



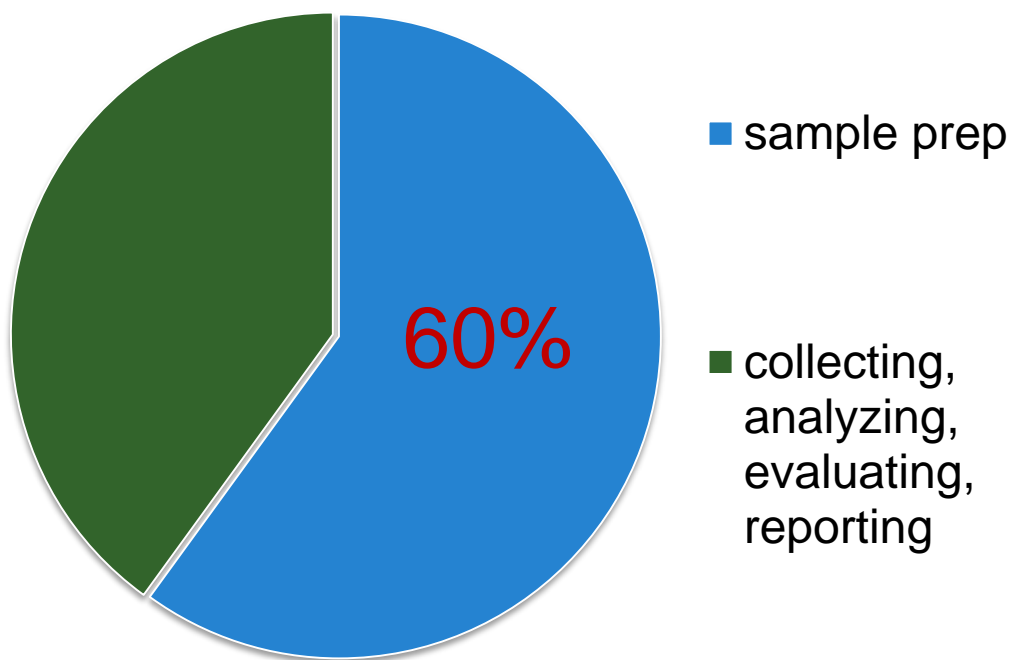
Sample Prep Solutions for Environmental Contaminants

October 2017

The world leader in serving science

What is the longest step in your analysis of environmental contaminants?

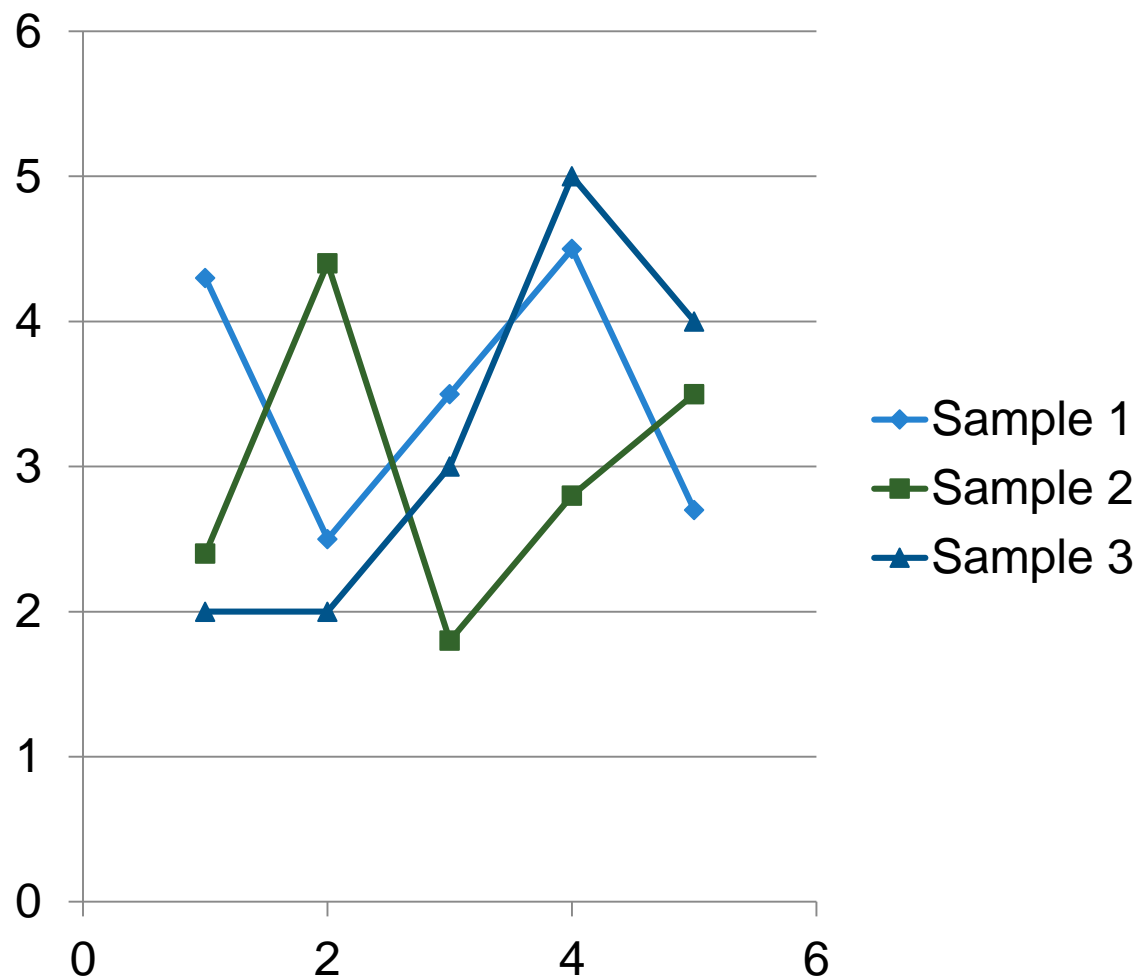
Sample Analysis



- In fact, preparing the sample is the most time consuming part of the analytical procedure (>60%)*
- And it is the single largest source of errors in the workflow (>30%)**

- * Majors, R.E. *LC-GC*, **1995**, 13, 742-749
- **Majors, R.E. *LC-GC*, **1999**, 17, S8 - S13

Where does the variance in results come from?



- Eighty percent of the variance in an assay usually arises from the sample preparation”*

• R. Stevenson, “Pittcon® ‘98: Part 3 Sample Prep: The Place to Make a Difference” American Laboratory, Vol. 30, No. 14 p.21, 1998

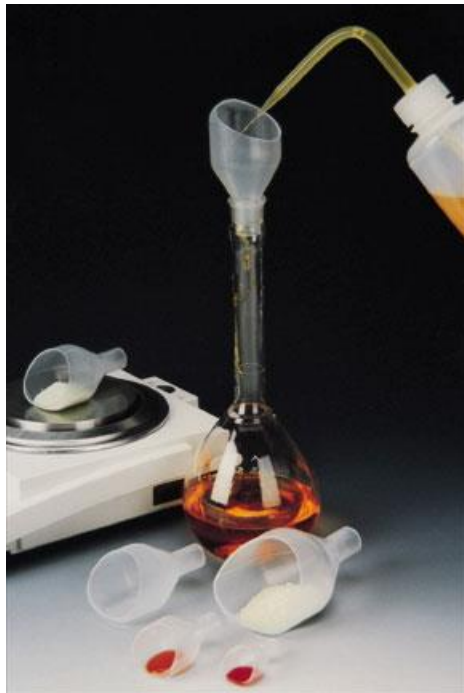
Think about that....



- 80% of the variance arises from the sample prep
- Yet what is the first thing that we blame when something is wrong with the result?

– The GCMS itself!

What can go wrong during sample prep?



