

Extraction of Furazolidone from Animal Feeds

Introduction

Furazolidone and other nitrofurant antimicrobial compounds have a history of use in the poultry industry as feed additives and also as therapeutic agents. However, nitrofurant residues in food are carcinogenic and may cause allergic reactions in sensitive individuals and therefore must be monitored to prevent health problems.

The extraction of Furazolidone from feed samples is typically accomplished with a Goldfisch continuous extraction apparatus, but the extraction time is lengthy, from 8 to 18 hours, and significant amounts of organic solvent are consumed in the process.

Significant time savings and reduction of solvent usage can be achieved by the extraction of Furazolidone and other nitrofurant antimicrobials from feed samples by using the pressurized solvent extraction technique.

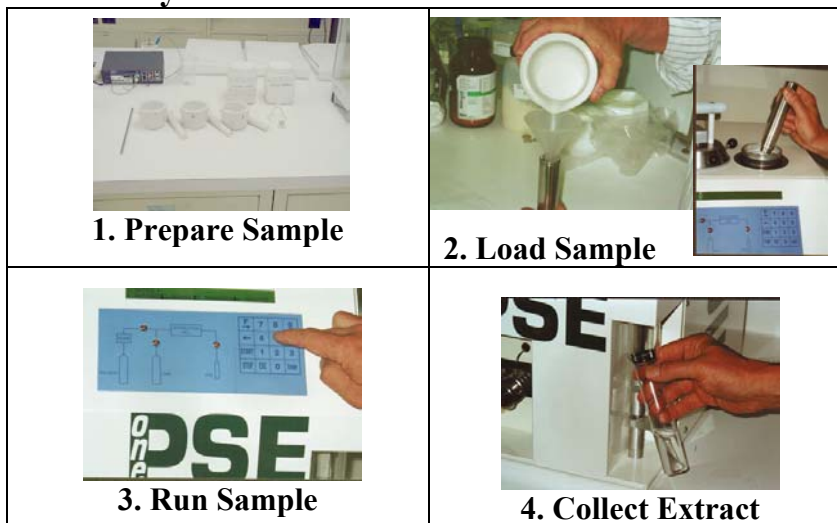
Equipment

- ✓ Applied Separations' Pressurized Solvent Extractor
 - *one* PSE or *fast* PSE
- ✓ 11mL or larger Extraction Vessel
 - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Collection Vials – 60mL for extract collection
- ✓ HPLC/UV

Solvents and Materials

- ✓ Acetone (reagent grade)
- ✓ Tetrahydrofuran (reagent grade)
- ✓ Water (reagent grade)
- ✓ Tetraethylammonium bromide solution (5% in water)
- ✓ *Spe-ed*TM Matrix (Catalog #7950)
- ✓ Cellulose disk (Catalog#10711)
- ✓ Collection vials – 60mL (Catalog#10650)
- ✓ Sand ,Ottawa standard,(Catalog#10548)

Summary of Method



Procedure

Prepare Sample

Place disposable cellulose filter into extraction vessel, on top of the vessel outlet frit.

Grind a dry sample to a size 10 mesh or finer. Weigh 5-10g of ground sample into the extraction vessel. Mix 5g of a wet sample with 5g *Spe-ed* Matrix and grind in a mortar and pestle. Place ground sample in extraction vessel and fill with sand.

Load Sample

Place the extraction vessel into the instrument as described in the *onePSE* operator's manual.

Place a pre-cleaned collection vial in the instrument for each sample. Ensure that the collection vial is sufficiently large to hold the extract.

Run Sample

Select the appropriate stored extraction program and start.

Extraction Conditions

*Program the following extraction parameters on the one PSE
Program A Mode – 11,22,or 33mL vessel*

Oven temperature:	100°C
Pressure:	100 BAR
Static time:	5 minutes
Cycles	1
Solvent:	Acetone/Water (93/7)
Purge:	N=2
Flush program:	Solvent/gas/repeat flush: 20 sec/2min/0

Optimize the conditions as needed. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the samples. Any pressure in the range of 100 BAR should suffice.

Once established, the same parameters should be used for all samples extracted for the same analysis type.

Collect Extract

Collect each extract in a clean 60mL vial. Evaporate the extract to dryness. If water is evident, add 25 ml of acetone and reevaporate, repeat process as necessary.

Add 5 ml of DMF to residue and gently heat to dissolve all the residue. Add 5 ml of 5% TEAB solution and pour into a 15 ml centrifuge tube and let cool. Centrifuge @ 2000 rpm for 5 minutes and then remove the fat layer floating on the supernate with a disposable pipet.

ANALYSIS – HPLC/UV

Detector – UV 365nm

Column - C18, 10 micron

Mobile Phase - 2% acetic acid in acetonitrile/DI water
(20/80)

Flow rate – 1.5 mls/minute

Injection volume – 20ul

References

AOAC Method 985.51

Safety

The use of organic solvents, elevated temperatures, and high pressures present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. However, the following sections describe additional steps that should be taken.

Extraction cells in the oven are hot enough to burn unprotected skin. Allow the cells to cool before removing them from the oven or use appropriate protective equipment (e.g., insulated gloves or tongs), as recommended by the manufacturer.

During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Connect this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere.