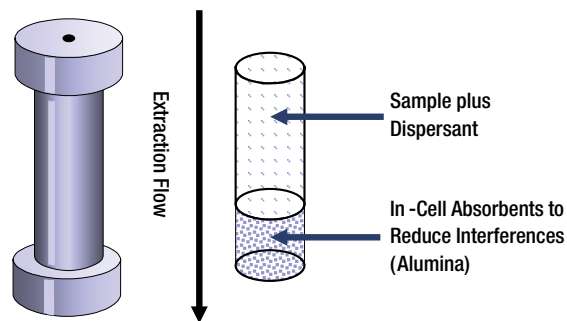
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Schematic of selective extraction using accelerated solvent extraction.

PAH Recoveries - Mussel (N = 6)			
Compound	% Recovery	SD	% RSD
Nitrobenzene-d5**	84.8	0.11	12.46
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Compound	% Recovery	SD	% RSD
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Persistent organic pollutants (POPs) in ambient air must be monitored in the interest of public health. Polychlorinated biphenyls (PCBs) and polyaromatic hydrocarbons (PAHs) are two classes of organic pollutants that have demonstrated toxic effects in humans and are subject to monitoring by regulatory agencies such as the U.S. Environmental Protection Agency (EPA).

The monitoring process consists of using air sampling cartridges to trap the pollutants, elution from the filters using organic solvents, and analysis by gas chromatography.

Results

The accelerated solvent extraction technique has proven to yield equivalent results to the traditional Soxhlet procedure while using less time and solvent. It takes advantage of enhanced solubilization kinetics that occur at temperatures higher than those commonly used to perform solvent extractions. As the efficiency of the extraction process is improved, less solvent and less time are required.

Sample #	Accelerated Solvent Extraction Technique Recovery (µg)	Soxhlet Recovery (µg)	U.S. EPA Method 608 Acceptance Criteria
1	4.63	4.87	
2	4.44	4.93	
3	4.72	5.16	
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 spiked value of 5.00 µg Aroclor 1248

Comparison of spike recoveries between accelerated solvent extraction and Soxhlet extraction of PCB congeners from Polyurethane Foam (PUF) cartridges*

	Soxhlet Technique	Accelerated Solvent Extraction Technique
Solvent Consumption Cleaning	900–1500 mL	87.5 mL
Time Used for Cleaning	15–24 h	2.5 h
Solvent Consumption Elution	300–500 mL	50 mL
Time Used for Elution	5–8 h	18 min
Total Solvent Consumption	1200–2000 mL	550 mL
Total Time Used	20–32 h	3 h

Time and solvent use comparison (accelerated solvent extraction technique vs Soxhlet).



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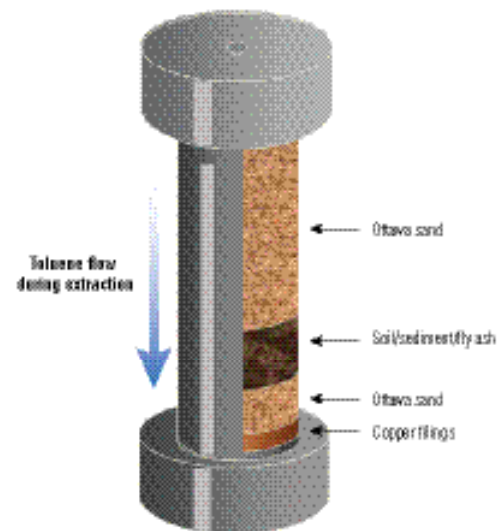
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The measurement of dioxins and furans (PCDD/Fs) in the environment is a widespread activity carried out by many regulatory agencies globally. The chronic toxicity of these compounds to humans and wildlife at extremely low concentrations requires that the techniques used in determination be both sensitive and selective. This application note demonstrates the analysis of PCDDs/PCDFs in sediments, soils, and fly ash (as incineration by-products) using the Dionex ASE Accelerated Solvent Extractor system and the Thermo Scientific™ TSQ Quantum™ XLS Ultra GC-MS/MS.

Results

The Dionex ASE 150/350 system allows for efficient sample extractions within the performance limits required for PCDDs/PCDFs in sediments, soils, bottom, and fly ash. Additionally, using ASE allows the cleanup of unwanted interferences in-cell which eliminates additional sample handling and time.



