

Supercritical Fluid Application Notes

**SCF
522**

Extraction of Irganox 1076 from Polystyrene

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Introduction

Since the presence and amount of additives may affect a polymer's performance, it is necessary to monitor additive concentrations for QC. Traditionally, additives are extracted from the polymer by methods that are labor intensive and rely on large volumes of solvent, such as methanol.



SFE is an alternative technique using supercritical carbon dioxide to extract additives

from polymers. It reduces the use, exposure to, and disposal of hazardous solvents, while providing comparable extraction results in less time. This application describes the extraction of Irganox 1076 from polystyrene using supercritical fluid.

Equipment

- ✓ Applied Separations' *Spe-ed*TM SFE or Helix Supercritical Extraction System
- ✓ SFE Modifier Pump
- ✓ Polymer Grinder – Cyro Grinder

Materials

- ✓ Methanol
- ✓ Methylene Chloride
- ✓ Carbon dioxide – welding or industrial grade with cleanup column
- ✓ C18 SPE Cartridge – 500mg/6mL, Applied Separations (Cat. #12006)
- ✓ *Spe-ed* Glass Wool (Cat. #7953)
- ✓ Ottawa Sand (Cat. #10548)

Method

Grind 1.0 gram of polymer sample under liquid nitrogen and sieve. Mix sample with 15 grams of sand. Place a plug of glass wool into an extraction vessel and pour the prepared sample into the vessel using a funnel, then place a plug of glass wool on top. Compress the sample with a tamping rod, and then seal the vessel. Install the vessel into the *Spe-ed* SFE. Place a 6mL C18 SPE cartridge on the discharge fitting, and extract sample according to the specified extraction conditions. Remove SPE cartridge and elute with 5mLs of methanol/methylene chloride (1:1).

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Extraction Conditions

Extraction vessel:	24 mL
Sample:	1.0 g
Pressure:	7000 psi
Temperature:	125°C
Valve temperature*:	130°C
CO ₂ composition	CO ₂ : Methanol (85:15)
CO ₂ Flow Rate:	2 L/min (gas)
Collection:	C18 SPE cartridge
Static time:	15 minutes
Dynamic time:	30 minutes
SPE Elution:	5 mLs methylene chloride/methanol (1:1)

*Adjust valve temperature to maintain a minimum of 50°C in SPE collection tube. This will prevent modifier condensation in the trap.

Analyte Recovery

Elute SPE column with 5 mLs elution solvent and analyze by HPLC.

Conclusion

The supercritical carbon dioxide extraction of Irganox 1076 offers a viable alternative to solvent-based procedures. The accuracy and precision of the results were comparable to standard methods, such as Soxhlet, while extraction times were reduced. In addition, the use of hazardous solvents was significantly reduced.

References

Ashraf-Khorassani, M.; Boyer, D.; and Levy, J.M.
“Quantitative Extraction of Polymer Additives from Different Polymers using On-line SFE/SFC.” J. Chromatog. Sci., **29**, 517 (1991).

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