Table of Contents

Accelerated Solvent Extraction

Environmental Technical Resource Guide

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

Thermo
SCIENTIFIC

Table of Contents

Uncomplicated Operation for Efficient Extractions

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.

Table of Contents

Polyaromatic Hydrocarbons

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.

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

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
Acenaphtylene	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
Benz(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

Thermo
SCIENTIFIC

Download Application Update 313: Accelerated Solvent Extraction — GC-MS Analysis and Detection of Polycyclic Aromatic Hydrocarbons in Soil

PAHs and PCBs

Table of Contents

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

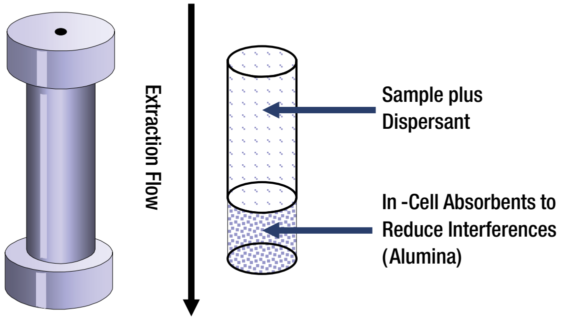
In this application note, the extraction of PAHs and PCBs are combined into one method in mussel tissue and soil with excellent percent recoveries using the accelerated solvent extraction technique. Another benefit derived from this work, is the use of alumina in the extraction cell to retain lipids,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

Download Application Note 1025: Simultaneous Extraction of PAHs and PCBs from Environmental Samples Using Accelerated Solvent Extraction

Table of Contents



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
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.

Download Application Note 1025: Simultaneous Extraction of PAHs and PCBs from Environmental Samples Using Accelerated Solvent Extraction

POPs

Table of Contents

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 extracton technique vs Soxhlet).

Download Application Note 71064: Accelerated Solvent Extraction for Monitoring Persistent Organic Pollutants in Ambient Air

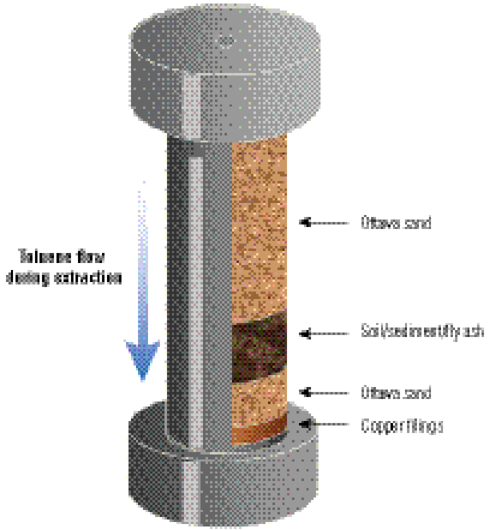
Dioxins and Furans

Table of Contents

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.

Download Application Note 10336: Determination of PCDD/Fs in Environmental Samples Using Accelerated Solvent Extraction (ASE) and GC-MS/MS

Table of Contents

	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)

Download Application Note 10336: Determination of PCDD/Fs in Environmental Samples Using Accelerated Solvent Extraction (ASE) and GC-MS/MS

Pesticides

Table of Contents

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* (= 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.

Download Application Brief 152: Extraction of Organochlorine Pesticides from Oyster Tissue Using Accelerated Solvent Extraction

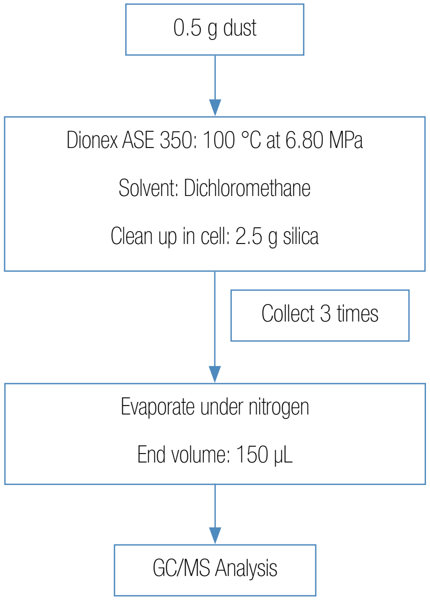
Table of Contents

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 pollutants[1]. Sources of human intake are typically via ingestion and the inhalation of indoor and house dust.