Weighting & diluting



extracting



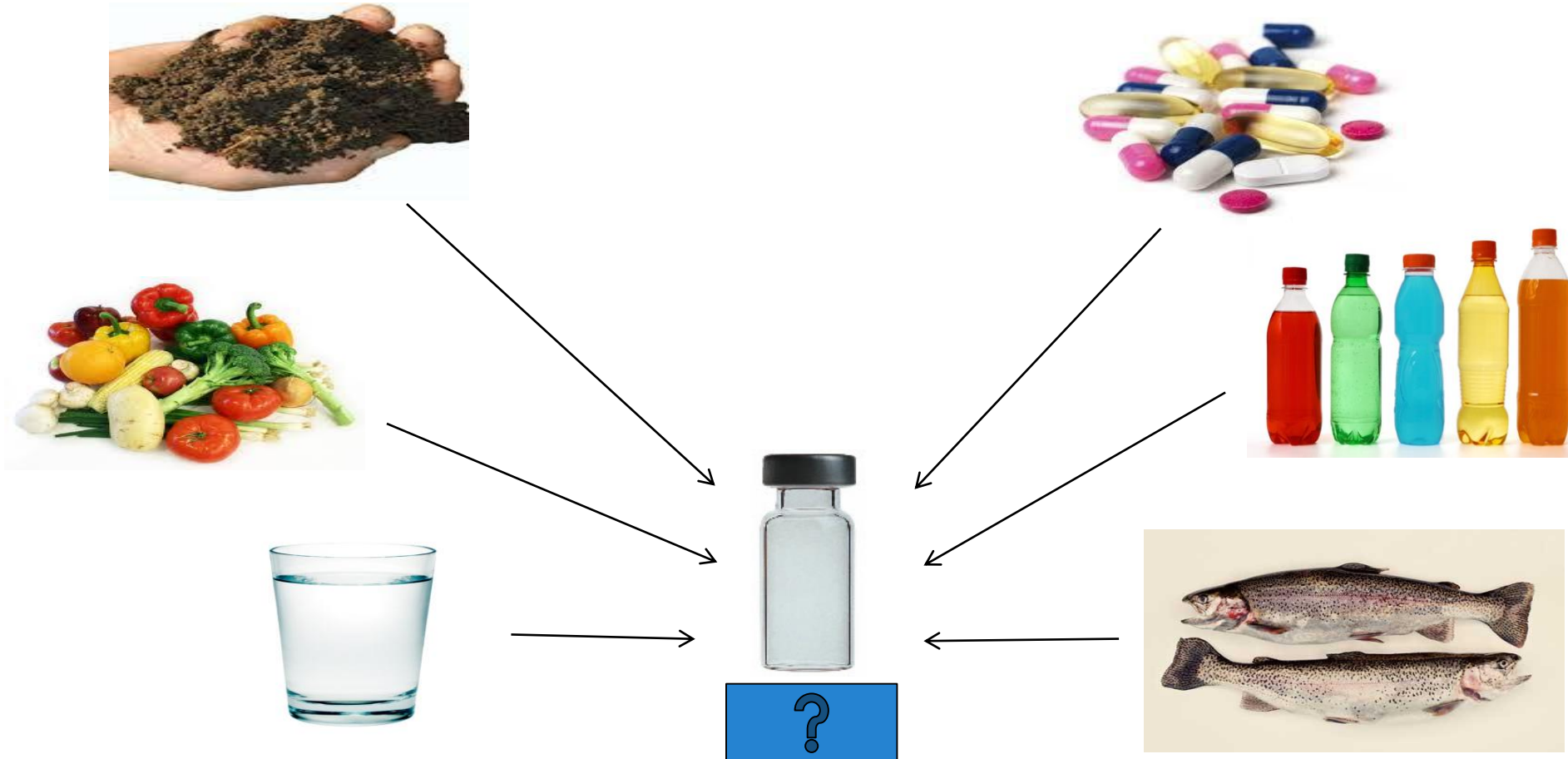
- Today we are going to talk about 2 of these sample prep techniques...



...and some pretty good tools to achieve it!

For Extraction, the question is:

How do we get analytes out of these samples and into the GC vial?



Things to consider.....

Things to consider.....

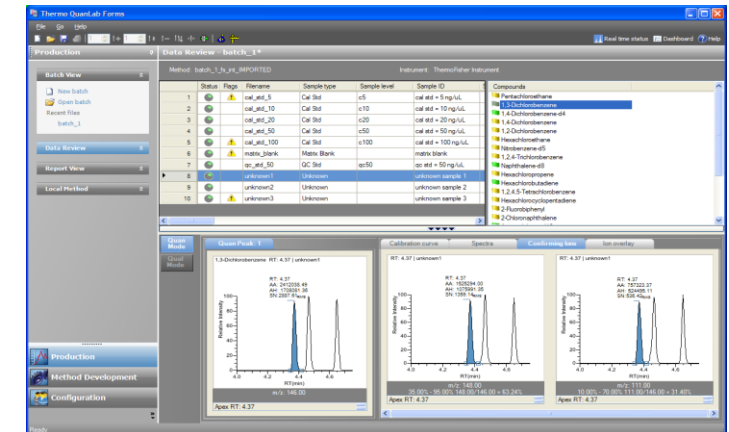
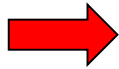
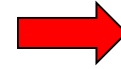
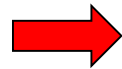
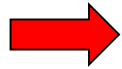
- Is my sample a solid or a liquid
- What is the chemical nature of my analyte of interest – solubility? Polarity?
- What is the chemical nature of my sample itself
- Is the analyte bound up in any way? Chemical or physical?
- How do I maximize the surface area of my sample without losing my analyte?

And finally,

- How much time and effort is this going to take?
- I've got other work to do, is there any way to automate this?



Automated Sample Extraction: Part of the GC-MS Total Workflow



Integrated Workflow Solutions for Sample Preparation

Thermo Scientific Dionex Sample Prep Product Line



Thermo Scientific™ Dionex™ ASE™ 150 and 350
Accelerated Solvent Extractor



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



Thermo Scientific Dionex
SolEx™ SPE Cartridges



Genevac Rocket™ Evaporator

Novel & Innovative Solutions

Accelerated Solvent Extraction for solid samples

- Automates sample preparation for solid and semisolid samples using solvents at elevated temperatures.
- Operates above the boiling point of extraction solvents by using elevated pressure.
- Walk-away system that extracts and clean up to 24 samples unattended.
- Well established and proven technique that is superior to Soxhlet and approved for U.S. EPA Method 3545A.



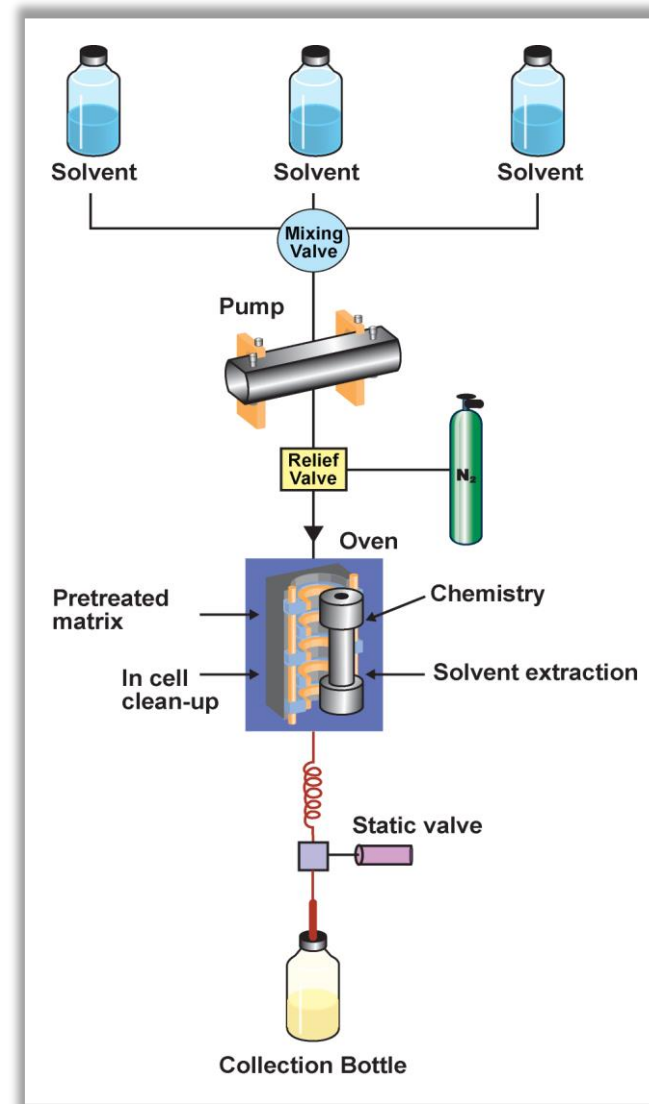
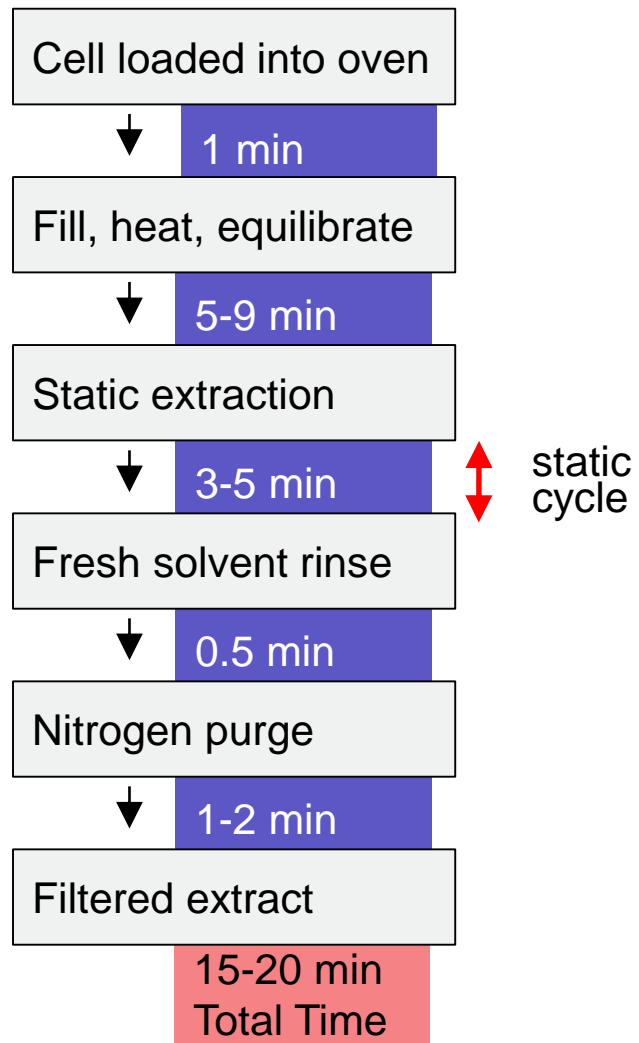
ASE 350

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



How Does Accelerated Solvent Extraction Work?



