Sample Preparation PFAS in Soil

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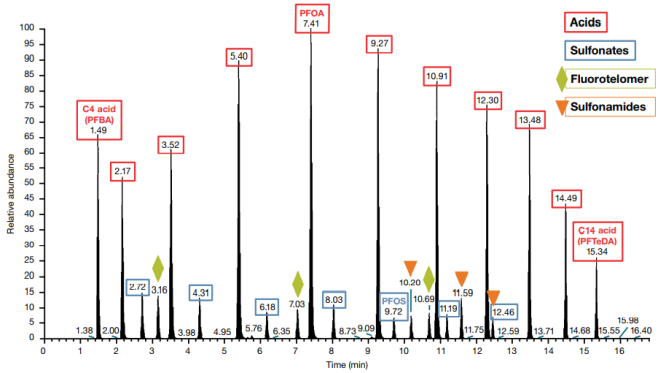
General Workflow for Sample Preparation

SAMPLE HANDING → **SAMPLE PRAPARATION** → **ANALYSIS**

1. Solvent Extraction
2. Vortex/Sonicate
3. Clean up (SPE)
4. Preconcentration



Accelerated

Solvent

Extraction

AUTOMATED

Extraction Method

Solvent Extraction

or Liquid-Liquid Extraction (LLE)

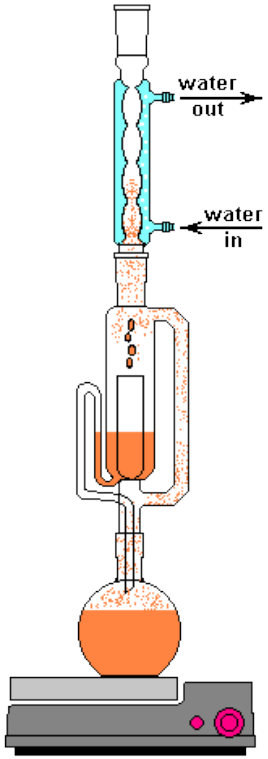
Distribution Coefficient

$$K_D = \frac{[S]_1}{[S]_2}$$

where K_D is the distribution coefficient and the subscripts represent solvent 1 (e.g., an organic solvent) and solvent 2 (e.g., water). If the distribution coefficient is large, the solute will tend to be quantitatively partitioned in solvent 1.

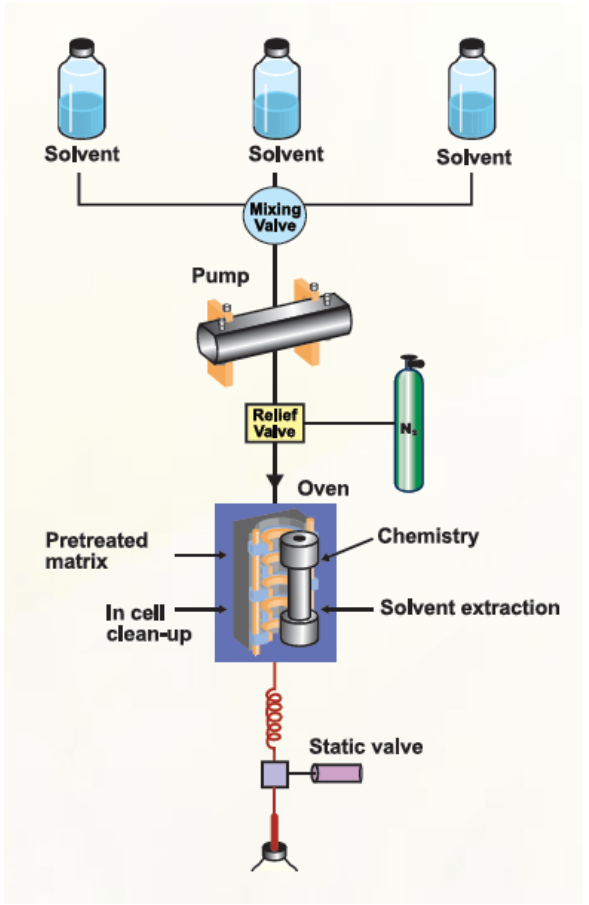
Analytical Chemistry, 7th Edition, Gary D. Christian, Purnendu K. Dasgupta, Kevin A. Schug ©2014

