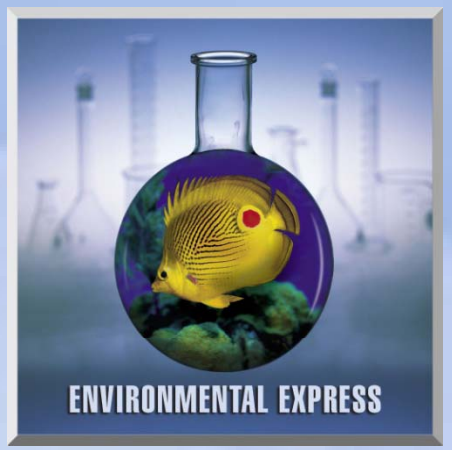


Soil Extraction Cell: An Alternative Method of Soil Extraction for Organics

Joe Boyd

Environmental Express

Charleston, SC

Various Extraction Techniques

- Sonication by SW846 Method 3550C
- Soxhlet by SW846 Method 3540C
- Accelerated Solvent Extraction by SW846 Method 3545A
- Microwave Extraction by SW846 Method 3546

Advantages to Method 3546

- Significant decrease in solvent consumption
- Multiple extractions at one time
- Short extraction time
- Closed environment

Method 3546 Specifics

- Written for the microwave
- Uses elevated temperature and pressure to achieve analyte recoveries
- Applicable to semi-volatile compounds including organophosphorous pesticides, organochlorine pesticides, chlorinated herbicides, phenoxyacid herbicides, substituted phenols, PCBs, and PCDDs/PCDFs

Recommended Extraction Conditions

- Temperature: 100 – 115°C
- Pressure: 50 – 150 psi
- Time at Temperature: 10 – 20 minutes
- Cooling: To room temperature
- Filtering / Rinsing: With same solvent system

Equipment Specifications

- Section 11.9.1 of Method 3546 states “In general, the pressure is not a critical parameter, since it is a result of the solvent system vapor pressure at the elevated temperature.”

- Section 6.1.1 requires that the system “be capable of sensing the temperature to within +/- 2.5°C and automatically adjusting the …. output power within 2 sec of sensing. Temperature sensors should be accurate to +/- 2°C.”

- The HotBlock™ meets the requirements of Section 6.1.1

Performance Based Measurement System (PBMS) Approach

Goal of PBMS is to break down barriers

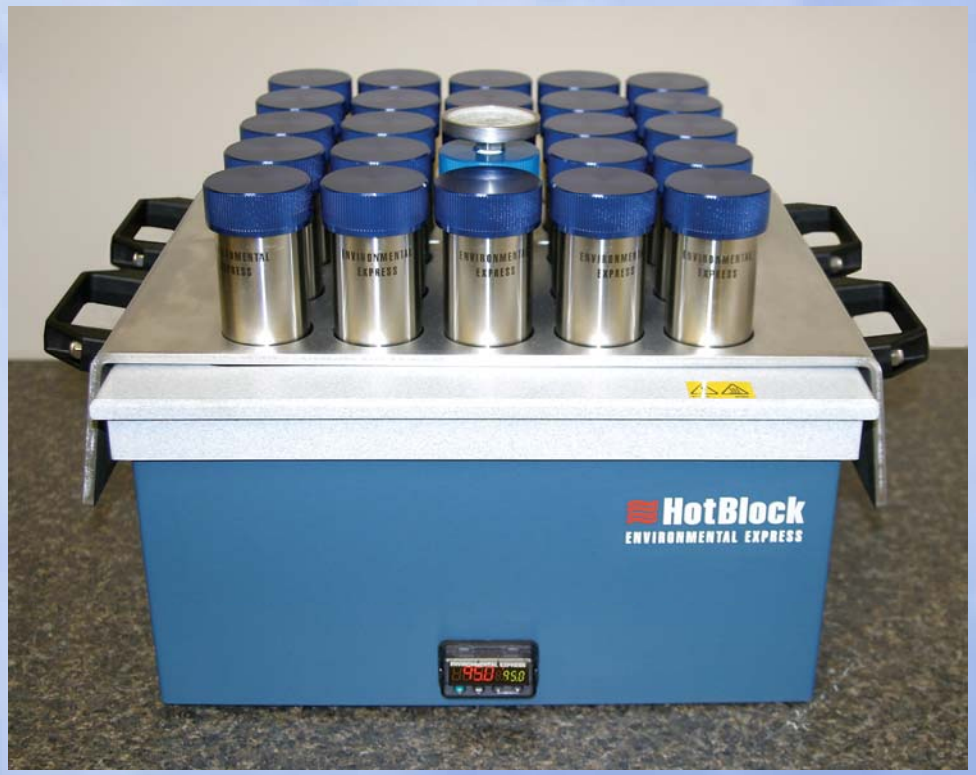
- Conveys “what” needs to be accomplished, but not prescriptively “how” to do it
- Allows for flexibility in method selection
- Expedites approval of new and emerging technologies to meet mandated monitoring requirements
- Develops and uses cost-effective methods that meet program requirements and their associated performance criteria

