Extraction of Pyrethrin and Pyrethroid Pesticides from Fish Using the QuEChERS Approach

UCT Product Number:
EC4MSSA50CT-MP (4000 mg MgSO₄ and 1000 mg sodium acetate)
CUMPSC18CT (150 mg MgSO₄, 50 mg PSA and 50 mg endcapped C18)

January 2011

The QuEChERS approach is used for the determination of trace levels of natural pyrethrins and synthetic pyrethroids (cypermethrin & deltamethrin) in fish.

1. Extraction
   a) Weigh 10 grams of homogenized fish into a 50 mL centrifuge tube
   b) Add 500 ng cis-permethrin (phenoxy-¹³C₆) surrogate standard
   c) Add 10 mL 1% acetic acid in acetonitrile
   d) Add the contents of pouch EC4MSSA50CT-MP
   e) Shake vigorously for 1 minute then centrifuge

2. Clean-up, Dispersive Solid-phase (dSPE)
   a) Transfer 1 mL of supernatant to a 2 mL micro-centrifuge tube
      CUMPSC18CT
   b) Shake for 1 minute then centrifuge
   c) Transfer 0.5 mL of extract to a graduated tube then evaporate to near dryness
   d) Add 50 ng trans-permethrin (phenoxy-¹³C₆) and bring to exactly 0.5 mL with trimethyl phosphate (TMP)
   e) Add MgSO₄ to the 0.2 mL mark then vortex
   f) Transfer supernatant to injection vial for analysis
3. **Analysis**
   
a) Use GC/MS in CI mode

b) Column: HP-5, 30m X 0.32 mm with 0.25 μm film (or equivalent)

c) Splitless mode @ 240°

**GC Oven program:**

- Initial 80°C, hold 1 minute
- 50°C/min to 200°C
- 5°C/min to 285°C
- 50°C/min to 325°C, hold 5 minutes
- Transfer line 250°C

**MS Conditions:**

- Source 150°C
- Methane reagent gas
- Selected Ion Monitoring Mode

Calibration using matrix matching may be required

*Adapted from Roscoe, Veronica, Judge, Judy, Rawn, Dorothea F.K., “Application of the QuEChERS Extraction Method for the Analysis of Pyrethrin and Pyrethroid Pesticides in Fin and non-Fin Fish”, Health Products and Food Program, Winnipeg, Manitoba and Bureau of Chemical Safety, Food Research Division, Ottawa, Ontario, Canada, Florida Pesticide Residue Workshop, July 2009*