

Pesticides in Fatty Matrices Extraction



UCT Part Numbers

ECPSAC1856 500 mg endcapped C18 and 500 PSA in 6 mL cartridge	CUMPSC18CT 150 mg MgSO_4 , 50 mg PSA and 50 mg C18 in a 2 mL centrifuge tube
ECMAG00D 500 g organic free MgSO_4 anhydrous	ECNACL05K 5 kg NaCl

Procedure:

1. Sample Preparation

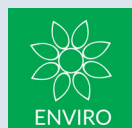
- Weigh 20.0 ± 0.10 grams (g) of homogenized sample into a 250 mL plastic centrifuge bottle, tared on a balance capable of weighing to 0.01 grams
 - Fortify each sample with process control spiking (PCS) solution
 - Add 50 mLs of ethyl acetate (EtAc) to each tube
 - Fortify each sample with internal standard (ISTD) spiking solution
 - Reduce sample material particle size by using a high speed disperser for approximately 1 minute
 - Add 2 g of anhydrous MgSO_4 (**ECMAG00D**) and 0.5 g anhydrous NaCl (**ECNACL05K**).
- Note:** Carefully add the reagents to the tube to avoid contaminating the threads or rims of the tubes otherwise leaks may result
- Seal the tube and shake vigorously for approximately 1 minute either mechanically or by hand. Make sure the solvent interacts well with the entire sample and that crystalline agglomerates are broken up
 - Cool the sample in a -20°C freezer for approximately 30 minutes
 - Centrifuge at 10,000 RCF for 5 minutes
 - Decant at least 50 mL of the EtAc layer into a 50 mL glass graduated centrifuge tube using a funnel and filter paper. Allow the extract to come to room temperature and adjust the volume with EtAc to 50 mL using a Pasteur pipette
 - Concentrate the extract under a stream of nitrogen with a 70°C water bath until the volume remains constant (this will be ~ 3 mLs and will take about 1 hour)
 - Dilute to 20 mLs with acetonitrile (MeCN) and cap with a glass stopper, vortex for 1 minute
 - Freeze at -70°C for 30 minutes
 - Centrifuge the extract while frozen for 3 minutes (The MeCN will thaw during centrifugation)
 - Directly after centrifugation in step n), filter > 15 mLs of the MeCN layer of the extract with a $0.45\ \mu\text{m}$ syringe filter into a 15 mL glass centrifuge tube
 - Allow the extract to come to room temp, adjust the volume to 15 mL, and concentrate to 2.25 mL under a stream of nitrogen with a 70°C water bath

2. LC-MS/MS Analysis

- Transfer 1 mL of extract to a 2 mL mini-centrifuge tube (**CUMPSC18CT**)
- Vortex for 1 minute and centrifuge
- Transfer to auto sampler vial. Sample is now ready for analysis

3. GC Analysis

- For GC analyses, use the dual layer cartridge (**ECPSAC1856**)
- Add approximately 0.75 – 0.80 grams ($\sim 0.6\ \text{cm} = 0.25$ inches) of anhydrous MgSO_4 added to the top of the cartridge
- Condition the SPE cartridge by adding one cartridge volume (4.0 mLs) of MeCN using a UCT positive pressure SPE manifold
- Elute to waste
- Place a labeled 15 mL graduated disposable plastic centrifuge tube below the cartridge in the positive pressure SPE manifold
- Quantitatively transfer 1 mL of the sample extract from step 15 to the SPE cartridge
- Elute SPE cartridge in a drop wise manner (Regulated Flow Pressure = 35 psi) into a labeled 15 mL graduated glass centrifuge tube using MeCN
- Collect the eluate while washing the SPE cartridge three times with 4 mLs of eluant. Do not allow the cartridge to go dry until step 24
- After the last 4 mL portion of eluant has passed through the cartridge move the switch of the positive pressure SPE manifold from "Regulated Flow" to "Full Flow/Dry" to dry the SPE cartridge for approximately 1 minute
- Using an N-Evap (or equivalent) with the water bath set at 50°C and N_2 flow set at < 10 liters per minute (LPM) (typical setting is 2 – 6 LPM), evaporate the sample to approximately 0.5 mL
- Add 3 mL of toluene to the centrifuge tube containing the sample
- Evaporate again to < 0.5 mL. (This is to insure all other solvents have been removed from the sample.)
- Bring the volume to 1.0 mL with toluene and vortex to mix solvent into sample
- Analyze by GCMS-EI and GCMS-NCI



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