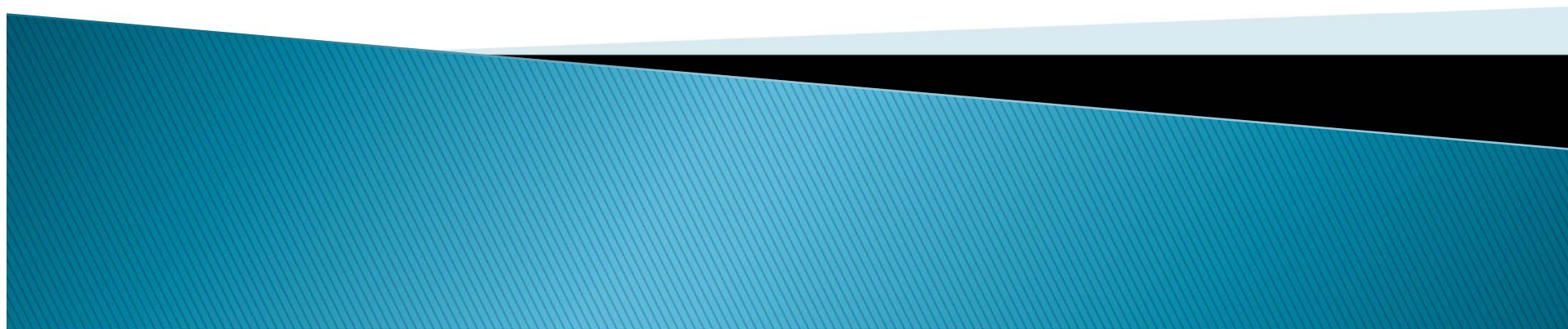


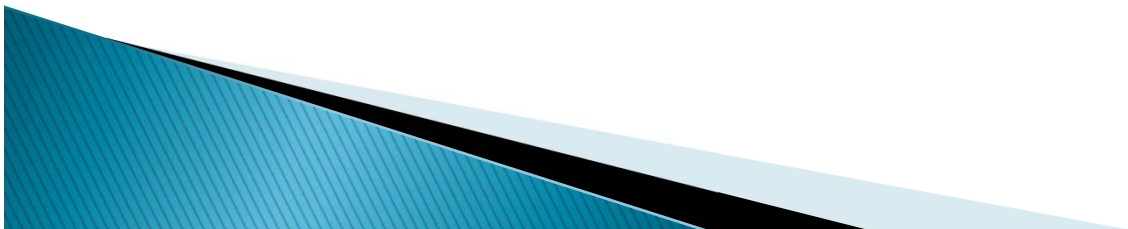
Evaluation of High Throughput, No Methylene Chloride, Low Cost Sample Clean Up for POPs Analysis

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Watertown MA



Introduction

- POPs (PCDD/Fs, PCBs) continue to attract interest around the world due to strict regulations enforced in many countries
- Rapid and quality sample clean up and analysis is needed for many laboratories processing samples
- Processing times and cost are important considerations



Challenges of POPs Sample Prep

- Labor intensive, prone to error
- Compliance with regulatory procedures and accreditation (lengthy method validation)
- Strict QA/QC requirements
- Sample matrix complexity
- Native background and interferences (can be orders of magnitude higher than analytes)
- Pico/femto-gram analyses require ultra pure extract and excellent instrument sensitivity