Extraction Solvent:	Toluene
Extraction Pressure:	1500 psi
Oven Temperature:	175 °C
Heating Time:	8 minutes
Static Time:	5 minutes
Flush:	60% Cell volume
Purge:	N ₂ 240 Seconds

Cell schematic and method conditions for the extraction of PCDD/Fs from soil, sediment, and fly ash samples.





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	Soil		Sediment 1		Sediment 2		SETOC 738 (CRM)	
	GC-MS/MS ng/kg	GC-HRMS ng/kg	GC-MS/MS ng/kg	GC-HRMS ng/kg	GC-MS/MS ng/kg	GC-HRMS ng/kg	GC-MS/MS ng/kg	GC-HRMS ng/kg
2,3,7,8 TCDF	0.992	1.23	16.0	10.4	2.15	1.51	17.9	17.1
2,3,7,8 TCDD	ND	ND	1.85	2.29	2.03	5.10	23.1	23.9
1,2,3,7,8 PeCDF	1.26	<1.79	25.4	25.4	4.90	4.52	7.36	6.76
2,3,4,7, 8 PeCDF	1.57	1.96	41.5	44.2	10.7	9.29	47.8	45.4
1,2,3,7,8, PeCDD	0.436	ND	5.49	5.71	9.14	7.95	7.26	6.91
1,2,3,4,7,8,-HxCDF	1.89	1.89	56.1	57.3	64.8	71.9	43.8	45.1
1,2,3,6,7,8,-HxCDF	1.81	2.14	64.8	55.0	223	197	15.1	14.7
2,3,4,6,7,8,-HxCDF	2.53	2.83	86.9	91.5	9.47	11.5	20.2	18.4
1,2,3,4,7,8,-HxCDD	0.271	ND	5.38	6.34	15.0	10.9	10.4	8.62
1,2,3,6,7,8,-HxCDD	0.382	0.905	8.33	8.58	17.6	16.7	28.1	20.1
1,2,3,7,8,9-HxCDD	0.469	ND	6.62	6.97	17.5	14.0	21.4	22.4
1,2,3,7,8,9-HxCDF	0.932	ND	25.8	31.4	15.2	13.4	4.65	5.40
1,2,3,4,6,7,8-HpCDF	12.9	14.2	464	473	5.05	4.58	214	202
1,2,3,4,6,7,8-HpCDD	4.80	5.78	63.4	62.2	18.3	16.6	416	433
1,2,3,4,7,8,9-HpCDF	1.90	3.07	48.8	48.8	68.3	54.4	15.1	15.4
OCDD	24.7	23.8	153	191	6.38	5.38	3020	3030
OCDF	258	291	475	554	47.7	39.5	290	316

Calculated concentrations of TCDD/F congeners in soil and sediment samples run on both GC-MS/MS and GC-HRMS (ND=not detected)





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Organochlorine pesticides (OCPs) are a class of chemicals that were used to control insect pests starting in the 1940s. The use of OCPs was banned in the later part of the last century due to their persistence in the environment and neurotoxic effects.

This application brief discusses the use of Thermo Scientific™ Dionex™ ASE™ Prep MAP, a proprietary polymer that has a high-capacity for water removal and does not suffer from the limitations of clumping observed when using sodium sulfate.

Results

Highlighted is a simple and reliable method to extraction OCPs from oyster tissue. This application compares the extraction efficiency of OCPs using both Dionex ASE Prep MAP and sodium sulfate as drying agents. As shown in the Table to the right, drying samples with Dionex ASE Prep MAP produced much higher recoveries of OCPs from spiked oyster tissue.

It is also important to note that the proprietary polymer's water-absorbing ability does not depend on the ionic strength of the sample matrix.



Accelerated Solvent Extraction Conditions

Oven Temperature:	100 °C
Pressure:	1500 psi
Static Time:	5 min
Static Cycles:	3
Rinse Volume:	60%
Solvent:	Hexane/Acetone (1:1, v/v)
Total Extraction Time:	22-25 min

Compound	% Recovery Oyster dried with Dionex ASE Prep MAP and Dionex ASE Prep DE* (n = 3)	% Recovery Oyster dried with sodium sulfate** (n = 3)
Lindane	91	81
Heptachlor	93	64
Aldrin	94	66
Dieldrin	105	75
Endrin	106	70
DDT	114	69
Total	101	71

In-cell moisture removal of oyster sample using Dionex ASE Prep MAP and Dionex ASE Prep DE, in comparison to sodium sulfate.