Soxhlet extraction



Wikipedia

Accelerated and Microwave-Assisted Extraction



Pressure and Temperature are the key parameters

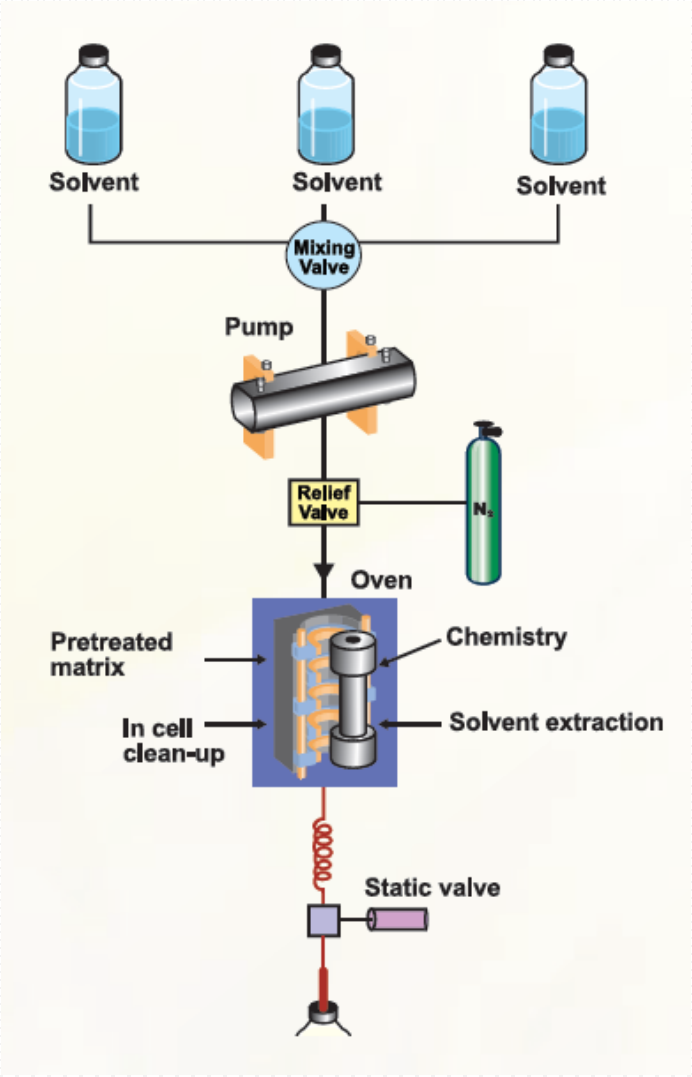
Increased Pressure

- enables the organic solvents to remain a liquid at temperatures higher than their boiling points

Increased Temperature

- Increases the solubility of the analyte into the solvent
- Decreases the viscosity of the solvent to improve migration throughout the sample matrix
- Decreases the surface tension of the solvent to improve analyte diffusion

Accelerated Solvent Extraction



- Extraction of analytes from a solid or semi-solid sample matrix into a solvent

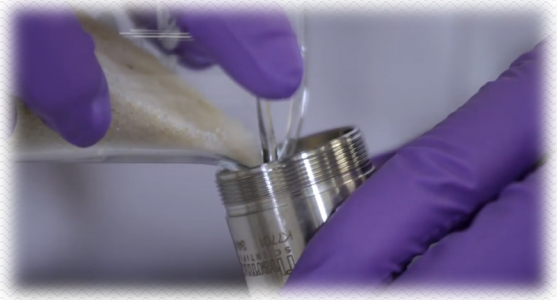
- Enhancement of extraction efficiency by
  - High pressure → allow heating above the boiling point
  - High temperature → accelerate the dissolution of analytes in the solvent

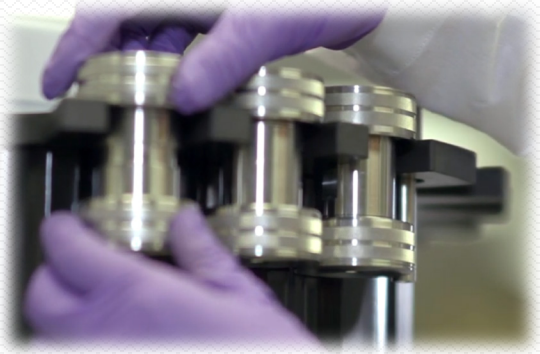
- Both time of extraction and the volume of solvent needed are greatly reduced over atmospheric extraction.