Automated System

Advantages of Automated Sample Prep

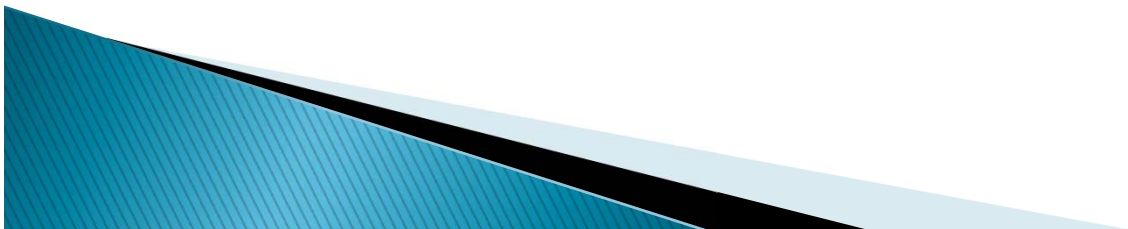
- Rapid Turn Around Time: 30 Minutes for 6 Samples
- Cleaner Background Interferences: Closed Loop System
- Quality Results: Certified Prepacked Columns
- Green Technology: No DCM, Low Solvent, as low as 90mL
- QA/QC & Accreditation Requirements: Easier to Manage
- Computerized Method: Instrumentation based prep



Manual Sample Prep

- ▶ **Advantages of Manual Sample Prep**

- ▶ Most labs use Manual Methods for the following reasons:
 - No electronics or mechanical components to fail
 - No down time due to the system failure
 - No service contract
 - No capital equipment cost



Challenges of Manual Prep

- Labor intensive, prone to error
- Compliance with regulatory procedures and accreditation (lengthy method validation)
- Strict QA/QC requirements
- Sample matrix complexity, lack of certified column to use
- Open columns, native background and interferences (can be orders of magnitude higher than analytes)
- Pico/femto-gram analyses require ultra pure extract and excellent instrument sensitivity



Automated vs Manual

Task	Manual	Automated EP-110	Ideal System Semi-automatic
Labor intensive	Days	30-45 min	30-45 min
Accreditation/validation	Slow	Fast	Fast
QA/QC	Varies	High recoveries	High recoveries
Matrix	Depends	Many	Many
Background	High	Low	Low
Trace analysis	Interferences possible	Good	Good
Instrument to run & Maintain	No	Yes	No
Failure & Downtime	No	Some times	No



Automated System Attributes

- Fully automated: From sample loading to elution and fraction collection
- Closed loop system: Cleaner background, lower detection limit
- High recoveries & Excellent precision: Certified columns
- Green Technology: No DCM, Low solvents, only Hexane and Toluene.
- Fast: Total Clean Up time 30-45 min.
- Low volumes: 100-250 mLs.



Semi-Automated System

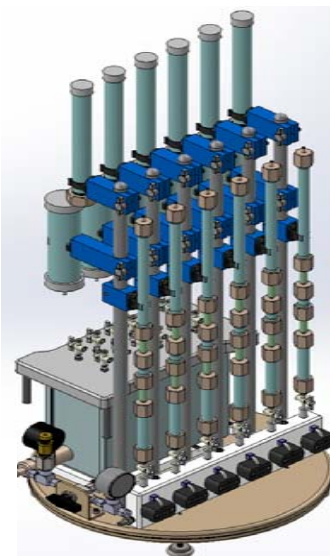
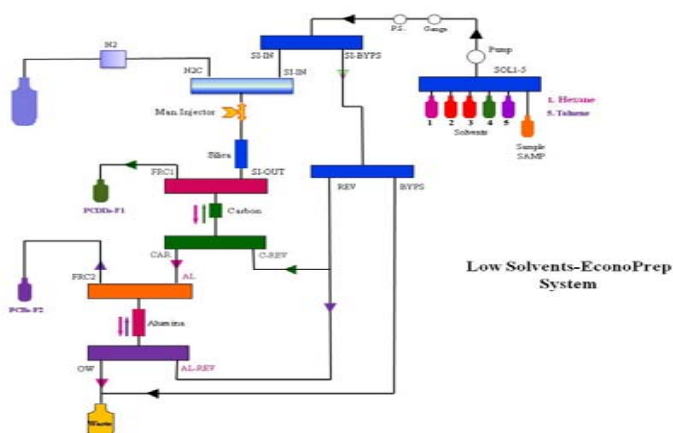
- Simple to run, no computerized instrumentation
- Fast: 30 ~ 40 min
- Closed loop system to give a clean background, low level detection
- Use certified columns
- Green technology, no DCM
- Low solvents, as low as 90mL
- Economical column kits, choice of low fat, and high fat column kits
- No capital equipment cost
- No electronics or mechanical equipment to fail
- No downtime