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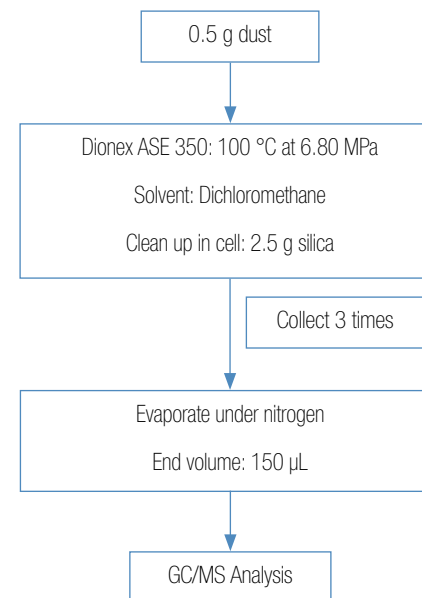
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Polybrominated diphenyl ethers (PBDEs) were introduced in the 1960s as flame retardants and were used in a wide variety of household apparatuses, consumer electronics, furniture and more. Environmental levels of PBDEs have been continuously increasing due to their persistence.

Certain congeners have been banned completely and are currently in the list of the Stockholm convention's persistent organic pollutants¹. Sources of human intake are typically via ingestion and the inhalation of indoor and house dust.

Results

Using the Dionex ASE 350 system in combination with an in-cell cleanup offers a quick and easy methodology for extracting PBDE in household dust samples. in-cell clean up removes unwanted interferences, eliminating additional sample handling and time.



Sample preparation flowchart.





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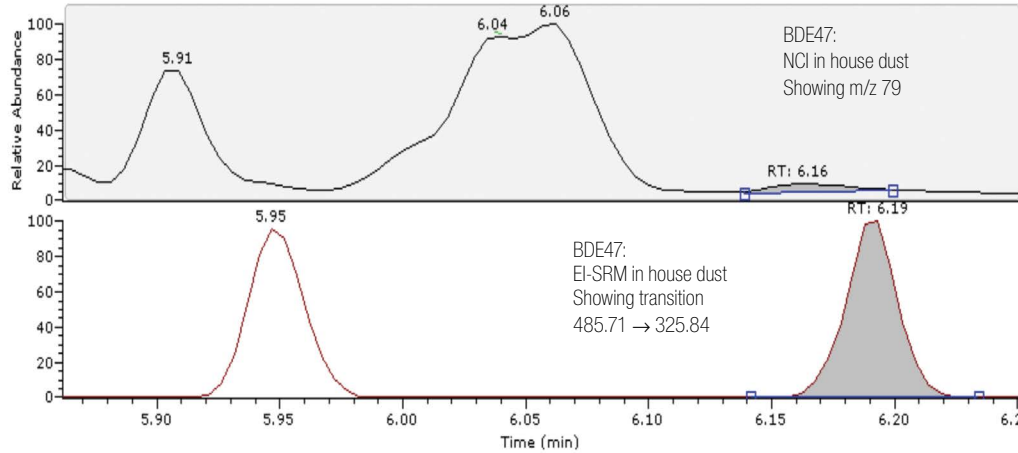
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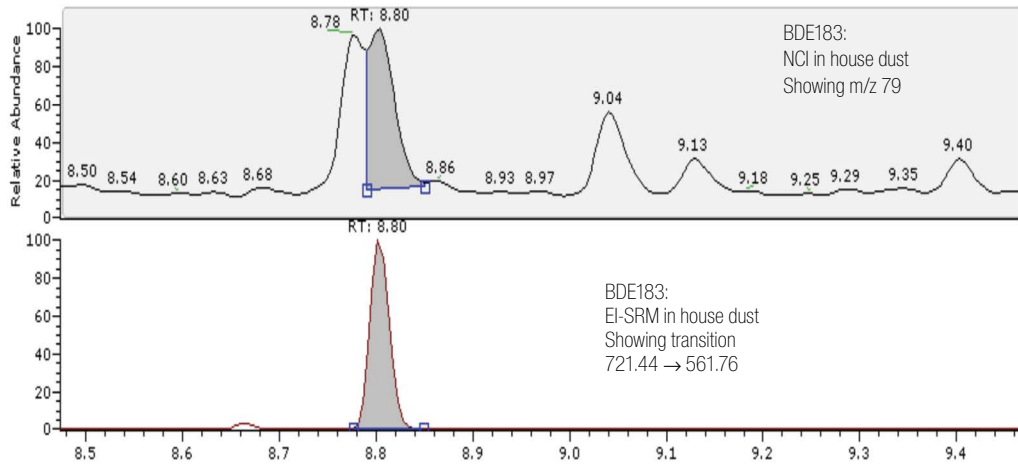
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House dust sample in NCI (above) and EI-SRM (below) showing BDE47 at a level of 0.07 ppb