Results

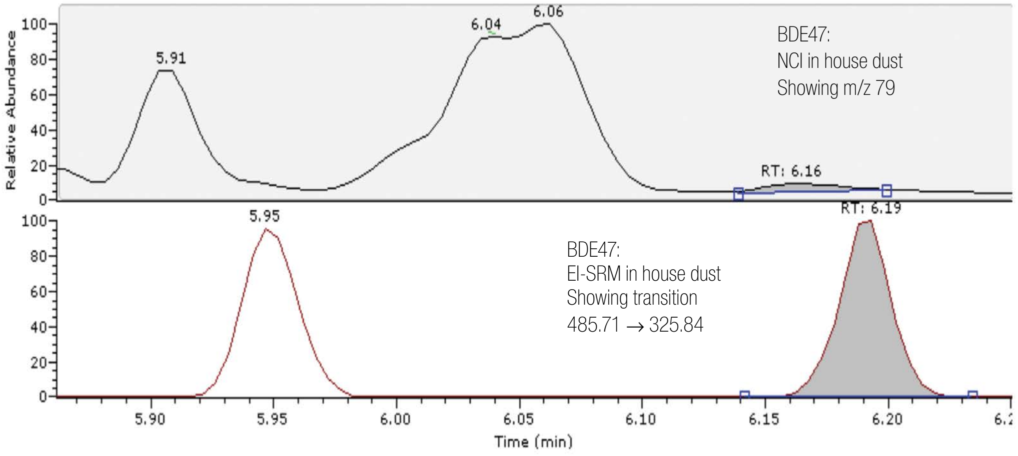
Using the Dionex ASE 350 system in combination with an in-cell cleanup offers a quick and easy methodology for extracting PBDE in household dust samples. in-cell clean up removes unwanted interferences, eliminating additional sample handling and time.



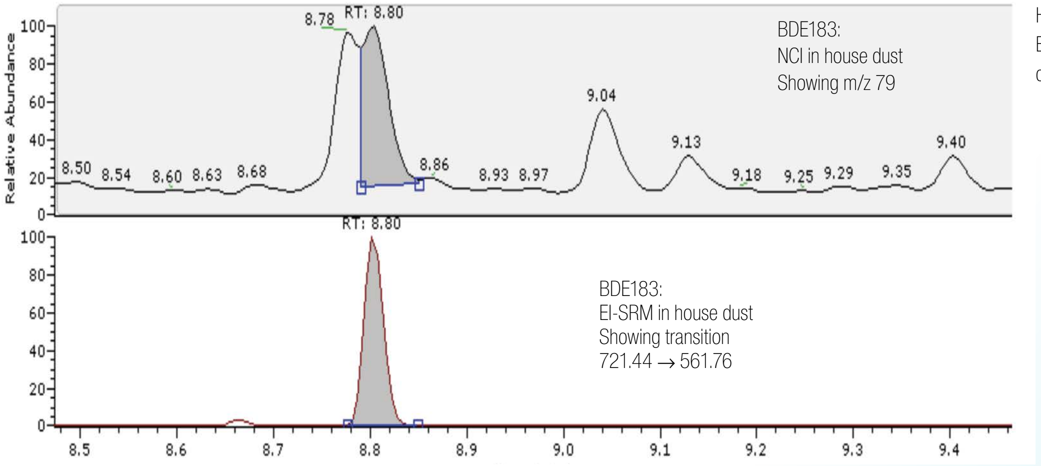
Sample preparation flowchart.

