



Pesticides in Fatty Matrices Extraction

January 20, 2011

UCT Products:

ECPSAC1856 (500 mg endcapped C18 and 500 PSA in 6 mL cartridge)

CUMPSC18CT (150 mg MgSO₄, 50 mg PSA and 50 mg C18 in a 2 mL centrifuge tube)

ECMAG00D (500 g organic free Mg SO₄ anhydrous)

ECNAACL05K (5 kg NaCl)

1) Sample Preparation

- a) 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
- b) Fortify each sample with process control spiking (PCS) solution
- c) Add 50 mLs of ethyl acetate (EtAc) to each tube
- d) Fortify each sample with internal standard (ISTD) spiking solution
- e) Reduce sample material particle size by using a high speed disperser for approximately 1 minute
- f) Add 2 g of anhydrous MgSO₄ (**ECMAG00D**) and 0.5 g anhydrous NaCl (**ECNAACL05K**).

Note: Carefully add the reagents to the tube to avoid contaminating the threads or rims of the tubes otherwise leaks may result

- g) 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
- h) Cool the sample in a -20 °C freezer for approximately 30 minutes
- i) Centrifuge at 10,000 RCF for 5 minutes

- j) 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
- k) 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)
- l) Dilute to 20 mLs with acetonitrile (MeCN) and cap with a glass stopper, vortex for 1 minute
- m) Freeze at -70 °C for 30 minutes
- n) Centrifuge the extract while frozen for 3 minutes (The MeCN will thaw during centrifugation)
- o) Directly after centrifugation in step n), filter > 15 mLs of the MeCN layer of the extract with a 0.45 µm syringe filter into a 15 mL glass centrifuge tube
- p) 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

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

3) GC Analysis

- a) For GC analyses, use the dual layer cartridge **ECPSAC1856**
- b) Add approximately 0.75 – 0.80 grams (~ 0.6 cm = 0.25 inches) of anhydrous MgSO₄ added to the top of the cartridge

- c) Condition the SPE cartridge by adding one cartridge volume (4.0 mLs) of MeCN using a UCT positive pressure SPE manifold
- d) Elute to waste
- e) Place a labeled 15 mL graduated disposable plastic centrifuge tube below the cartridge in the positive pressure SPE manifold
- f) Quantitatively transfer 1 mL of the sample extract from step 15 to the SPE cartridge
- g) Elute SPE cartridge in a drop wise manner (Regulated Flow Pressure = 35 psi) into a labeled 15 mL graduated glass centrifuge tube using MeCN
- h) 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
- i) 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
- j) Using an N-Evap (or equivalent) with the water bath set at 50°C and N₂ flow set at <10 liters per minute (LPM) (typical setting is 2 – 6 LPM), evaporate the sample to approximately 0.5 mL
- k) Add 3 mL of toluene to the centrifuge tube containing the sample
- l) Evaporate again to < 0.5 mL. (This is to insure all other solvents have been removed from the sample.)
- m) Bring the volume to 1.0 mL with toluene and vortex to mix solvent into sample
- n) Analyze by GCMS-EI and GCMS-NCI

DCN-110210-126