Method 3546 Statements

- Section 1.1 states “Other systems and other types of vessels may be used, provided that the analyst demonstrates appropriate performance for the specific application.”

- Section 1.5 states “This method has been validated using a solvent mixture of hexane and acetone (1:1) …. This solvent system or other solvent systems may be employed, provided that adequate performance is demonstrated for the analytes of interest.”

Soil-XCell System

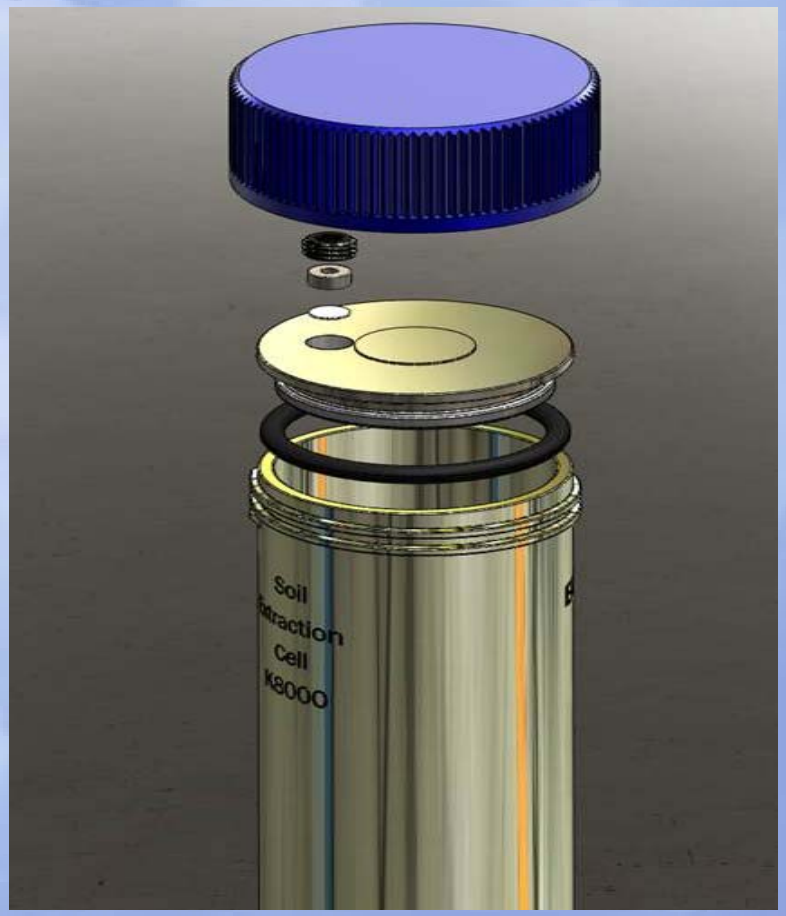


Soil Extraction Cell Procedure

- Weigh 10 – 30g of sample into the Soil Extraction Cell.
- Add approximately 30 mls of solvent.
- Add any surrogates or spikes required by the determinative method.
- Hand-tighten the outer cap onto cell.
- Heat HotBlock to 130°C. This will yield an internal solvent temperature of 100 - 115°C.
- Heat cells in HotBlock for 10 – 20 minutes once the temperature has stabilized.
- Remove cells and allow to cool to room temperature.
- Filter and rinse with same solvent system.

Soil-XCell and Method 3546

- The Soil-XCell System creates the same condition inside the soil extraction cells as the microwave creates in it's vessels
- Soil-XCell methods validated using Methylene Chloride as solvent system



Temperature and Pressure Studies

- Temperature and pressure studies were performed using 30 g of soil and 30 mls of Methylene Chloride
- Solvent temp = 110°C
- Temp at top of cell = 79°C
- Pressure calculated to 91 psig using Antoine's Coefficient

Results of a CRM for PAHs

- Wibby Lot # 7037-13
- 14 replicates analyzed using the Soil-XCell System for prep and Method 8270 for the analysis
- All 47 compounds present in the CRM were detected within the certified acceptance range
- 9 additional compounds that were not in the CRM were analyzed for and resulted in non-detects
- Data available at www.envexp.com

Results of a CRM for TPH

- Wibby Lot # 6038-10
- Certified value of 914 mg/kg
- 15 replicates analyzed using the Soil-XCell System
- Average result of 698 mg/kg with all 15 replicates falling within the acceptance range of the standard

TPH CRM data

Replicate	Result	Certified Value	% Recovery	Acceptance Range
Blank	ND	N/A	N/A	N/A
Rep 1	690 mg/kg	914 mg/kg	75.5%	236 – 1650 mg/kg
Rep 2	698 mg/kg	914 mg/kg	76.4%	236 – 1650 mg/kg
Rep 3	680 mg/kg	914 mg/kg	74.4%	236 – 1650 mg/kg
Rep 4	663 mg/kg	914 mg/kg	72.5%	236 – 1650 mg/kg
Rep 5	695 mg/kg	914 mg/kg	76.0%	236 – 1650 mg/kg
Rep 6	644 mg/kg	914 mg/kg	70.5%	236 – 1650 mg/kg
Rep 7	605 mg/kg	914 mg/kg	66.2%	236 – 1650 mg/kg
Rep 8	662mg/kg	914 mg/kg	72.4%	236 – 1650 mg/kg

TPH CRM data

Replicate	Result	Certified Value	% Recovery	Acceptance Range
Rep 9	747 mg/kg	914 mg/kg	81.7%	236 – 1650 mg/kg
Rep 10	662mg/kg	914 mg/kg	72.4%	236 – 1650 mg/kg
Rep 11	658 mg/kg	914 mg/kg	72.0%	236 – 1650 mg/kg
Rep 12	718 mg/kg	914 mg/kg	78.6%	236 – 1650 mg/kg
Rep 13	750 mg/kg	914 mg/kg	82.1%	236 – 1650 mg/kg
Rep 14	804 mg/kg	914 mg/kg	88.0%	236 – 1650 mg/kg
Rep 15	796 mg/kg	914 mg/kg	87.1%	236 – 1650 mg/kg
Average	**698 mg/kg**	**914 mg/kg**	**76.4%**	**236 – 1650 mg/kg**

Results of Real-World Samples for PAHs Compared to Method 3545A

- “Real-World” samples analyzed side-by-side using the Dionex™ ASE™ System and Soil-XCell System
- Samples extracted using Soil-XCell method with an internal solvent temperature of 110°C
- Methylene Chloride used as the solvent system