Automated vs Semi-Automated

Micro Computer Control

- Place the columns
- Load samples
 - **Start the system & walk away**
- Collect fractions 30 minutes later



Human Control

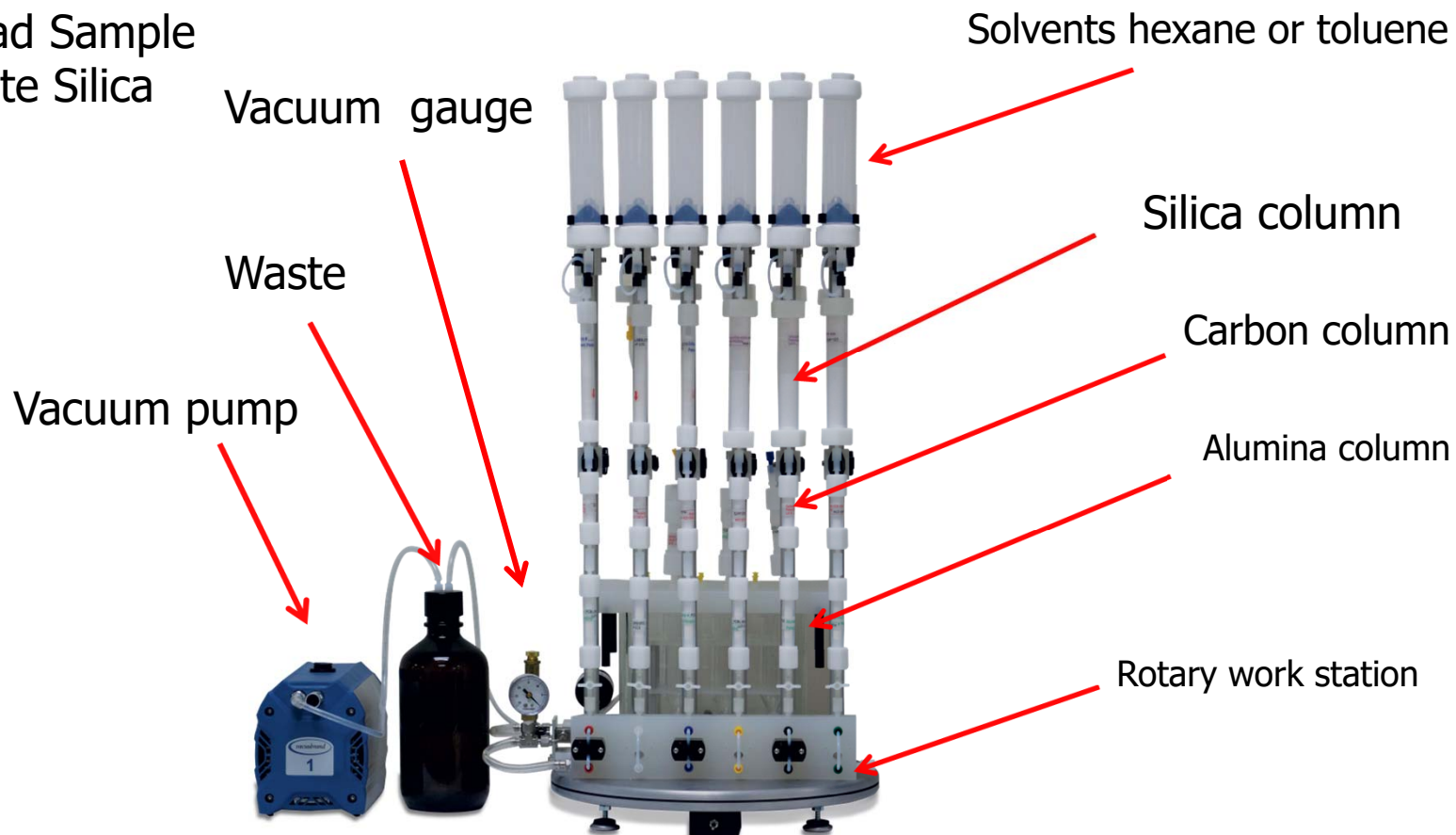
- Place the columns
- Fill Solvent reservoirs
 - **Start 3 step clean up**
- Load samples 5 Min
- Elute silica 20 Min
- Elute Dioxins & PCBs 5 Min