Key Advantages to your workflow:



VS



- ✓ 20-30 min per sample
- ✓ 5-200 ml per sample
- ✓ Filtered extract
- ✓ Extract is cool to touch
- ✓ Can perform in-cell cleanup

- 4-48 hrs per sample
- 150-1000 ml per sample
- Have to manually filter extract
- Extract is hot, has to cool down
- Manual cleanup of extract

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Accelerated Solvent Extraction

Environmental Technical Resource Guide

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

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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 w/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	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
Dibenz[a,h]anthracene	110.7	106.5	4.8
Benzo[e]pyrene	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

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POPs

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



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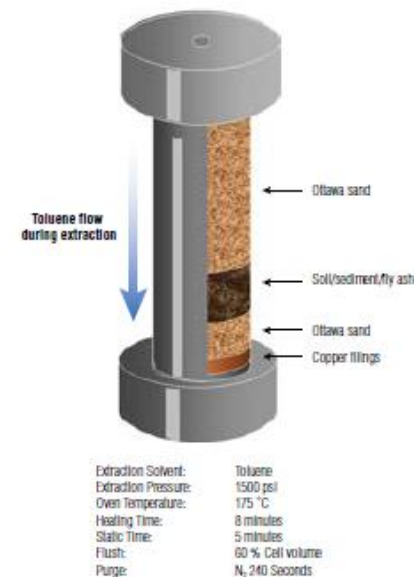
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The measurement of dioxins and furans (PCDD/Fs) in the environment is a widespread activity carried out by many regulatory agencies globally. The chronic toxicity of these compounds to humans and wildlife at extremely low concentrations requires that 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.

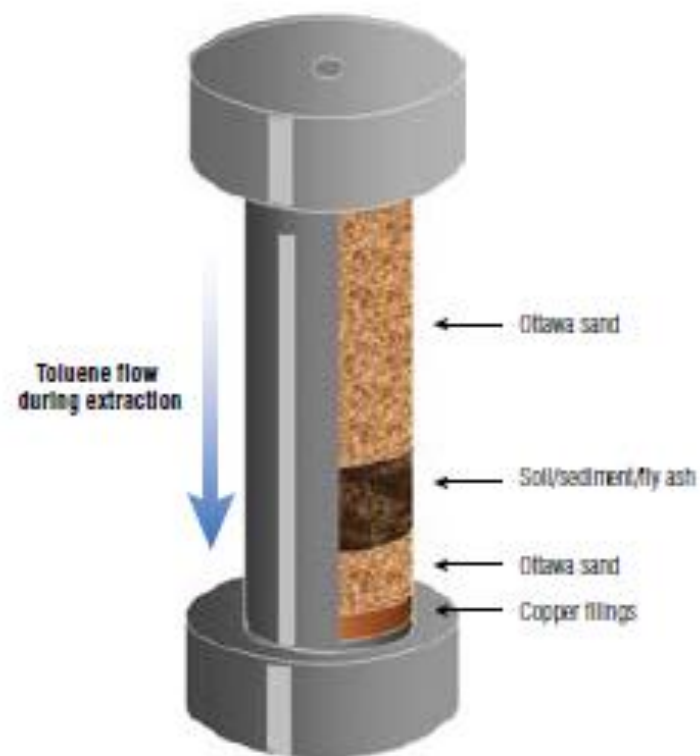
Results

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.



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





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.



Dioxins and Furans

	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)

Accelerated Solvent Extraction

Environmental Technical Resource Guide

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

<http://www.thermofisher.com/order/catalog/product/083114>

Scroll down to product literature

Automated Solid Phase Extraction for Liquid Samples

- Its automated!
 - No constant attendance necessary
- Uses a pump for each sample, not a vacuum for all
 - Consistent flow rates
 - Improved analytical precision
- Uses syringes to deliver solvents
 - Precise solvent delivery every time
- Achieve lower detection limits
 - Can load up to 4L of sample



AutoTrace 280

What exactly is Solid Phase Extraction

- Solid-phase extraction (SPE) is a **sample preparation process** by which **compounds** that are dissolved or suspended in a liquid mixture **are separated** from other compounds in the mixture **according to their physical and chemical properties**. Analytical laboratories use solid phase extraction to concentrate and purify samples for analysis. Solid phase extraction can be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water, beverages, soil, and animal tissue.

- Wikipedia

SPE vs. Liquid-Liquid Extraction (LLE)



Separatory Funnel used for LLE

- **LLE :**

- Subject to emulsion formation
- Incomplete phase separations
- Poor analyte recoveries
- Uses expensive, breakable glassware
- Uses great volumes of solvent
- Must evaporate large volumes of solvent

- **SPE:**

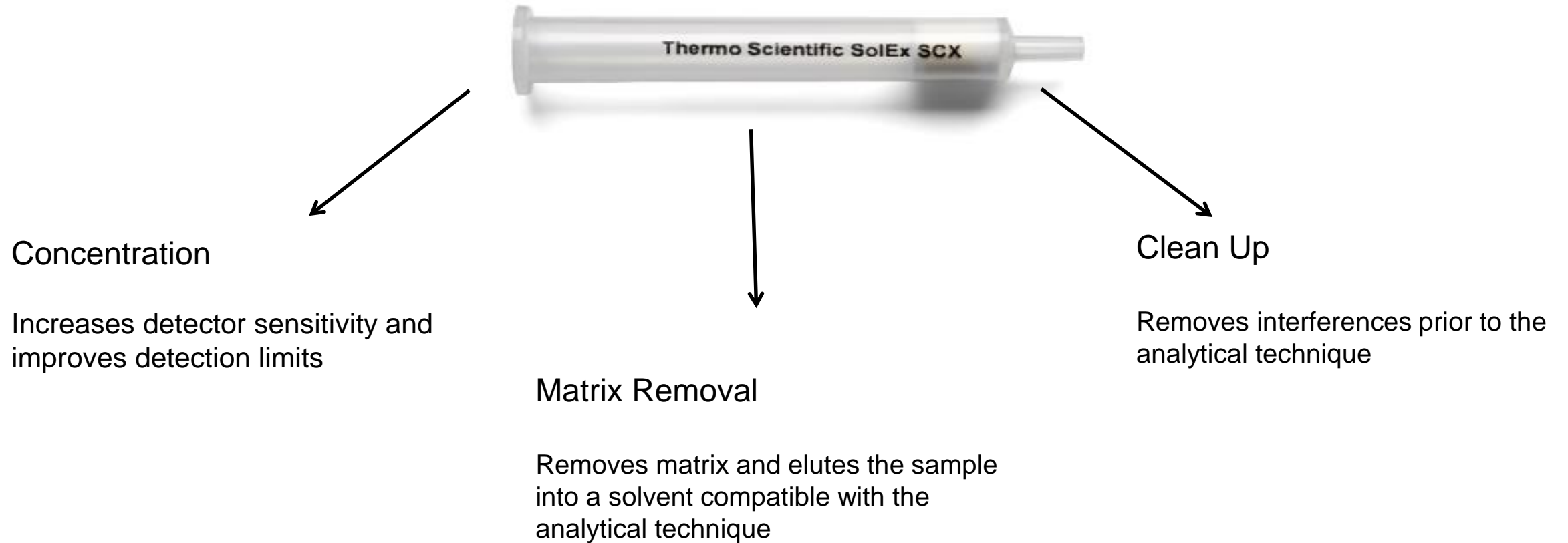
- Faster
- Can be automated
- No emulsions
- Has excellent recoveries
- Very low solvent consumption

SPE operates like a crude liquid chromatograph

- The sample is passed over a cartridge or disk containing a specific sorbent
- The sorbent isolates either a single or a group of compounds from everything else
- Flow is achieved using either a vacuum or pump



Why is SPE Used?



Thermo Scientific SolEx SPE cartridges

Silica-Based Sorbents



Silica substrate for Normal Phase separations
Bonded Silica (C8 or C18) for Reversed Phased Separations

Polymeric Sorbents



Styrene-divinyl benzene (DVB) copolymers that can be modified to create ion exchangers through animation or sulfonation