ASE350: Accelerated Solvent Extractor

ASE350 : Product Highlight

- ✓ Sample cells (1, 5, 10, 22, 34, 66, and 100 mL) stainless steel cells and 66 and 100 mL Dionium cells with fingertight fittings
- ✓ Sensors for temperature, pressure, and solvent and liquid leaks alert the operator if there is a problem, sound an audible alarm, and shut down the system if necessary





PFAS Extraction Cleanup Workflow

Weigh out 2 g of soil sample in a 250 mL glass beaker

Add 10 g diatomaceous earth to the beaker and mix with the soil sample.

Transfer the sample mixture into a 100 mL stainless steel ASE cell with cellulose filter at the bottom of the cell

Top up the cell with diatomaceous earth and Spike extraction surrogate and native standard

Extraction settings		
Cell type	Stainless steel	
Oven temperature	100 °C	
Static cycle	Time	300 s
	Solvent	80:20 methanol/ acetonitrile
	Volume	50 mL
Cycles	3	
Purge time	120 s	

PFAS Extraction and Cleanup Workflow

Condition SPE
- 15 mL methanol
- 20 ml water

↓

Sample Load
Transfer the extracted sample solution to the SPE cartridge

↓

Elution
Rinse the sample with 10 mL methanol and collected

↓

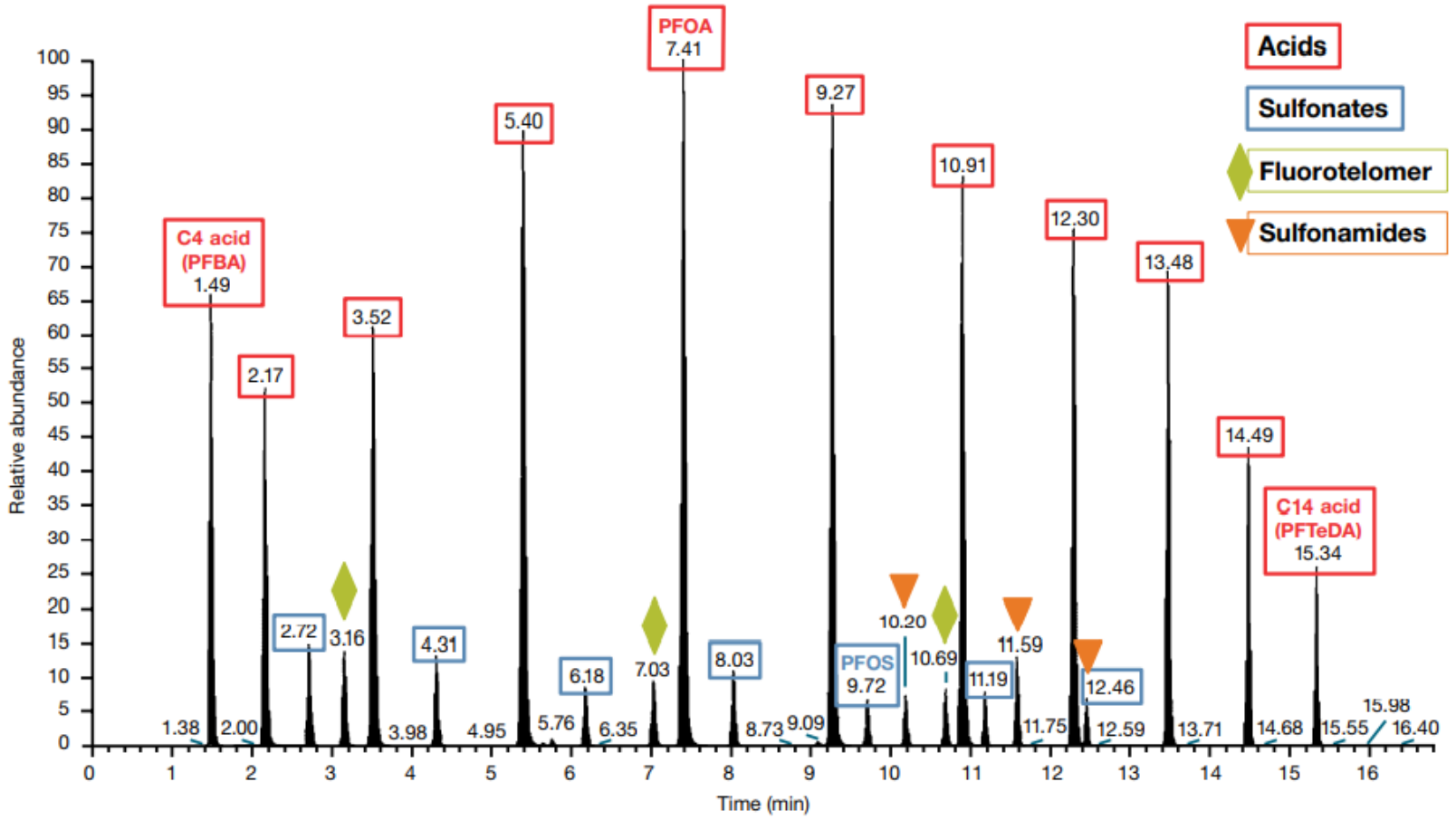
Inject to LC-MSMS

Styrene-divinylbenzene (SDVB)

polymer SPE cartridge (500 mg, 6 mL)

Automated SPE cleanup & injection

Quantitative in PFAS



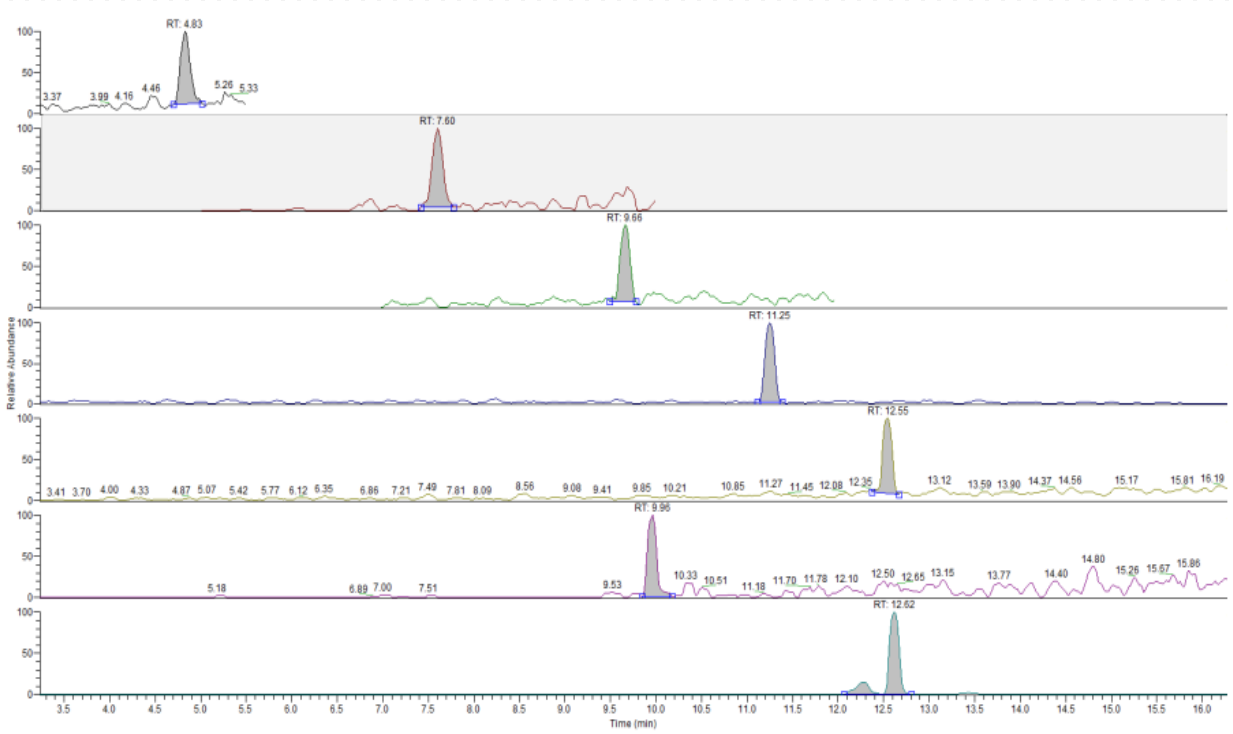
- 24 Compound PFAS
- SRM 1 ng/g

Quantitative in PFAS

Chromatogram of a soil sample

Native analytes found at appreciable levels in the soil sample .

