

Supercritical Fluid Application Notes

SCF
520

Extraction of Drugs and other Chemical Residues from Tissues using SPE Trapping Techniques

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Introduction

Recovery of trace level (ppm-ppb) residues such as pharmaceuticals from tissue is generally performed using a variety of analytical techniques that include liquid-liquid, solid phase extraction, and immuno-affinity chromatography. There are many problems associated with these techniques, including low recoveries of target analytes, labor intensive procedures, difficult and costly preparation of antibodies and the use of and disposal of hazardous organic solvents.



Pharmaceutical analytes may be easily extracted from biological matrices and trapped using the SFE/SPE

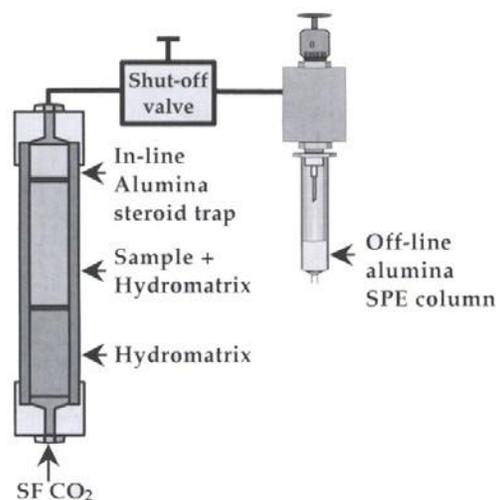
extraction technique. SFE is an alternative technique using supercritical carbon dioxide to extract trace level drug residues from an analyte/fat matrix while eliminating the use, exposure to, and disposal of hazardous solvents.

Isolation of Drug Residue from Tissue Matrices by SFE

There have been some problems identified with using typical SFE methods to isolate drug residue from tissue matrices. One of the main difficulties is that when trace levels of residues are isolated from fat tissue by SFE using CO₂, fat is co-extracted. If a modifier is used with CO₂, the resultant extract becomes more complex and the desired analyte is more difficult to recover from the mixture.

A solution to these problems is to use an SFE instrument and method that simplifies the separation and recovery of trace level drug residues from an analyte/fat matrix. This application describes a method to extract nitrosamines, sulfonamides, nitrobenzamides, anabolic steroids, and melengestrol acetate from various biological matrices without co-extracting fat from the sample.

Analyte Trapping Technique



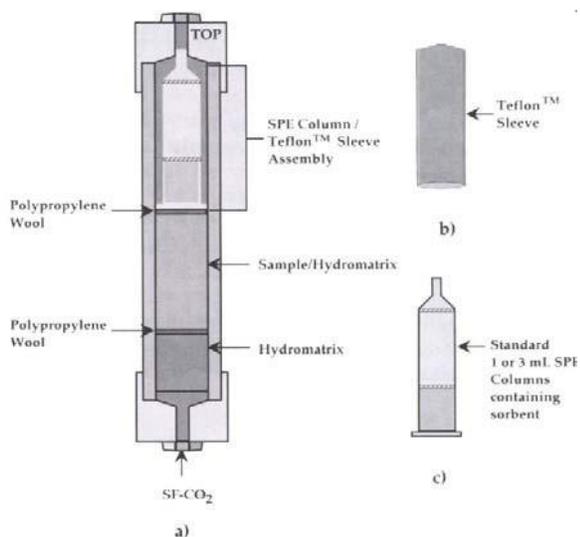
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Offline Trapping:

One Channel of the Applied Separations *Spe-ed* SFE configured with in-line sorbent trap and off-line micrometering valve interfaced to an SPE column.

Inline Trapping:

Components of the inline assembly: (a) SPE column-Teflon sleeve packed in an extraction vessel together with the sample matrix; (b) Teflon sleeve; and (c) standard SPE column with cropped flange.



Overview of SFE/SPE Applications

Analyte	Matrix	Fortification Level	% Recovery
Nitrobenzamides	Chicken Liver	1 ppm	82-96
Nitrosamines	Frankfurters	20 ppb	88-101
Sulfonamides	Chicken Tissues	1 ppm	77-89
Steroids	Chicken Liver	500 ppb	53-100
Melengestrol Acetate	Bovine Fat Tissues	25 ppb	90-124
Steroids	Urine	12 ppb	91-94
Clenbuterol	Bovine Liver	0.5 ppb	82-112
Avermectins	Bovine, ovine, and porcine liver	2 ppb	76-97
Organochlorine pesticides	Eggs	50 ppb	82-108

Conclusion

SFE/SPE extraction is an effective method for trace level analysis of drugs from biological matrices. Analyte/fat mixtures extracted by SFE/SPE are easily cleaned up for analysis. In addition, SFE/SPE methods significantly reduce solvent consumption and sample preparation time.

Equipment

- ✓ Applied Separations' *Spe-ed*TM SFE-2 or Helix Supercritical Extraction System

Materials

- ✓ *Spe-ed* Matrix (Cat. #7950)
- ✓ *Spe-ed* Wool (Cat. #7953)
- ✓ Carbon dioxide – SFE grade