



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

UCT Products:

EC4MSSA50CT-MP (4000 mg MgSO₄ and 1000 mg sodium acetate)

CUMPSC18CT (150 mg MgSO₄, 50 mg PSA and 50 mg endcapped C18)

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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:

- 1) Initial 80°C, hold 1 minute
- 2) 50°C/min to 200°C
- 3) 5°C/min to 285°C
- 4) 50°C/min to 325°C, hold 5 minutes
- 5) Transfer line 250°C

MS Conditions:

- a. Source 150°C
- b. Methane reagent gas
- c. 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

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