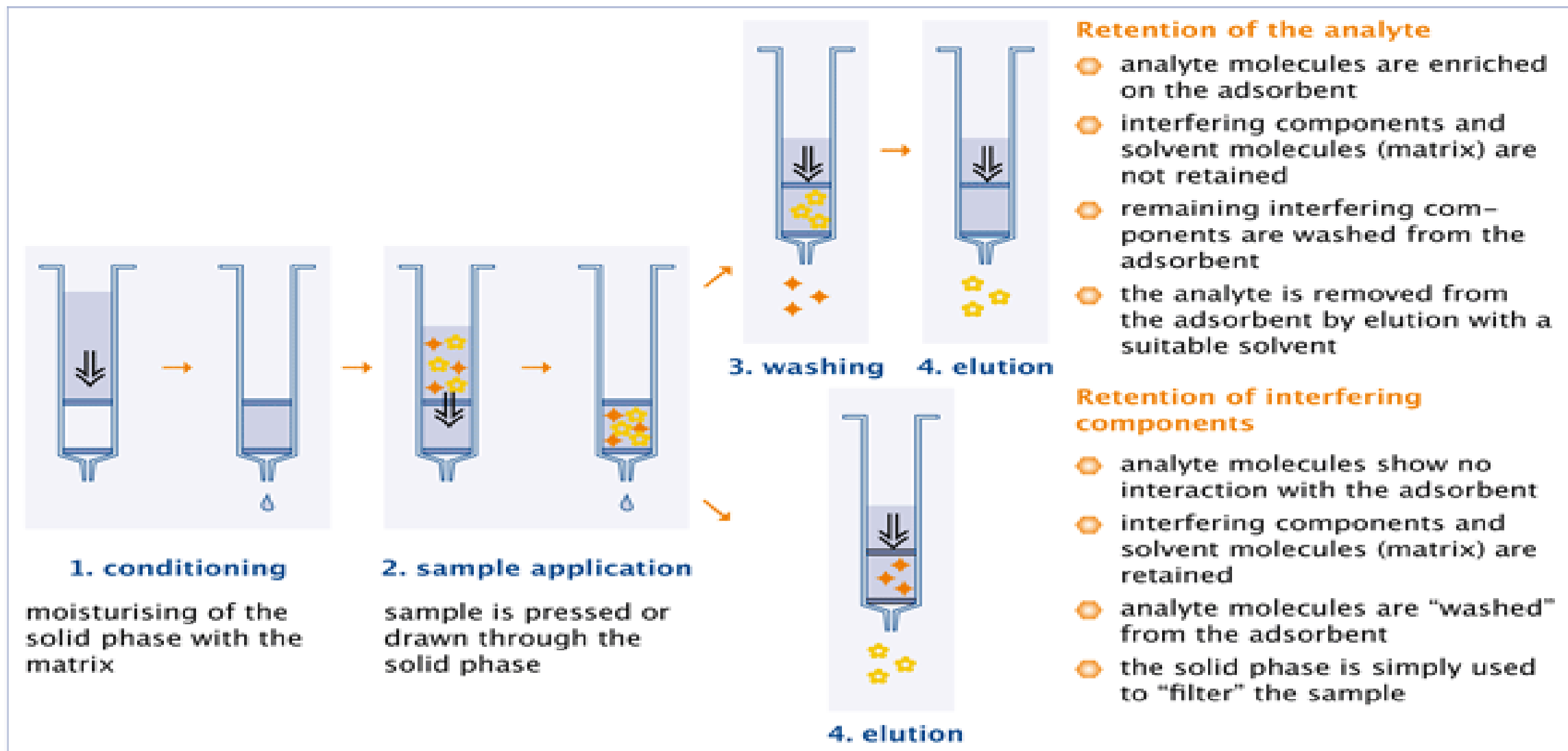
Activated Carbon



Adsorption primarily based on structure



The Process of SPE



How does the Autotrace 280 work?

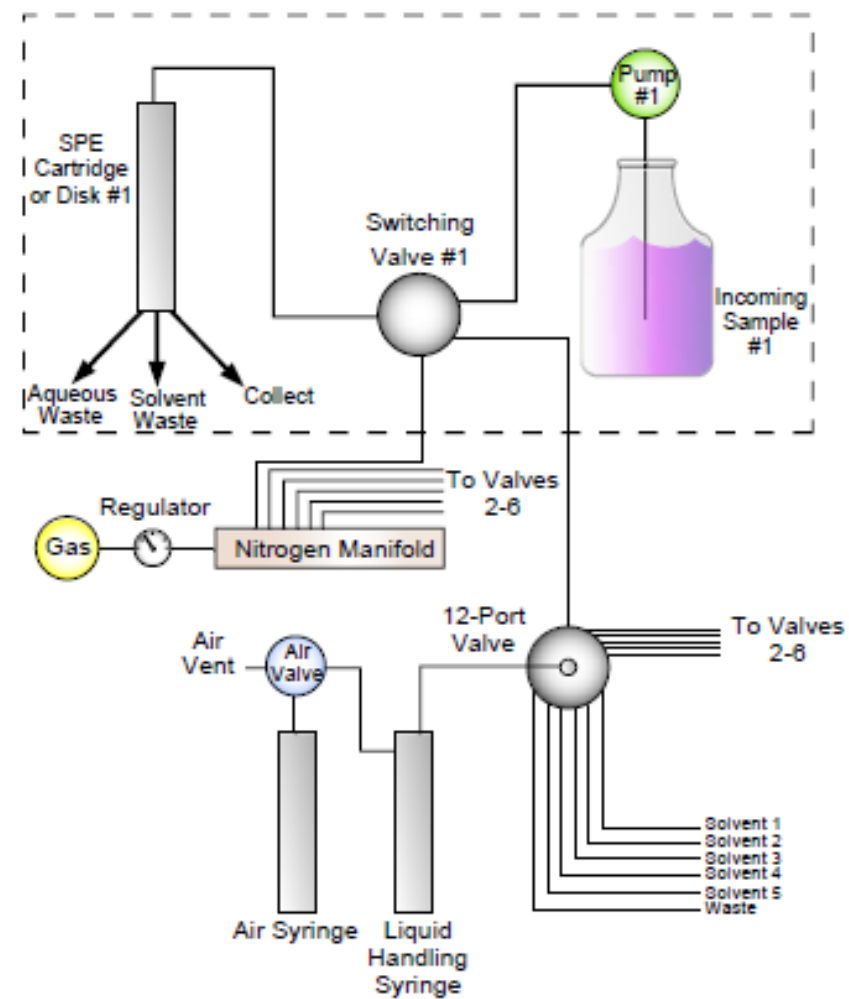
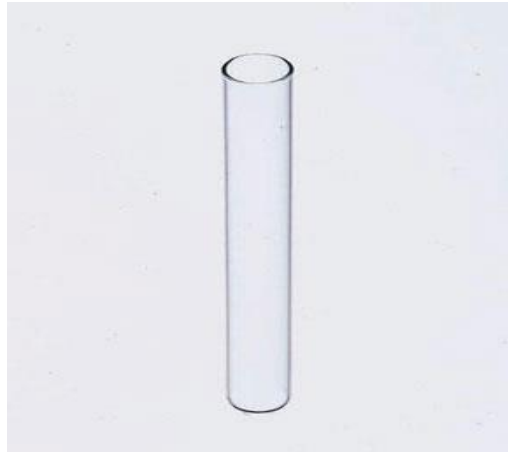
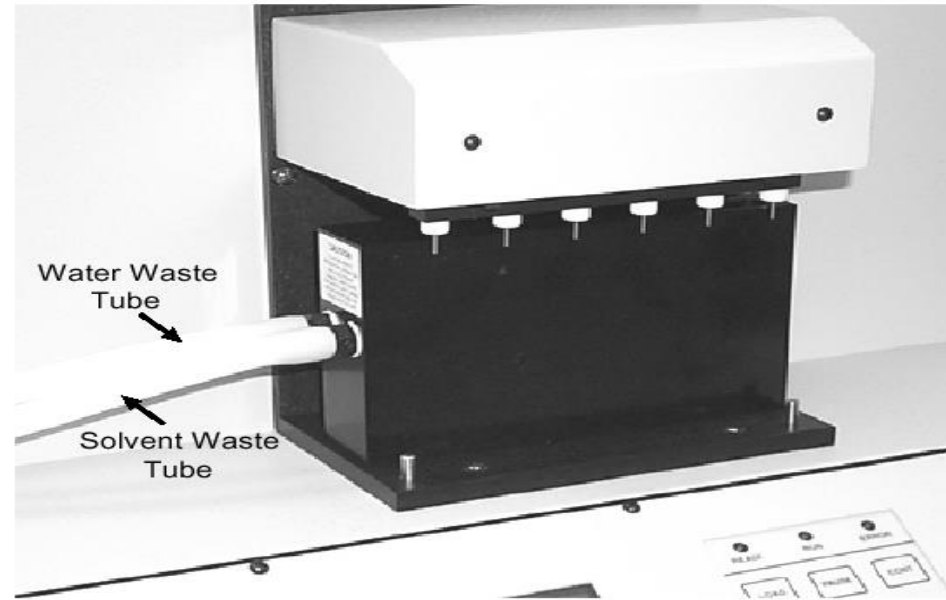


Figure 2-6. AutoTrace 280 Fluid Connections

Elution Rack: Six Sample Container Options



16 x 100 mm test tube



15 mL centrifuge tube



11 mm GC vial



4 mL screw cap vial



40 mL ASE vial



17 x 60 mm vial

PAHs and Phthalates in Surface Waters

Automated Solid-Phase Extraction (SPE) of Wastewaters and Surface Waters for Polyaromatic Hydrocarbons and Phthalates — Modification of EPA Method 625

Application Brief 876

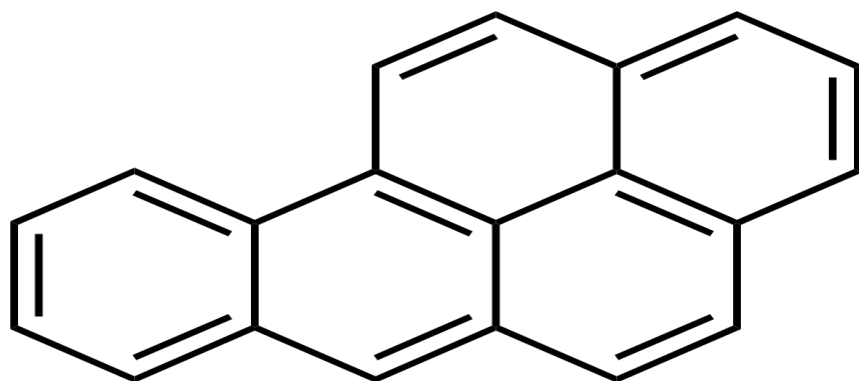
Introduction

EPA Method 525.2, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, can be used as a basis for developing a liquid-solid extraction method for EPA Method 625, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs).