Results of Real-World Samples for PAHs Compared to Method 3545A

Compound	ASE Replicate	Soil-XCell Rep 1	Soil-XCell Rep 2	Soil-XCell Rep 3
2-Fluorobiphenyl	66 % Recovery	70 % Recovery	66 % Recovery	71 % Recovery
Nitrobenzene-d5	66 % Recovery	63 % Recovery	68 % Recovery	64 % Recovery
Terphenyl –d14	39 % Recovery	66 % Recovery	66 % Recovery	66 % Recovery
2-Methylnaphthalene	< 260 mg/kg	< 260 mg/kg	900 mg/kg	710 mg/kg
Acenaphthene	840 mg/kg	951 mg/kg	3000 mg/kg	3100 mg/kg
Acenaphthylene	< 260 mg/kg	< 260 mg/kg	330 mg/kg	470 mg/kg
Anthracene	2600 mg/kg	2700 mg/kg	6900 mg/kg	9400 mg/kg
Benz(a)anthracene	5200 mg/kg	5600 mg/kg	19000 mg/kg	20000 mg/kg
Benzo(a)pyrene	4600 mg/kg	4800 mg/kg	16270 mg/kg	16000 mg/kg
Benzo(b)fluoranthene	5700 mg/kg	6800 mg/kg	22000 mg/kg	21000 mg/kg
Benzo(ghi)perylene	3200 mg/kg	3400 mg/kg	9100 mg/kg	8800 mg/kg
Benzo(k)fluoranthene	2100 mg/kg	2100 mg/kg	5800 mg/kg	5400 mg/kg

Results of Real-World Samples for PAHs Compared to Method 3545A

Compound	ASE Replicate	Soil-XCell Rep 1	Soil-XCell Rep 2	Soil-XCell Rep 3
Chrysene	5100 mg/kg	5200 mg/kg	17000 mg/kg	17000 mg/kg
Dibenz(a,h)anthracene	860 mg/kg	730 mg/kg	2800 mg/kg	2400 mg/kg
Fluoranthene	18000 mg/kg	14000 mg/kg	28000 mg/kg	27000 mg/kg
Fluorene	1200 mg/kg	1200 mg/kg	3300 mg/kg	3800 mg/kg
Indeno(1,2,3-cd)pyrene	2800 mg/kg	2700 mg/kg	8100 mg/kg	8000 mg/kg
Naphthalene	430 mg/kg	570 mg/kg	2800 mg/kg	2000 mg/kg
Phenanthrene	10000 mg/kg	9200 mg/kg	24000 mg/kg	24000 mg/kg
Pyrene	14000 mg/kg	11000 mg/kg	23000 mg/kg	24000 mg/kg

- More data available at www.envexp.com

Results of Real-World Samples for PCBs Compared to Method 3545A

- “Real-World” samples analyzed side-by-side using the Dionex ASE System and Soil-XCell System
- Samples extracted using Soil-XCell method with an internal solvent temperature of 110°C
- Methylene Chloride used as the solvent system

Results of Real-World Samples for PCB's Compared to Method 3545A

Compound	ASE Replicate	Soil-XCell Rep 1	Soil-XCell Rep 2	Soil-XCell Rep 3
DCBP	60% Recovery	104% Recovery	132% Recovery	68% Recovery
TCMX	55% Recovery	56% Recovery	55% Recovery	58% Recovery
PCB - 1016	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB - 1221	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB - 1232	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB - 1242	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB – 1248	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB – 1254	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB – 1260	2500 mg/kg	1900 mg/kg	2000 mg/kg	1300 mg/kg
PCB – 1262	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg
PCB - 1268	<440 mg/kg	<440 mg/kg	<440 mg/kg	<440 mg/kg

- More data available at www.envexp.com

Soil-XCell vs. Microwave

- Monetary investment is much less for the Soil-XCell
- Stainless Steel cells last longer than Teflon vessels
- Solvent systems used in the microwave systems must have a percentage (>10%) of a polar component as pure hydrocarbon solvents do not absorb microwave energy
- Soil-XCell System utilizes an external heat source so any solvent system may be employed creating more flexibility

Conclusions

- Soil-XCell System follows SW846 Method 3546
- Utilizes the PBMS approach to method flexibility
- Creates exact same conditions in soil cells for extraction as the microwave does in it’s vessels
- Is consistent with Method 3446 which allows for other types of heating devices and vessels to be used
- Shows acceptable recoveries for CRM materials
- Achieves similar analytical results for real-world samples when compared to Method 3545A

Questions?

ENVIRONMENTAL EXPRESS, INC.

1-800-343-5319

info@envexp.com

www.envexp.com