

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



UCT Part Numbers

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

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

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

Procedure:

1. Extraction

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

2. Clean-up, Dispersive Solid-Phase (dSPE)

- Transfer 1 mL of supernatant to a 2 mL micro-centrifuge tube **CUMPSC18CT**.
- Shake for 1 minute then centrifuge.
- Transfer 0.5 mL of extract to a graduated tube then evaporate to near dryness.
- Add 50 ng trans-permethrin (phenoxy-¹³C₆) and bring to exactly 0.5 mL with trimethyl phosphate (TMP).
- Add MgSO₄ to the 0.2 mL mark then vortex.
- Transfer supernatant to injection vial for analysis.

3. Analysis

- Use GC/MS in CI mode
- Column: HP-5, 30m X 0.32 mm with 0.25 µm film (or equivalent)
- Splitless mode @ 240°

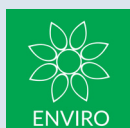
GC Oven Program

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



References:

[1] 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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