Unlike the drinking water methods, however, analysis of wastewaters and the streams they flow into, need to contend with particulates. The Thermo Scientific™

No.	Process six samples using the following procedure:
1	Wash syringe with 2 mL of CH ₂ OH.
2	Rinse column with 5 mL of EtOAc into SOLVENT WASTE.
3	Rinse column with 5 mL of DCM into SOLVENT WASTE.
4	Condition column with 10 mL of CH ₃ OH into SOLVENT WASTE.
5	Condition column with 10 mL of Water into AQUEOUS WASTE.
6	Pause and alert operator, resume when Continue is pressed.
7	Load 550 mL of sample onto column.
8	Dry column with gas for 10 min.
9	End.

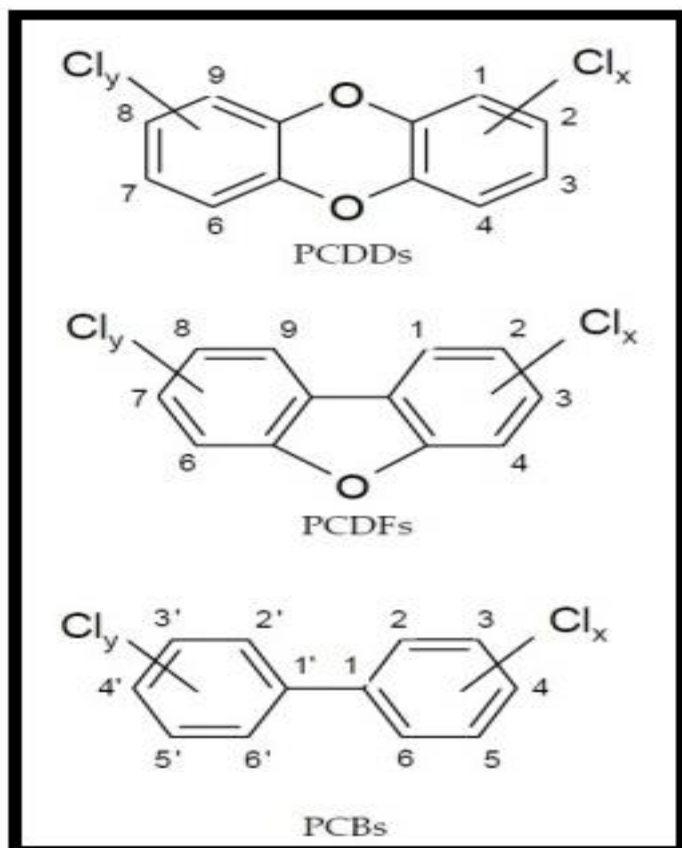
Application Brief 876



Benzo[a]pyrene

Compound	Mean	Std. Dev.
SS:2-Fluorobiphenyl	67.7	8.4
SS:Terphenyl-d14	90.1	4.9
SS:2,4,6-Tribromophenol	104.1	4
Acenaphthylene	50	8.4
Acenaphthene	45.8	7.7
Anthracene	70.6	6.4
Benzo[a]anthracene	79.1	6.8
Benzo[b]fluoranthene	77.8	7.2
Benzo[k]fluoranthene	79.4	5.8
Benzo[g,h,i]perylene	75.5	8.4
Benzo[a]pyrene	79.1	7
Chrysene	93	5.3
Dibenza[a,h]anthracene	74.7	6.4
Fluoranthene	67.4	5
Fluorene	52.4	6.6
Indeno[1,2,3-cd]pyrene	76.8	7.5
Naphthalene	49.5	13
Phenanthrene	66.9	5.7
Pyrene	79.4	7.7
Diethylphthalate	90.3	3

Dioxins, Furans, and PCBs in Surface Water



METHOD 8270C

SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

U.S. EPA Method 8270

No.	Method: Estimated time 1hr 30 min
1	Process six samples using the following method steps:
2	Condition column with 5.0 mL of CH_3OH into solvent waste.
3	Condition column with 5.0 mL of water into aqueous waste.
4	Load 500.0 mL of sample into column.
5	Rinse column with 3.0 mL of CH_3OH /water 40:60 into solvent waste.
6	Wash syringe with 5.0 mL of 70%EtOH/toluene.
7	Dry column with gas for 10 min.
8	Collect 5.0 mL fraction into sample tube using 70%EtOH/toluene.
9	Wash syringe with 5.0 mL of CH_3OH .
10	End.

Application Brief 805

Semivolatile Organic Compounds in Drinking Water

Application Note: 819

- Automated SPE
- EPA Method 525.2
- Semivolatiles
- Drinking Water
- AutoTrace 280
- GC
- GC/MS

EPA Method 525.2: Extraction of Semivolatile Organic Compounds from Water Using AutoTrace 280 Solid-Phase Extraction Cartridges

Jennifer Peterson, David Knowles, Richard Carlson and Bruce Richter

Introduction

Traditionally, semivolatiles in drinking water have been extracted using liquid-liquid extraction techniques such as separatory funnel or continuous liquid-liquid extraction. While these techniques are effective, they use large volumes of organic solvent (up to 300 mL per sample) and require extensive user intervention or monitoring. Solid-phase extraction (SPE) is an alternative accepted extraction technique for USEPA Method 525.2 which uses significantly less solvent (up to 60 mL per sample). The Thermo Scientific Dionex AutoTrace 280 extraction instrument offers an automated SPE technique that saves solvent and time, and decreases user intervention thanks to automation.

Equipment

Dionex AutoTrace 280 Automated Large Volume SPE instrument for 6 mL Cartridges (Thermo Scientific PN 071385)
15 mL Conical Tubes (pack of 12) (Thermo Scientific PN 071056)

Required Solvents

Ethyl acetate (EtOAc)
Methylene chloride (CH_2Cl_2)
Methanol (MeOH)
Water

Standards

- Automated SPE with Thermo Scientific GC and GC/MS Systems
- Combined extraction method for 80 semivolatile compounds in one run
- Recoveries meet or exceed the requirements of U.S. EPA Method 525.2

Compound	Std. Conc.	Result	% Recovery	% RSD
Isophorone	1.000	0.977	97.7	2.21
1,3-Dimethyl-2-nitrobenzene	5.000	5.107	102.1	0.73
Naphthalene	1.000	0.995	99.5	3.36
Dichlorvos	1.000	1.058	105.8	4.81
Hexachlorocyclopentadiene	1.000	0.846	84.6	3.81
EPTC	1.000	1.023	102.3	3.25
Dimethylphthalate	1.000	1.000	100.0	2.95
Acenaphthylene	1.000	0.884	88.4	0.87
Choroneb	1.000	1.028	102.8	4.78
Acenaphthene	1.000	0.975	97.5	2.59
Molinate	1.000	1.037	103.7	1.96
Diethylphthalate	1.000	1.037	103.7	1.24
Propachlor	1.000	0.991	99.1	2.81
Fluorene	1.000	0.912	91.2	3.93
Trifluralin	1.000	0.878	87.8	1.27
alpha-BHC	1.000	0.984	98.4	1.85
Simazine	1.000	0.974	97.4	6.72
Hexachlorbenzene	1.000	0.852	85.2	2.96
Atrazine	1.000	1.055	105.5	2.68
beta-BHC	1.000	1.045	104.5	2.87
Pentachlorophenol	4.000	4.145	103.6	5.51
gamma-BHC	1.000	1.001	100.1	1.74
Terbacil	1.000	1.093	109.3	2.75
Chlorothalonil	1.000	1.065	106.5	2.25
Phenanthrene	1.000	0.967	96.7	2.61
delta-BHC	1.000	1.030	103.0	4.24
Anthracene	1.000	0.891	89.1	3.21
Acetochlor	1.000	1.043	104.3	1.65
Metribuzin	1.000	0.918	91.8	2.30

Compound	Std. Conc.	Result	% Recovery	% RSD
DCPA (Dacthal)	1.000	1.031	103.1	3.67
Aldrin	1.000	0.914	91.4	1.55
Heptachlor Epoxide (isomer B)	1.000	1.044	104.4	2.29
Heptachlor Epoxide (isomer A)	1.000	1.084	108.4	3.41
Fluoranthene	1.000	1.013	101.3	3.79
Butachlor	1.000	1.126	112.6	2.35
alpha-Chlordane	1.000	0.980	98.0	1.20
gamma-Chlordane	1.000	0.983	98.3	1.61
Endosulfan I	1.000	1.113	111.3	4.07
Pyrene-d10	5.000	5.118	102.4	1.22
trans-Nonachlor	1.000	0.927	92.7	2.71
Pyrene	1.000	1.023	102.3	2.63
4,4' DDE	1.000	0.910	91.0	3.80
Dieldrin	1.000	1.064	106.4	1.43
Chlorobenzilate	1.000	1.121	112.1	2.76
Endrin	1.000	1.050	105.0	9.31
4,4' DDD	1.000	1.040	104.0	2.57
Endosulfan II	1.000	1.078	107.8	2.63
Endrin Aldehyde	1.000	1.149	114.9	2.64
Benzyl Butyl Phthalate	1.000	1.080	108.0	2.62
Di-(2-Ethylhexyl)adipate	1.000	0.852	85.2	1.20
4,4' DDT	1.000	0.709	70.9	5.44
Endosulfan sulfate	1.000	1.091	109.1	4.66
Triphenylphosphate	5.000	5.600	112.0	1.87
Methoxychlor	1.000	1.035	103.5	0.20
Endrin Keytone	1.000	1.269	126.9	2.28
Di (2-Ethylhexyl) Phthalate	1.000	0.963	96.3	1.15
Benz (a) anthracene	1.000	0.935	93.5	2.29
Chrysene	1.000	0.983	98.3	1.72

Application Note 819

Organochlorine Pesticides in Drinking Water

Application Note: 1004

Automated Solid-Phase Extraction of Organochlorine Pesticides from Drinking Water

Selvan Lingam, Bruce Richter, Richard Carlson, and Brett Murphy; Thermo Fisher Scientific Inc., Salt Lake City, UT, USA

Key Words

- Organochlorine
- Pesticides
- Extraction
- Water
- Soil

Introduction

Organochlorine pesticides (OCPs) are synthetic chemicals comprising a variety of compounds containing carbon, hydrogen, and chlorine; they are widely used around the world. These compounds can be highly toxic, and some agents—such as DDT—have been banned in the United States because of their unacceptably slow degradation. OCPs are hydrophobic, lipophilic, and extremely stable. Once in air, water, and soil, they are subject to global deposition processes and bioaccumulation in the food chain. Diet is the main source of human exposure, primarily through food consumption where OCPs have bioaccumulated.

Here, twenty chlorinated pesticides were extracted from drinking water using a Thermo Scientific Dionex AutoTrace 280 Solid Phase Extraction Instrument (AT 280). The efficiency of the Dionex AT 280® method

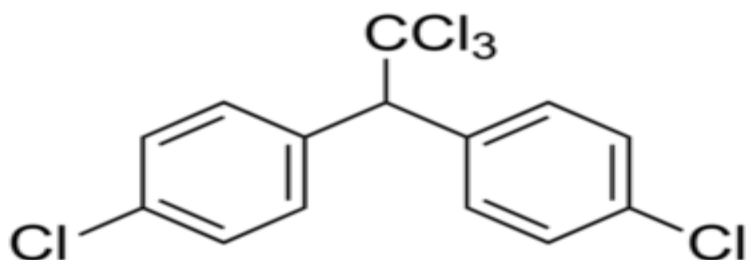
Solvents and Reagents

Dichloromethane (Thermo Fisher Scientific)
 Ethyl acetate (Thermo Fisher Scientific)
 Methanol (Thermo Fisher Scientific)
 Hydrochloric acid (Thermo Fisher Scientific)
 Sodium sulfate (Thermo Fisher Scientific)
 Screwcap amber vial (Thermo Fisher Scientific PN 033919)
 Organochlorine Pesticide Mix A/B
 Semivolatile Internal Standard Mix
 1 L sample bottle (Thermo Fisher PN 056284)
 60 mL collection vial (Thermo Fisher PN 048784)
 2 mL screw thread amber vial (Thermo Fisher PN 033919)

Procedure

Sample Preparation for Liquid-Liquid Phase Extraction

Application Note 1004

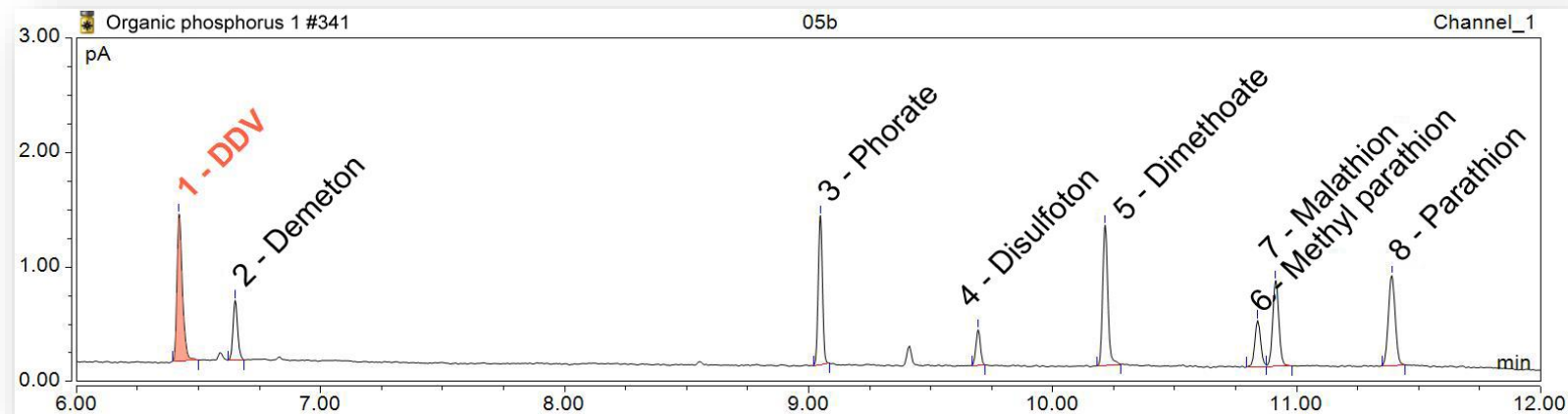


4,4'-DDT

Analyte	Spike Level (ng/mL)	SPE %RSD	LLE %RSD	% Recovery SPE vs. LLE
Alpha-BHC	50	13	13	113
Gamma-BHC	50	12	12	135
Beta-BHC	50	11	1	113
Heptachlor	50	12	13	64
Delta-BHN	50	12	12	128
Aldrin	50	13	11	134
Heptachlor epoxide	50	11	13	102
Cis-chlordane	50	12	11	138
Endosulfan I	50	11	12	99
4,4'-DDE	50	12	12	135
Dieldrin	50	12	10	107
Trans-chlordane	50	11	13	134
Endrin	50	13	11	125
4,4'-DDD	50	11	15	115
Endosulfate II	50	12	16	134
4,4'-DDT	50	11	11	135
Endrin aldehyde	50	10	11	94
Endosulfan sulfate	50	16	23	131
Methoxychlor	50	9	15	143
Endrin ketone	50	10	26	138

Organophosphorous Pesticides in Drinking Water

No.	Process six samples using the following procedure:
1	Condition cartridge with 5.0 mL methanol (MeOH) into solvent waste
2	Condition cartridge with 5.0 mL ethyl acetate (EtOAc) into solvent waste
3	Condition cartridge with 5.0 mL dichloromethane (DCM) into solvent waste
4	Condition cartridge with 5.0 mL ethyl acetate (EtOAc) into solvent waste
5	Condition cartridge with 5.0 mL MeOH into solvent waste
6	Condition cartridge with 5.0 mL water into solvent waste
7	Load 550.0mL of sample onto cartridge
8	Dry cartridge with gas for 10 min
9	Collect 4.0 mL Fraction into sample tube using EtOAc
10	Collect 4.0 mL Fraction into sample tube using EtOAc
11	Collect 4.0 mL Fraction into sample tube using DCM



Complete Workflow Solution



AutoTrace 280

+



TRACE 1310 GC

The Rocket: For Sample Evaporation

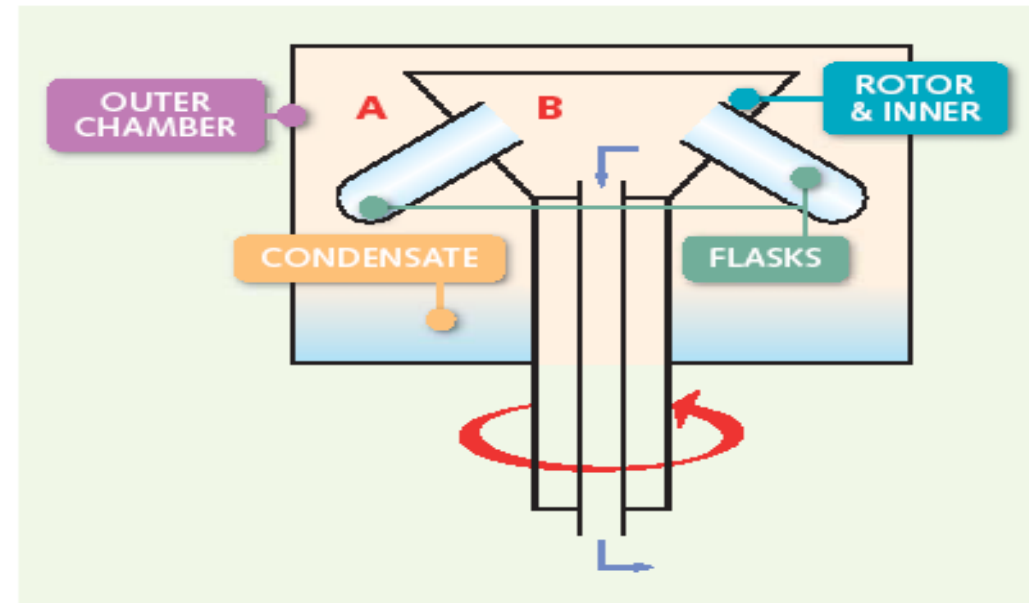
- Centrifugal evaporator that uses low temperature boiling
- Evaporates to dryness or concentrates to a fixed volume
- Allows direct transfer of 60 mL ASE vials to minimize sample handling
- Automatic end point detection
- Preprogrammed methods provide walk-away capability



- Vacuum evaporators rely on boiling the solvent at low temperature
- Low temperature boiling is achieved by using a vacuum pump to reduce the pressure in the system, and therefore the boiling point of the solvents
- When the boiling point is lower than the sample temperature, the solvent must boil!