These levels are between 1 and 5 ng/g,



Internal standard recoveries (5 ng/g)

Compound	Recovery (%)
$^{13}C_4$-PFBA	71
$^{13}C_5$-PFPeA	93
$^{13}C_5$-PFHxA	97
$^{13}C_4$-PFHpA	96
$^{13}C_8$-PFOA	94
$^{13}C_9$-PFNA	104
$^{13}C_6$-PFDA	99
$^{13}C_7$-PFUdA	95
$^{13}C_2$-PFDoA	97
$^{13}C_2$-PFTeDA	108

Compound	Recovery (%)
$^{13}C_3$-PFBS	98
$^{13}C_3$.PFHxS	95
$^{13}C_8$-PFOS	91
$^{13}C_3$-HFPODA	56
$^{2}H_3$-NMEFOSAA	93
$^{2}H_3$-NETFOSAA	90
$^{13}C_8$-FOSA	92
$^{13}C_2$-4:2FTS	110
$^{13}C_2$-6:2FTS	93
$^{13}C_2$-8:2FTS	98

Quantitative in PFAS

Spike level	1 ng/g	5 ng/g	20 ng/g	100 ng/g	400 ng/g	Slope	r^2
PFBA	0.979	5.05	21.7	101.0	408	1.020	1.000
PFPeA	1.035	5.22	22.0	101.4	423	1.058	1.000
PFHxA	1.024	5.15	22.1	102.4	429	1.073	1.000
PFHpA	0.985	5.28	22.4	99.2	423	1.056	1.000
PFOA	1.02	5.02	22.1	100.7	425	1.062	1.000
PFNA	1.032	5.21	21.8	102.6	426	1.064	1.000
PFDA	1.000	5.06	21.5	100.5	428	1.071	1.000
PFUdA	0.982	5.01	22.6	96.8	418	1.044	1.000
PFDoA	1.05	5.43	23.5	77.3	339	0.841	0.999
PFTRDA	0.567	3.65	15.5	45.4	200	0.496	0.998
PFTEDA	1.076	5.57	23.9	76.6	317	0.786	0.999
NMEFOSAA	1.13	4.86	22.8	97.2	368	0.915	1.000
NETFOSAA	1.097	5.18	19.5	117.0	424	1.061	0.999
FOSA	0.991	5.16	21.7	93.0	438	1.097	0.998
PFBS	0.966	5.14	22.4	104.6	453	1.133	1.000
PFPeS	0.915	4.93	21.1	99.6	433	1.084	1.000
PFHxS	0.945	4.98	21.6	110.6	451	1.129	1.000
PFHpS	0.976	4.55	22.8	111.4	467	1.169	1.000
PFOS	1.076	6.14	20.3	108.2	468	1.172	1.000
PFNS	0.893	5.51	21.4	107.1	462	1.156	1.000
PFDS	0.999	5.54	20.8	104.4	447	1.119	1.000
4:2FTS	1.129	5.89	22.2	57.8	272	0.672	0.997
6:2FTS	1.128	6.19	21.8	89.2	430	1.074	0.998
8:2FTS	1.149	5.43	21.0	79.8	384	0.958	0.998

PFAS in soil linearity

Range 1 – 400 ng/g

Linearity $R^2 > 0.997 – 1$

Conclusion…

- Accelerated solvent extraction can extract a variety of PFAS from soil including acids, sulfonates, fluorotelomer sulfonates, and sulfonamide compounds.
- Isotopic dilution quantification was utilized for most analytes and demonstrated linearity for all PFAS studied in soil over the range of 1 ppb to 400 ppb.
- Save Solvent and Save Time.

Solution on ASE 350

Table of Contents

Accelerated Solvent Extraction

Environmental Technical Resource Guide

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

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Solution on ASE 350

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.

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.

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

Analyte	% Recovery (50 ng/g)	% Recovery (200 ng/g)	%RSD
Naphthalene	113.8	92.6	12
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
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

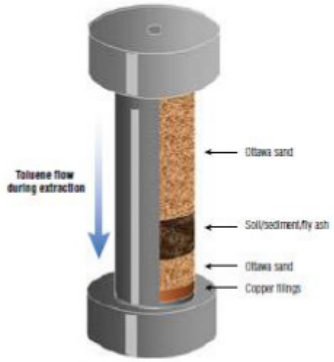
Download Application Update 313: Accelerated Solvent Extraction — GC-MS Analysis and Detection of Polycyclic Aromatic Hydrocarbons in Soil

Dioxins and Furans

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

Solution on ASE 350

Food and Beverage The accelerated solvent extraction technique is used for multiple applications in the food and beverage industry.