House dust sample in NCI (above) and EI-SRM (below) showing BDE183 at a level of 0.17 ppb





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Trivalent chromium is nontoxic in normal doses but can exhibit cytotoxicity at higher doses. Hexavalent chromium is extremely toxic and has been confirmed to have a carcinogenic effect. Since trivalent chromium and hexavalent chromium can interconvert under certain conditions and are both toxic to a certain degree, the simultaneous detection of trivalent chromium and hexavalent chromium content is of vital importance. Accelerated solvent extraction is used to extract trivalent chromium and hexavalent chromium from a plastic toy building block, thread from a toy rag doll, a rubber car tire sample, soil and solid waste samples. The extract was directly injected into a ion chromatography system for analysis.

Results

Detection results in the matrix samples showed that the combination of an improved accelerated solvent extraction technique with precolumn derivatization and IC with postcolumn derivatization is a perfect match for the simultaneous extraction, separation, and analysis of trivalent and hexavalent chromium.

Accelerated Solvent Extraction Conditions

Instrument:	Dionex ASE 350 Accelerated Solvent Extractor System
Extraction Solvent:	2 mmol/L PDCA, 2 mmol/L KI, 100 mmol/L NH ₄ AC, 2.8 mmol/L LiOH
Temperature:	100 °C
Static Extraction Time:	5 min
Number of Cycles:	2
Rinsing Volume:	60%
Purge Time:	90 s
Extraction Cell:	Thermo Scientific™ Dionium™ Extraction Cell, 66 mL (P/N 068102)
Total Extraction Time per Sample:	18 min

Sample	Cr(III)/µg/g	Cr(VI)/µg/g
Soil	0.182	BLD*
Solid Waste	0.103	BLD*
Leather	213	0.596
Textiles	3.10	BLD*
Plastics	0.095	BLD*
Rubber	0.186	BLD*

*Below limit of detection

Detection results and spiked recovery for each type.

Thermo Scientific Sample Preparation and Chromatography Portfolio

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Automated accelerated solvent extractor systems. Enables extraction of solid and semisolid samples using common solvents at elevated temperatures and pressure.



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A revolutionary solvent evaporator that concentrates or dries up to 18 ASE vials or 6 large-volume flasks unattended.



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Automated SPE instrument that extracts large-volume samples (20 mL– 4 L) for the isolation of trace organics in aqueous matrices. Produces analyte recoveries that are superior to manual liquid-liquid extraction techniques using less time and solvent.



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The Thermo Scientific liquid chromatography platform is the most complete LC solution provided by a single chromatography powerhouse. Thermo Scientific™ Dionex™ UltiMate™ 3000 LC Systems are all UHPLC compatible by design and provide an unprecedented level of flexibility to all users, in all laboratories for all analytes. We also provide the latest in state-of-the-art UHPLC with the Thermo Scientific™ Vanquish™ UHPLC system, delivering better separations, more results, and easier interaction than ever before.



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View a brief overview of the Dionex ASE 150/350 systems and the key benefits for using in your lab.

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Dionex ASE Prep sorbent cartridges can be used for post extraction cleanup of samples, resulting in cleaner extracts with low background and less frequent detector maintenance.

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Dionex ASE Prep MAP Polymer Solutions for Sample Pre-treatment

The Dionex ASE Prep MAP Polymer is a unique polymer designed to remove moisture and increase extraction efficiencies from wet samples under a variety of ionic strength conditions and accelerated solvent extraction conditions.

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- * Improving Dioxin Extraction Using In-Cell Pressurized Liquid Extraction: A Case Study for Solid Sample Matrices
- * Use of a Novel New Moisture Absorbing Polymer for Extraction Applications
- * Multi-Residue Pesticide Analysis in Herbal Products Using Accelerated Solvent Extraction with Triple Quadrupole GC-MS/MS

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