Pressure is the Key

- Two separate pressure systems
- A – pressure to create low pressure steam, e.g. 70mbar = 40° C. Varies with control temperature
- B – pressure to boil solvents, e.g. 10mbar to boil methanol
- Vacuum pump shared between the two chambers

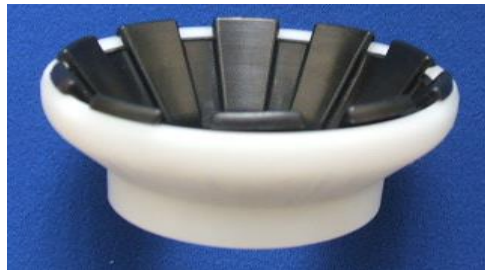


- The samples are safe from thermal damage, because:
 - Pressure controls the boiling point, and the sample temperature, while sample is wet
 - Steam is accurately temperature controlled
- “Bumping” is controlled by a patented control system called Dri-Pure®
 - Controls pressure gradient at start
 - Uses very high G-force to keep sample in flask

Flasks For Evaporating or Concentrating



- 450 mL Evaporation Flask
- Used for evaporating to dryness



- 400 mL flask that concentrates sample into GC autosampler vials
- Vial is insulated so that only solvent in flask evaporates



60 mL ASE Vials for Direct Transfer

- Pucks accept 60 mL ASE vials
- Each puck accepts 3 vials
 - Total capacity per system is 18 vials
- Allows direct sample transfer from the ASE 350 system



60 mL ASE Vial Flip Flop

- GC Vial is inserted into the flip flop funnel
- GC vial is insulated to prevent boiling of solvent
- ASE vial is inserted directly into the Rotor
- GC vial is placed directly into autosampler once complete



How Fast Does it Go?

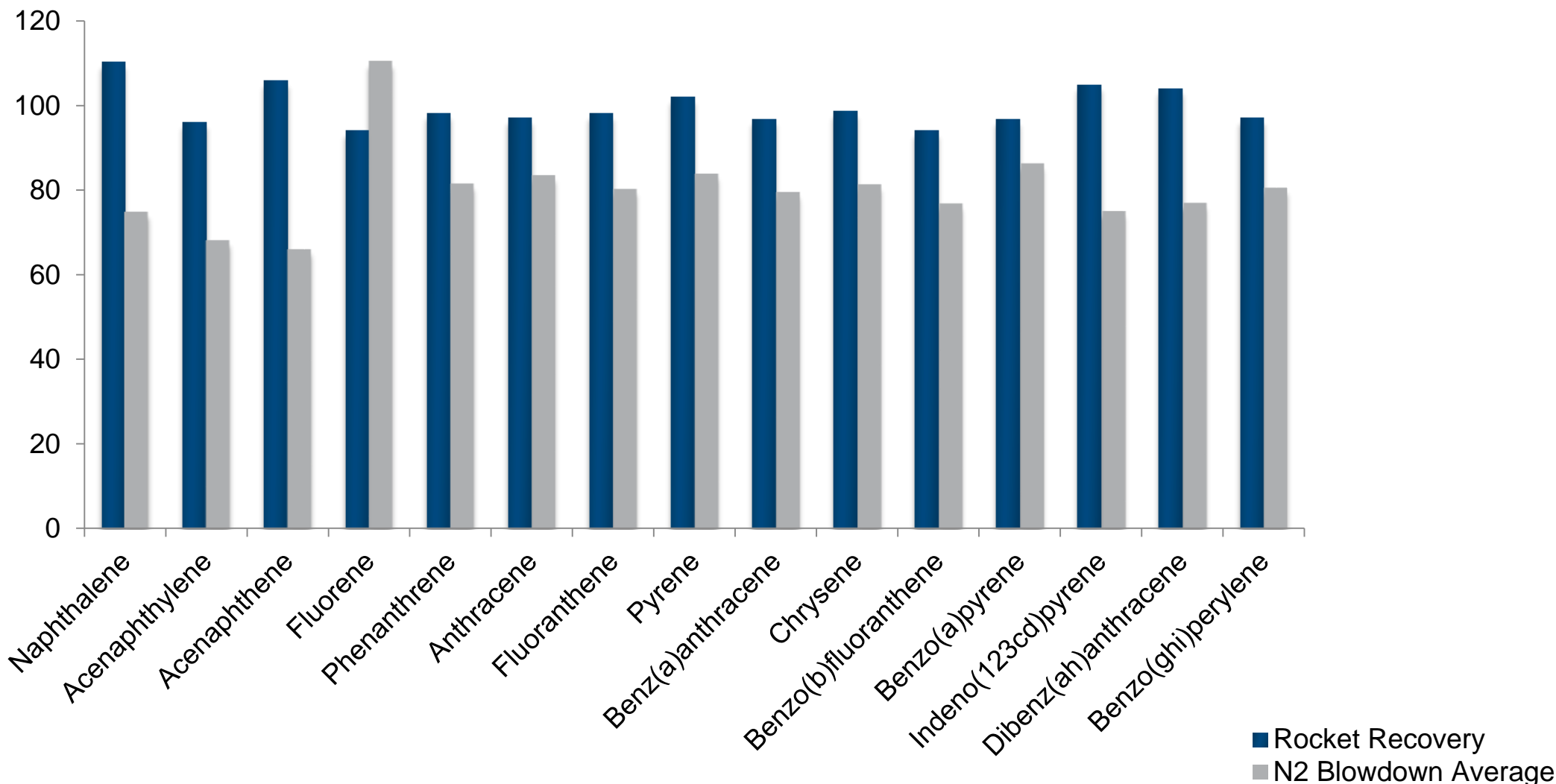
	100 mL	250 mL	450 mL
DCM	10 min	20 min	35 min
Methanol	20 min	45 min	1.5 h
DMF	30 min	1 h	2 h
Water	35 min	1.5 h	2 h
Water/ACN (1:1)	1 h	1.5 h	3 h

Times are given for complete dryness for 6 flasks simultaneously evaporated at 40 °C

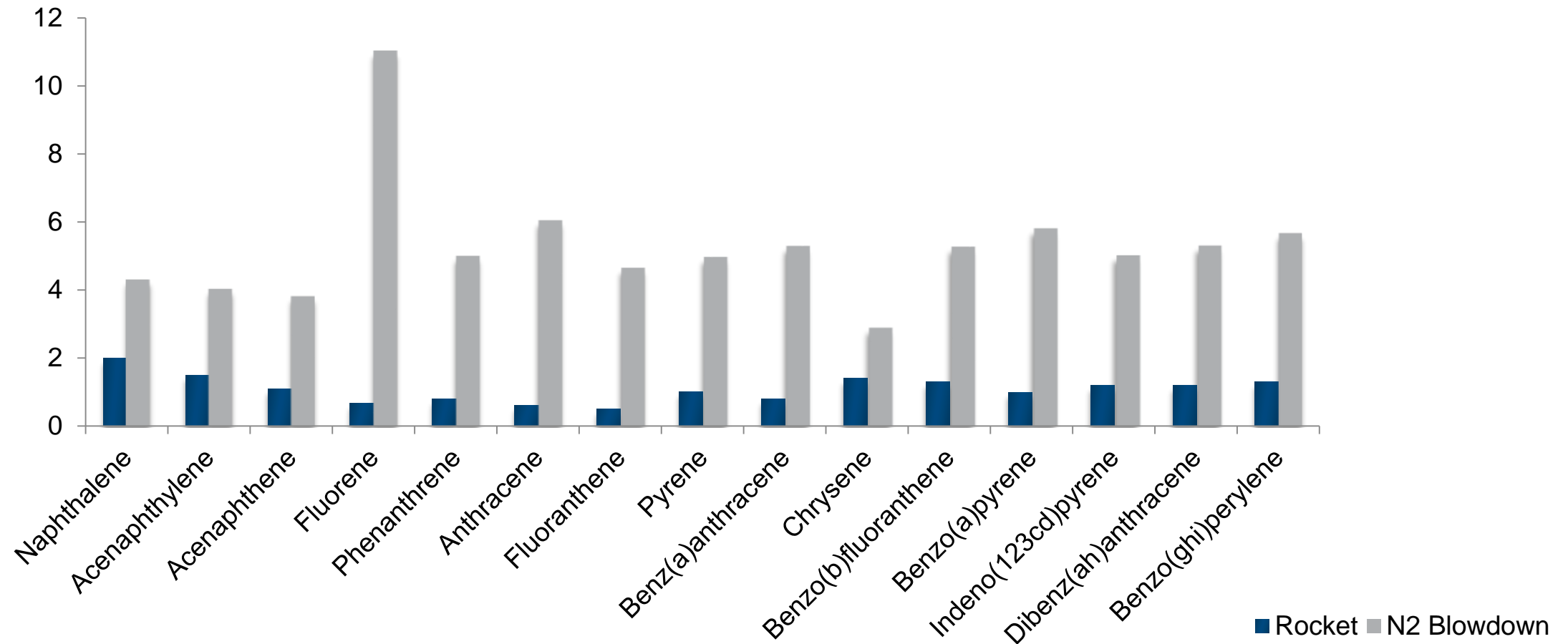
What About Solvent Recovery?