Characteristics of Semi-Automated System

Stage 1

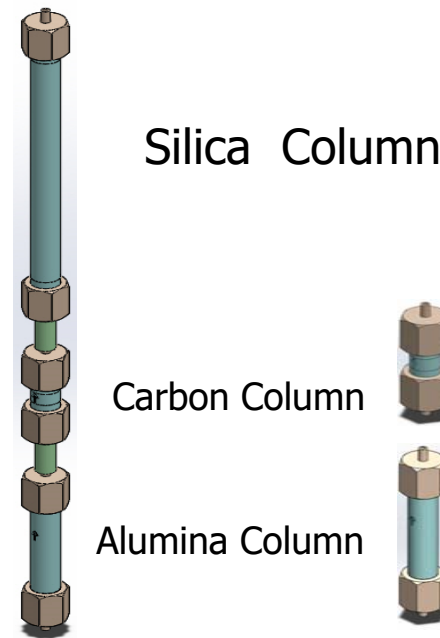
Step 1: Load Sample

Step 2: Elute Silica



Stage 1 to Stage 2

- Remove the Columns
- Remove Easy Connect fittings
- Disassemble the Columns
- Keep the Carbon & Alumina
- Rotate the work station to stage 2
- Place Carbon & Alumina on Stage 2 Manifold

