

Supercritical Fluid Application Notes

**SCF
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Introduction

It is necessary to monitor and analyze additive packages in polyolefin resins for QC in many industries. Traditionally, additives are extracted from the polymer by methods that are labor intensive and rely on large volumes of solvent, such as toluene.



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 antioxidant from high density polyethylene using supercritical fluid.

Extraction of Antioxidant from High Density Polyethylene



Equipment

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

Materials

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

Method

Grind 1.5 gram of polymer sample under liquid nitrogen and sieve. Next, mix sample with 15 grams of Ottawa 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 6 mL C18 SPE cartridge on the discharge fitting, and extract sample according to the specified extraction conditions. Remove SPE cartridge and elute with 5 mLs of methanol/methylene chloride (1:1).

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Extraction Conditions

Extraction vessel:	24 mL
Sample:	1.5 g
Pressure:	7000 psi
Temperature:	140°C
Valve temperature:	140°C
CO ₂ Flow Rate:	4 L/min (gas)
Collection:	C18 cartridge
Dynamic time:	30 minutes
SPE Elution:	5 mLs methanol/ methylenechloride (1:1)
Dynamic time:	30 minutes
SPE Elution:	5 mLs methylene chloride/methanol (1:1)

Analyte Recovery

Elute SPE cartridge with 5 mLs of methanol/methylene chloride (1:1) and analyze via HPLC.

Conclusion

The supercritical carbon dioxide extraction of additives from polymers offers a viable alternative to solvent-based procedures. The accuracy and precision of the results were comparable to the standard method while extraction times were reduced. In addition, the use of hazardous solvents was significantly reduced.

References

Ashraf-Khorassani, M.; Boyer, D.; and Levy, J. "Optimization of Experimental Parameters for the Determination of Polymer Additives using On-Line SFE-SFC." *Journal of Chromatographic Science*. 29 (1991) 517-521.

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