

Supercritical Fluid Application Notes

**SCF
524**

Determination of Pesticide Residue in Nonfatty Foods by SFE and GC/MS

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Introduction

Pesticides have been widely used by the farming industry to yield greater crops with less labor. The use of organochlorine pesticides are now regulated in most countries, however, due to their toxicity and highly persistent nature laboratories still test soil and food samples for pesticide residue. In particular, agricultural products like vegetables and fruits, which are most likely to be contaminated, are routinely tested.

Traditionally, the determination of pesticides in nonfatty foods such as apples, beans, and carrots is accomplished by solvent extraction. This method is labor intensive and requires a significant quantity of toxic organic solvent.

SFE is an alternative technique using supercritical carbon dioxide to extract pesticides quickly and easily in the laboratory. It eliminates the use, exposure to, and disposal of hazardous solvents, while providing comparable extraction results to standard methods in less time.



The AOAC Method 2003.03 has been used to extract carbofuran, diazinon, vinclozolin, chlorpyrifos, and endosulfan sulfate from apples. It has also been used to extract trifluralin, dacthal, quintozone, and chlorpyrifos in green beans, and atrazine, metalaxyl, parathion-methyl, chlorpyrifos, and bifenthrin from carrots.

Equipment

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

Materials

- ✓ Carbon dioxide – SFE grade
- ✓ C18, SPE Cartridge, 500 mg/6 mL Applied Separations (Cat. #12006)
- ✓ *Spe-ed* MatrixTM (Cat. #7950)
- ✓ Filter paper disk

Method

Before preparing sample, store test samples, containers, and *Spe-ed* MatrixTM in a freezer. When ready for extraction, place 1.0 g of homogenized frozen sample (either apples, green beans, or carrots) onto 1.1 g of cold *Spe-ed* MatrixTM and mix with a mortar and pestle or blender. Place a filter paper disk into an extraction vessel and pour the prepared sample into the vessel. Fill the void volume with *Spe-ed* Matrix, and then seal the vessel. Install the vessel into the *Spe-ed* SFE. Place a C18 SPE cartridge on the discharge tube. Extract sample according to the specified extraction conditions.



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Extraction Conditions

Extraction vessel:	10mL
Sample:	1.0g
Pressure:	5000 PSI
Temperature:	60°C
Valve temperature:	120°C
CO ₂ Flow Rate:	2 L/min (gas)
Collection:	C18 SPE cartridge
Dynamic time:	30 minutes
SPE Elution:	8 mLs acetone

Results

After extraction, reduce extract volume to 1.5 mLs. Analyze GC/MS (see AOAC Method 2002.03).

Conclusion

The supercritical carbon dioxide extraction of pesticides offers a viable alternative to solvent-based procedures. The accuracy and precision of the results were comparable to traditional methods while extraction times were reduced. In addition, the use of hazardous solvents was eliminated.

References

AOAC Method 2002.03

Lehotay, Steven J. "Determination of pesticide residues in nonfatty foods by SFE and GC/MS: Collaborative study." *Journal of AOAC International*, 2002, Vol. 85, No. 5.

Lehotay, Steven J. "Collaborative study to determine pesticide residues in nonfatty foods by SFE and GC/MS." 1999.