Supercritical Fluid Application Notes



Extraction of Sulfonamides from Chicken Liver using Supercritical Fluoroform (CHF₃)

Back to List

Introduction

This application describes a method to extract sulfonamides from fortified chicken liver using supercritical fluoroform (CHF₃). Methanol-modified CHF₃ provides an average recovery approaching 100% for various sulfonamides



and produces a significantly cleaner extract than other supercritical solvents.



Equipment

- ✓ Applied Separations' Spe-ed[™]SFE-2 or Helix Supercritical Extraction System
- ✓ SFE Modifier Pump

Materials

- ✓ Fluoroform, SFE grade, Air Products
- ✓ Methanol
- ✓ C18 SPE cartridges, Applied
 ✓ Separations (Cat. #12007)
- ✓ Spe-ed Matrix (Cat. #7950)
- ✓ *Spe-ed* Polyproplyene Wool (Cat.#7952)

Method

Spike 1 gram of homogenized chicken liver with 10 μ L of a drug mixture (0.6g/L of SMZ, 0.5g/L of SDM and 0.5g/L of SQA). Mix spiked chicken liver with 2 grams of *Spe-ed* Matrix. Place a plug of *Spe-ed* Wool into an extraction vessel and pour the prepared sample into the vessel using a funnel, then place a plug of *Spe-ed* Wool on top. Compress the sample with a tamping rod, fill the void volume with *Spe-ed* Matrix, then seal the vessel. Install the vessel into the *Spe-ed* SFE. Place a predried and preweighed collection vial containing a plug of *Spe-ed* Wool on the discharge tube. Extract sample according to the specified extraction conditions.

Extraction Conditions

| Extraction vessel: | 10 mLs | |
|-----------------------------|--------------------|--|
| Sample: | 1 g | |
| Pressure: | 6500 psi | |
| Temperature: | 40°C | |
| Valve temperature: | 120°C | |
| CHF ₃ Flow Rate: | 2 L/min | |
| Dynamic time: | 30 minutes | |
| Modifier: | Methanol 200µL/min | |
| Collection: | C18 SPE cartridge | |
| SPE Elution: | 5 mLs of 0.5M | |
| | phosphate buffer/ | |
| | methanol (50/50) | |



930 Hamilton Street · Allentown, PA 18101 610-770-0900 · 610-740-5520 (fax) www.appliedseparations.com

Supercritical Fluid Application Notes

Analysis

| HPLC: | Waters 6000 |
|---------------|----------------------|
| Column: | C18, 5 micron, 250 x |
| | 4.6 mm, Supleco |
| Mobile Phase: | 60/40 0.5M phosphate |
| | buffer/ MeOH pH 7.1 |
| Flow Rate: | 1 mL/min |
| Detection: | 265 nm |
| | |

Results

Percent recovery of sulfamethazine (SMZ), sulfadimethoxine (SDM), and sulfaquinoxiline (SQA) from chicken liver.

| | % Recovery | RSD |
|-----|------------|-----|
| SMZ | 99.8 | 4 |
| SDM | 85.8 | 12 |
| SQA | 31.2 | 14 |

Conclusion

The supercritical fluoroform extraction of sulfonamides from chicken liver offers a viable alternative to traditional procedures that are time consuming and solvent intensive. The use of fluoroform reduced the amount of coextracted fat and simplified the post extraction cleanup. Simple off-line collection of the extracts with SPE cartridges is efficient and requires no further sample cleanup.

References

Ashraf-Khorassani, M. and Taylor, L. "Comparison of Supercritical CHF₃ and CO₂ and Methanol-modified CHF₃ and CO₂ for Extraction of Sulfonamides from Chicken Liver." *J. of AOAC*. **1996**, 22 (5), 1043-1049.



930 Hamilton Street · Allentown, PA 18101 610-770-0900 · 610-740-5520 (fax) www.appliedseparations.com