Solvent	Recovery
DCM	80%
DMF	99%
Ethanol	99%
Methanol	98%
Water	99%
Water/acetonitrile	98%

PAH Recoveries: Rocket v. Nitrogen Blow down



PAH Recoveries: Standard Deviations

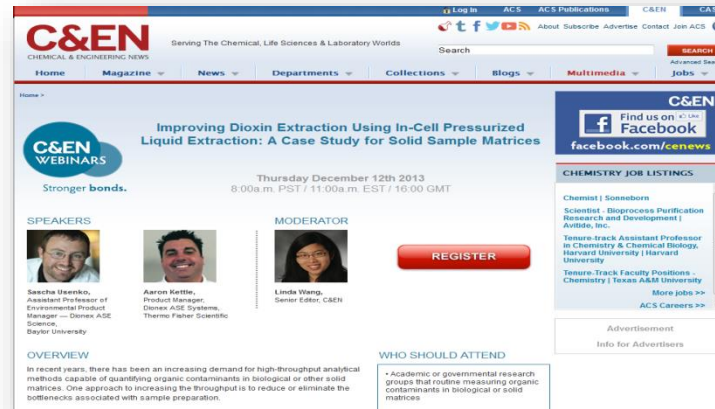


Where Can I Find Out More?

GC Farms Customer Testimonial: Accelerated Solvent Extraction and Ion Chromatography



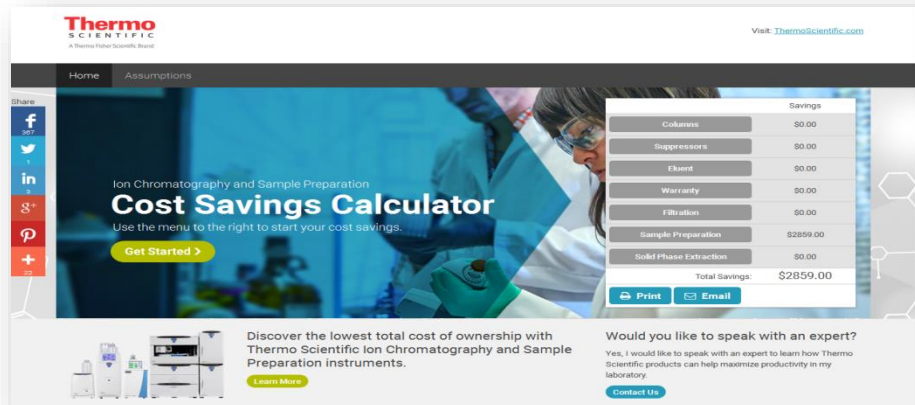
Customer Testimonials



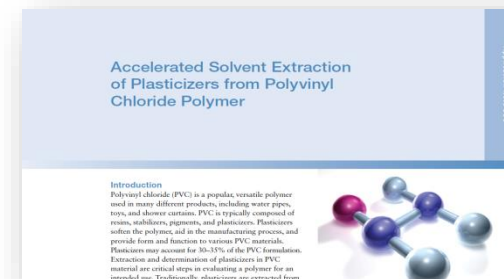
Educational Webinars



Three Year Warranty



Cost Savings Calculator

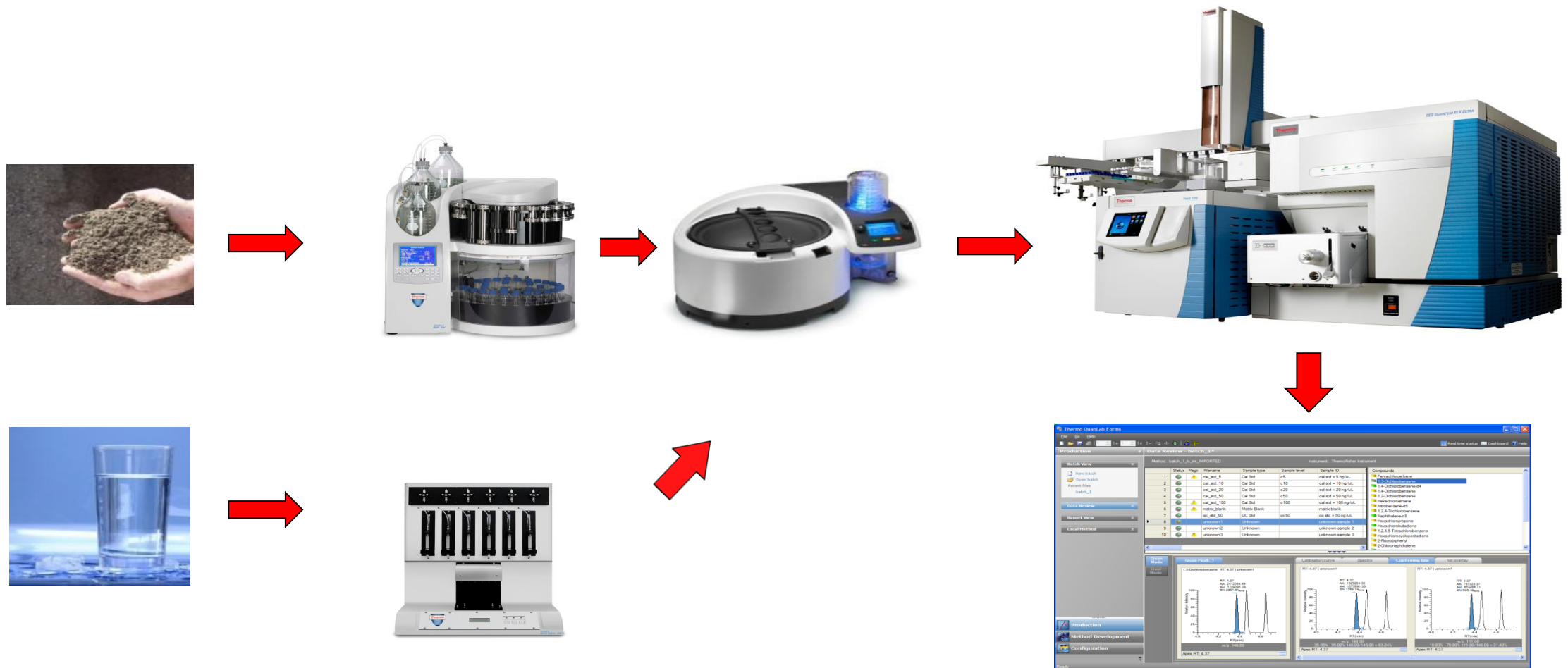


Application Notes & Brochures

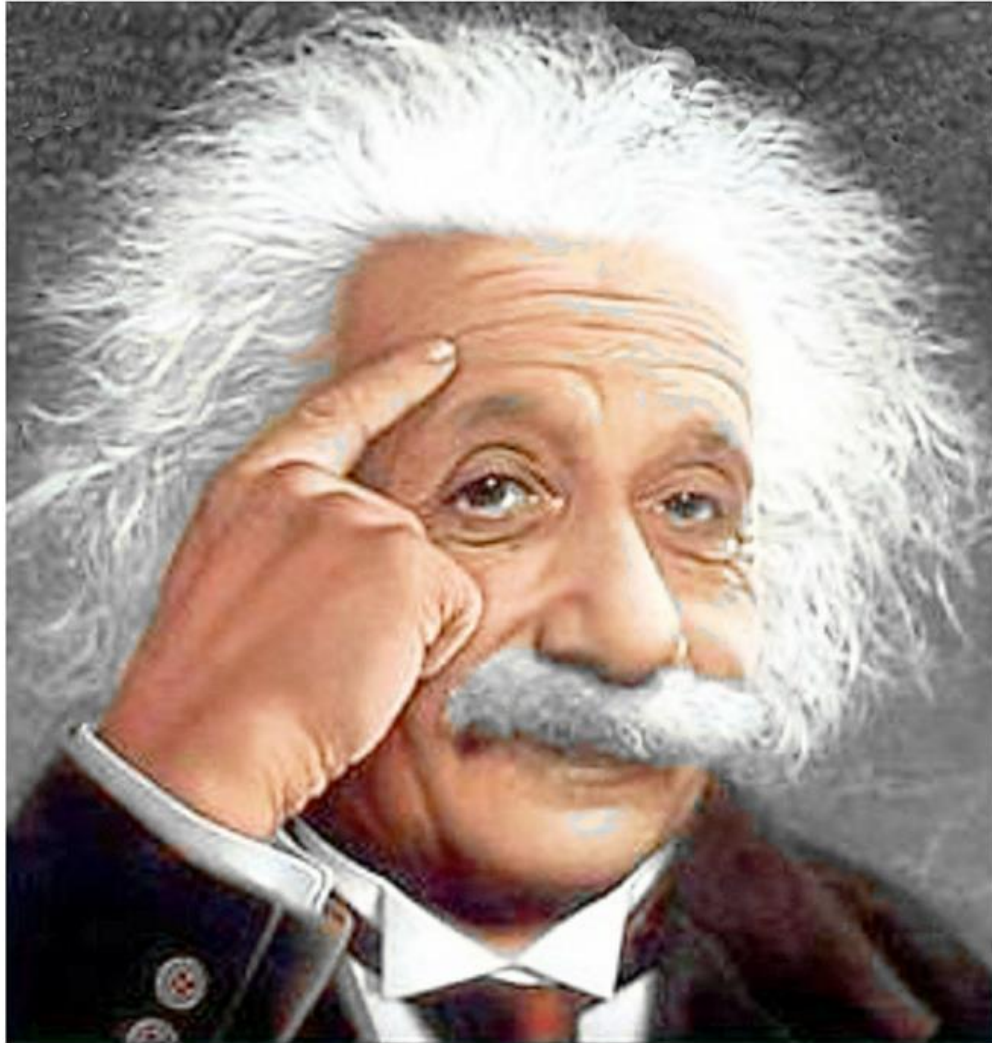


<http://www.thermofisher.com/us/en/home/industrial/chromatography/chromatography-sample-preparation.html>

Total Workflow with Fast Simplified Sample Prep



Integrated Workflow Solutions for Sample Preparation



Thank you!

Work Smarter Not Harder!