

Supercritical Fluid Application Notes

**SCF
509**

Extraction of PAHs from Urban Dust Using Supercritical Fluids

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Introduction

Supercritical CO₂ alone easily dissolves polyaromatic hydrocarbons (PAHs) from spiked matrices, but not from weathered, aged samples. The addition of a small amount of derivatization reagent rapidly displaces the PAHs from the surface of a weathered sample for complete extraction.

The addition of a fixed quantity of Tri-sil to standard reference material, SRM 1649 prior to supercritical CO₂ extraction shows increased recoveries of PAHs when compared to US EPA liquid/solid extraction techniques.

Equipment

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

Materials

- ✓ SRM 1649, Standard Reference Material (PAHs in Urban Dust)
- ✓ SFE grade CO₂
- ✓ C18 Collection cartridge (1g/6mL #12007)
- ✓ Tri-sil (2:1, HMDS:TMCS) Pierce

Method

Place 1.0g of soil/sediment into a 5mL extraction vessel and add 1mL of tri-sil. Extract sample at specified conditions.

Extraction Conditions

Extraction vessel:	5mL
Sample:	1.0 g
Pressure:	5000 psi
Temperature:	60°C
CO ₂ Flow Rate:	2L/min (gas)
Collection:	SPE cartridge 1g/6mL C18 or 5mL Toluene
Static:	10 minutes
Dynamic:	30 minutes
SPE Rinse:	5mL methanol spiked with 5µL of I.S. tetrachloroethylene at concentration of 50mg/mL

Results

Extraction of PAHs from SRM 1649 by SFE (ug/g)

	Certified Value	SFE + Tri-sil
Phenanthrene	4.5	9.5
Fluoranthene	7.1	6.7
Pyrene	6.5	6.9
Chrysene	3.6	5.8

Analysis

GC-FID

Conclusion

A fixed quantity of a derivatizing reagent added to an urban dust sample prior to extraction gave excellent results with 1mL of Tri-sil added. Only small quantities of Tri-sil are required to displace the PAH analytes from the sediment matrix.



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