- Pesticide residues in a variety of sample types marketed for human or animal consumption
- Fats and lipids using acid hydrolysis
- Fats and total lipids using alkaline saponification
- Acrylamide
- Antibiotic residues
- Antioxidants

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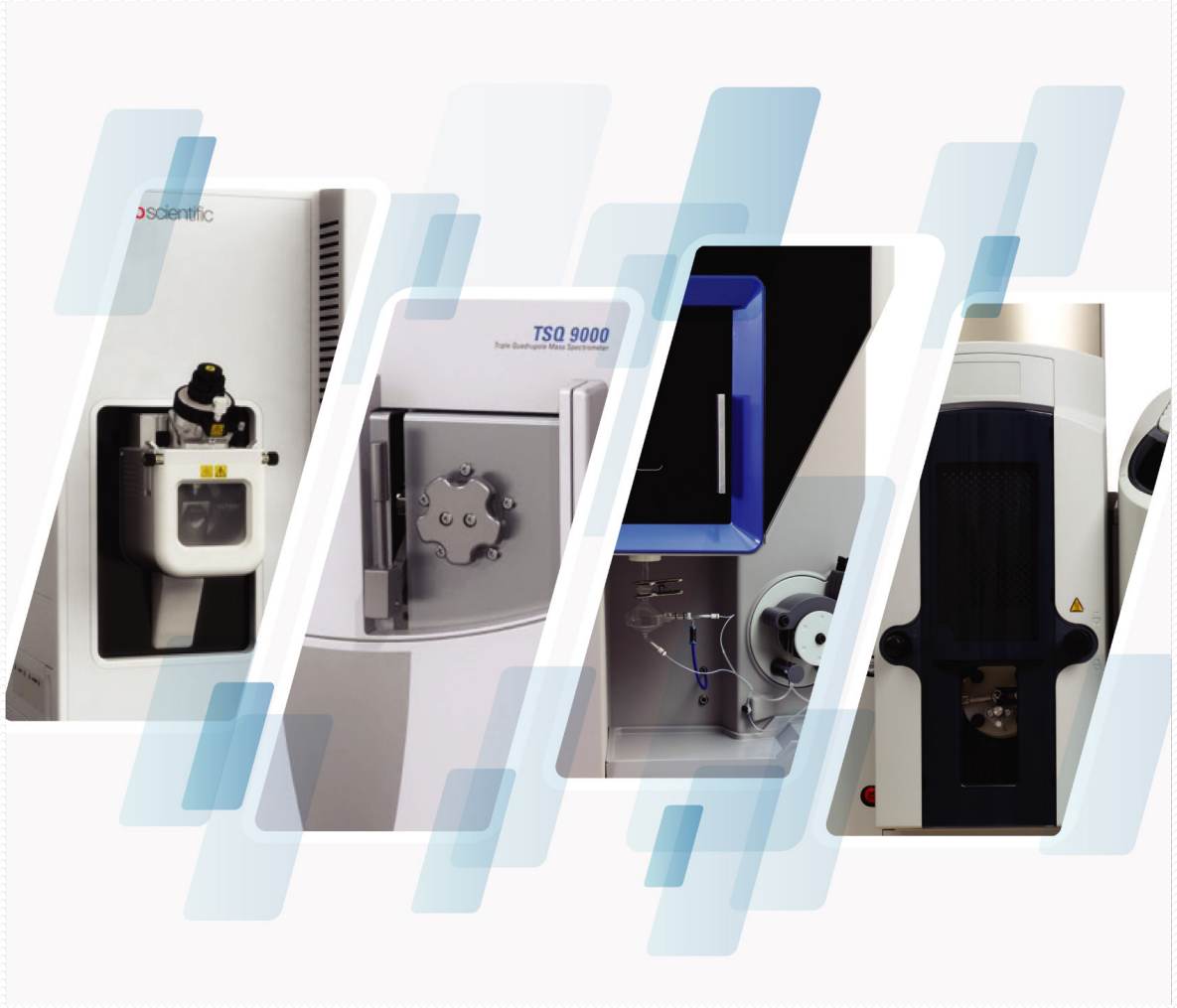

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