Download Application Note 52294: Analyzing PBDEs in House Dust Samples with the Thermo Scientific TSQ Quantum XLS Ultra GC-MS/MS in EI-SRM Mode

Table of Contents



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

Download Application Note 52294: Analyzing PBDEs in House Dust Samples with the Thermo Scientific TSQ Quantum XLS Ultra GC-MS/MS in EI-SRM Mode

Trivalent and Hexavalent Chromium

Table of Contents

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.

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_4AC, 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

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.

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.

Download Application Note 1079: Simultaneous Determination of Trivalent Chromium and Hexavalent Chromium Using Accelerated Solvent Extraction and Ion Chromatography

Thermo Scientific Sample Preparation and Chromatography Portfolio

Table of Contents

Dionex ASE 150/350 Systems

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

Thermo Scientific™ Rocket Evaporator

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

Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction (SPE) Instrument

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.

Thermo Scientific™ TRACE™ 1300 Series GC Systems

The first and only gas chromatograph featuring user-exchangeable miniaturized, instant connect injectors and detectors that eliminate maintenance downtime and enable the user to quickly tailor instrument capability to specific applications and daily workload.

Thermo Scientific™ TSQ™ Duo Triple Quadrupole GC-MS/MS System

A simply unique system tailored especially for chromatography and single-quadrupole GC-MS users who need easy access to powerful new workflows through MS/MS. The system provides excellent performance in both single and triple quadrupole modes that is easily achievable, even for less experienced users.

Thermo Scientific Liquid Chromatography Systems

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.

Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System Software

One scalable software platform for LC, GC, IC, and MS that provides Operational Simplicity™ by streamlining your entire analysis process – ultimately boosting your lab's overall productivity and increasing the quality of your analytical results.

Thermo
SCIENTIFIC

Table of Contents

Recommended Resources

Download this Brochure

Dionex ASE 150/350 Accelerated Solvent Extraction System

Experience unsurpassed extraction technology with the Dionex ASE 150 / 350 Accelerated Solvent Extractor systems

Download this White Paper

Use of Accelerated Solvent Extraction to Improve Laboratory Workflow

Accelerated solvent extraction can help the analytical laboratory greatly improve productivity by reducing both the time and solvent required to achieve sample extraction.

Download this Poster

Complete Sample Preparation Workflow Solutions

Sample Preparation, Gas Chromatography-Mass Spectrometry, and Data Analysis

Download this Video

Dionex ASE 150/350 Accelerated Solvent Extraction Systems Video

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

Download this Product Spotlight

Dionex ASE Prep Sorbents

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.

Download this Product Spotlight

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 strengthconditions and accelerated solvent extraction conditions.

View these Webinars

* 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

www.thermofisher.com/samplepreparation

Africa	+43 1 333 50 34 0
Australia	+61 3 9757 4300
Austria	+43 810 282 206
Belgium	+32 53 73 42 41
Brazil	+55 11 3731 5140
Canada	+1 800 530 8447
China	800 810 5118 (free call domestic) 400 650 5118
Denmark	+45 70 23 62 60
Europe-Other	+43 1 333 50 34 0
Finland	+358 9 3291 0200
France	+33 1 60 92 48 00
Germany	+49 6103 408 1014
India	+91 22 6742 9494
Italy	+39 02 950 591
Japan	+81 6 6885 1213
Korea	+82 2 3420 8600
Latin America	+1 561 688 8700
Middle East	+43 1 333 50 34 0
Netherlands	+31 76 579 55 55
New Zealand	+64 9 980 6700
Norway	+46 8 556 468 00
Russia/CIS	+43 1 333 50 34 0
Singapore	+65 6289 1190
Sweden	+46 8 556 468 00
Switzerland	+41 61 716 77 00
Taiwan	+886 2 8751 6655
UK/Ireland	+44 1442 233555
USA	+1 800 532 4752

Thermo SCIENTIFIC

A Thermo Fisher Scientific Brand

AN71294 EN 08/16S