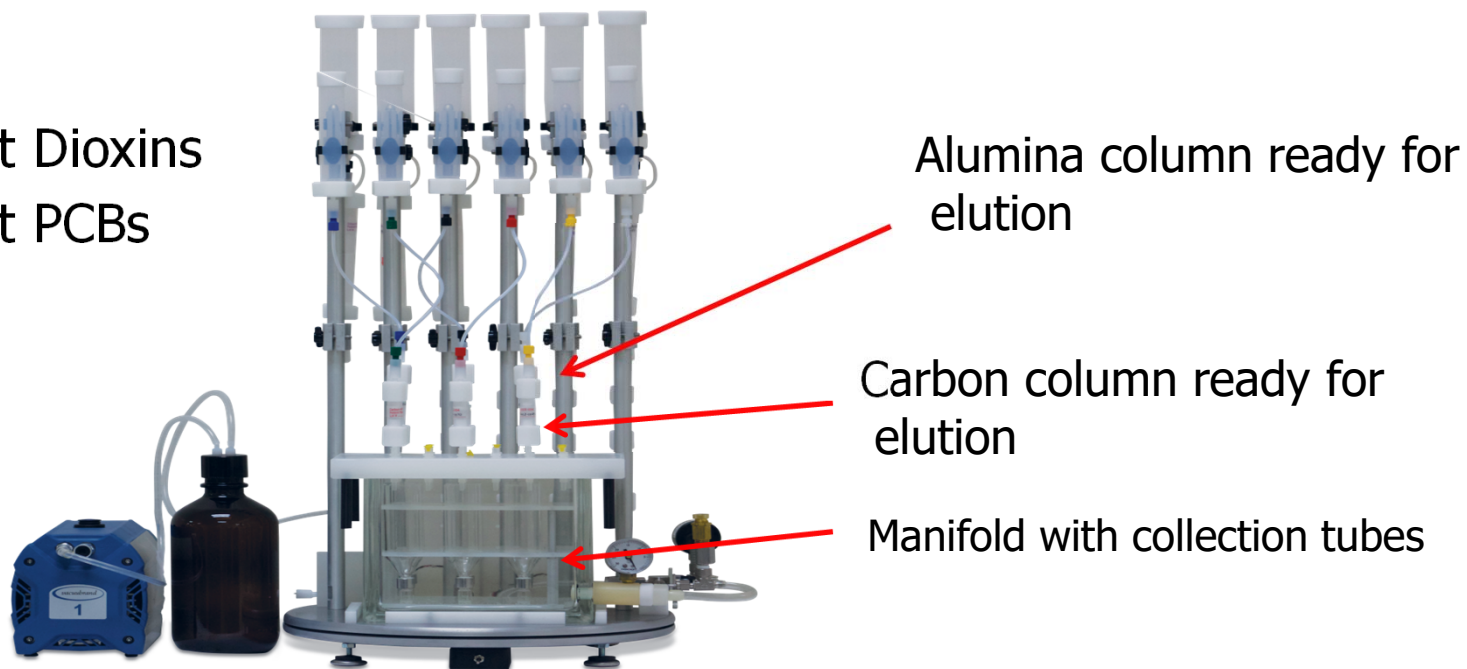


Stage 2: Collection of Fractions

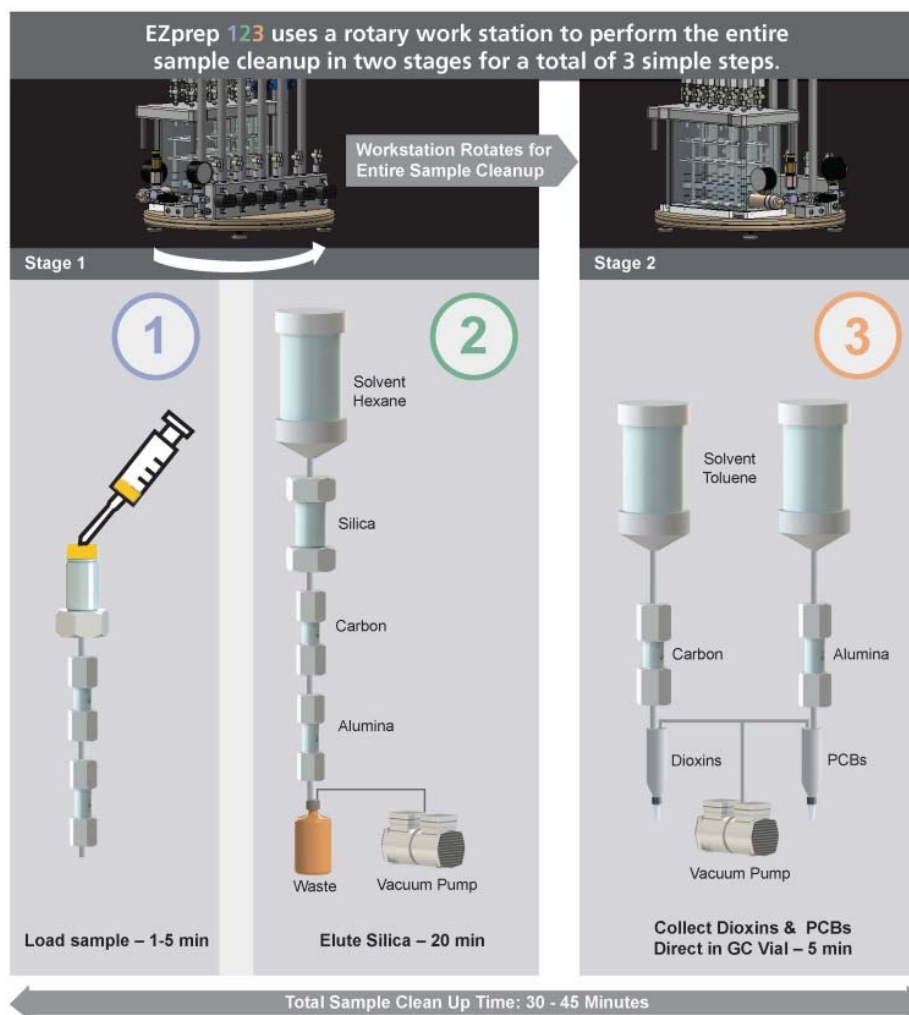
Stage 2

Step 3:

- ▶ Collect Dioxins
- ▶ Collect PCBs



Plumbing and Steps



Attributes (1)

- Closed loop system:
 - Eliminates background contaminants
 - No washing needed.
 - Capped solvent reservoirs
- Optimized for solvent reduction while obtaining highest possible recoveries
- Dichloromethane eliminated from clean up
- Ideal for food applications with PCDD/Fs and co-planary PCBs in one fraction and mono- and di-ortho PCBs in other fraction



Attributes (2)

- Easy sample loading on top of silica column via injection or sample vial (step 1)
 - Connect to carbon and alumina columns with Quick Connect connections
- Elute silica columns with hexane and transfer all target compounds to carbon and alumina
 - Hexane goes to waste (step 2)
- Rotate turntable (step 3)
 - Install carbon and alumina columns
 - Elute carbon and alumina with toluene simultaneously
 - Carbon Fraction 1 with PCDD/F and co-PCBs and alumina
 - Fraction 2 with mono- and di-ortho PCBs
 - Collection step ~ 1 min



Column Kits with various fat removal capacities

		Stage 1 (volumes in mLs)				Stage 2 (volume in mLs)			
						PCBs/PBDEs	PCDD/Fs		Total
	Fat Removal	Hexane	Hexane	Hexane		Toluene	Toluene		time
Column kits	Capacity	Conditioning	sample loading	Elute silica		Reverse alumina	Reverse alumina		(min)
Mini kit	0.15 g	20 mL	10 mL	80 mL		40 mL	40 mL		30
Classical	0.5 g	20 mL	10 mL	90 mL		40 mL	40 mL		30
Classical Plus	1.0 g	20 mL	30 mL	100 mL		40 mL	40 mL		40
High Capacity	2.5 g	40 mL	30 mL	180 mL		40 mL	40 mL		60
Extra high Capacity	5.0 g	80 mL	30 mL	220 mL		40 mL	40 mL		75



13C PCBs Recoveries EZPrep

	Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
	5 g	10 g	18 g	2 g	2 g	
PCB 28	93	104	72	103	100	95
PCB 52	90	108	70	100	97	95
PCB 77	90	103	120	98	102	108
PCB 81	92	99	62	102	98	92
PCB 101	93	110	73	106	102	98
PCB 105	108	101	62	110	104	106
PCB 114	111	102	65	105	97	104
PCB 118	86	103	60	91	89	102
PCB 123	106	97	70	92	96	93
PCB 126	107	102	90	102	98	115
PCB 138	104	96	77	92	110	111
PCB 153	101	102	69	102	114	102
PCB 156	102	99	60	113	104	105
PCB 157	93	97	61	103	99	108
PCB 167	119	106	60	105	105	107
PCB 169	98	98	81	96	96	117
PCB 170	103	107	80	103	105	117
PCB 180	98	106	85	102	102	107
PCB 189	108	97	63	95	88	107



13C PCDD/F Recoveries EZPrep

	Soil	Feed	Egg yolk	Olive oil	Fish oil	Hexane
	5 g	10 g	18 g	2 g	2 g	
2378-TCDF	95	92	83	96	89	89
2378-TCDD	104	101	70	101	96	101
12378-PeCDF	86	92	85	97	78	80
23478-PeCDF	102	94	69	102	98	91
12378-PeCDD	85	93	75	60	71	100
123478-HxCDF	88	105	79	92	92	95
123678-HxCDF	103	109	80	102	94	99
234678-HxCDF	73	66	80	60	95	104
123789-HxCDF	107	92	92	95	89	95
123478-HxCDD	107	95	79	95	92	95
123789-HxCDD	82	84	87	81	86	91
1234678-HpCDF	76	82	82	83	87	87
1234789-HpCDF	91	84	93	84	81	84
1234678-HpCDD	76	80	87	82	79	74
OCDD	60	67	60	60	91	70



