

Supercritical Fluid Application Notes

SCF 502

The Extraction of PCBs from River Sediment

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Introduction

Supercritical CO₂ alone easily dissolves polychlorinated biphenyls (PCBs) from spiked matrices, but not from weathered, aged samples. The addition of a small amount of co-solvent is necessary to displace the PCBs from the surface of a weathered sample for complete extraction.



The addition of a fixed quantity of methanol to standard reference material, SRM 1939 prior to supercritical CO₂ extraction shows increased recoveries of PCBs when compared to US EPA liquid/solid extraction techniques (EPA procedure 3550).

Equipment

- ✓ Applied Separations' *Spe-ed* SFE Supercritical Extraction System

Materials

- ✓ SRM 1939, Standard Reference Material (PCBs in River Sediment)
- ✓ SFE grade CO₂
- ✓ C18 Collection cartridge (1g/6mL #12007)
- ✓ Pesticide grade methanol

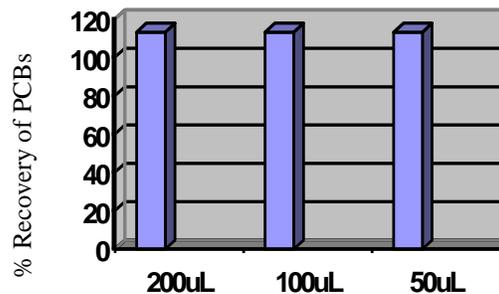
Method

Place 0.5g of soil/sediment into a 1mL extraction vessel and add 100µL of methanol. Extract sample at specified conditions.

Extraction Conditions

Extraction vessel:	1mL
Sample:	0.5 g
Pressure:	7000 psi
Temperature:	80°C
CO ₂ Flow Rate:	2L/min (gas)
Collection:	SPE cartridge 1g/6mL C18
Static:	10 minutes
Dynamic:	30 minutes
SPE Rinse:	5mL methanol spiked with 5µL of I.S. tetrachloroethylene at concentration of 50mg/mL

Results



Amount of Methanol spiked into extraction vessel.
Average % PCBs: 113%

Analysis

GC-ECD

Conclusion

A fixed quantity of co-solvent addition to a sediment sample prior to extraction gave



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excellent results with 50 μ L to 200 μ L of methanol are added. Only small quantities of methanol are required to displace the PCB analytes from the sediment matrix and do not desorb the analyte from the solid phase extraction cartridge used to trap the analyte.

References

Ashraf-Khorassani, Taylor. American Laboratory. 12/95