Comparison Native Data with Automated System

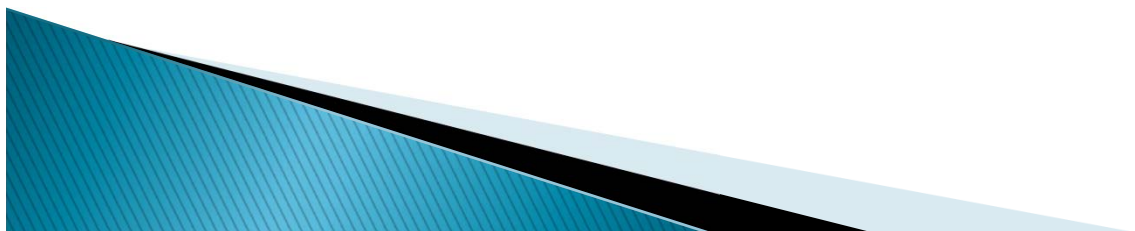
2 g Fish Oil pg/g

	EP-110	EZPrep
PCB 28	6398	6324
PCB 52	9549	10150
PCB 118	7566	7542
PCB 138	17816	19270
PCB 156	657	616
PCB 157	208	227
PCB 167	540	501
PCB 170	2013	1994
PCB 189	64	50



EZPrep Environmental

- ▶ Have all PCBs in one fraction, PCDD/Fs in other.
- ▶ Does use DCM.
- ▶ Load samples in hexane or toluene onto silica and alumina.
- ▶ Elute columns with hexane and toluene (or 10% DCM/hexane) and collect all PCBs in one fraction.
- ▶ PCDD/Fs still on alumina, add carbon column below.
- ▶ Elute PCDD/Fs off alumina onto carbon with DCM and elute carbon in reverse with toluene.



13C Recoveries with EZPrep Environmental

	10 g soil			10 g soil
2378-TCDF	101		PCB-28	80
2378-TCDD	118		PCB-52	74
12378-PeCDF	91		PCB-101	80
23478-PeCDF	91		PCB-81	71
12378-PeCDD	98		PCB-77	82
123478-HxCDF	85		PCB-123	62
123678-HxCDF	78		PCB-118	67
234678-HxCDF	81		PCB-114	54
123789-HxCDF	94		PCB-105	68
123478-HxCDD	91		PCB-126	67
123678-HxCDD	81		PCB-153	99
1234678-HpCDF	74		PCB-138	83
1234789-HpCDF	89		PCB-167	78
1234678-HpCDD	82		PCB-156	88
OCDD	123		PCB-157	77
			PCB-169	100
			PCB-180	87
			PCB-170	89
			PCB-189	93



13C PBDEs Recoveries in Sediment with EZPrep Environmental

	Sediment
	1g
BDE-28	67
BDE-47	71
BDE-99	81
BDE-100	80
BDE-153	79
BDE-154	77
BDE-183	80
BDE-209	60



Conclusions (1)

- EZprep is low solvent and fast clean up system (30 – 45 min)
 - 180 mL for low fat (< 0.15 g fat, mini kit)
 - 360 mL for high fat (up to 5 g fat, HC kit)
- High sample throughput → 18 samples/hour
 - 6 samples in parallel per station
 - 3 stations fit in one hood
- System gives excellent recoveries for PCDD/F, PCB and PBDEs comparable to FMS' automated systems
- Use of certified pre-packaged columns guarantees low native background



Conclusions (2)

- No DCM used in clean up
- No worries about breakdown or downtime
- No washing needed
- No cross-contamination
- Low cost
- EZPrep Environmental gives all PCBs and all PCDD/Fs in separate fractions.

