



Solid-Phase Extraction and Determination of Organotin by Micro-Liquid Chromatography Electrospray Ion Trap MS (Part A Water Samples)

UCT Product Number:

ECUNIC18 (C18 endcapped, 1100 mg/83 mL cartridge)

EPA Method 8323

March 2010

Analytes Determined Using This Method

Analyte	CAS No	LOD
Tributyltin chloride	1461-22-9	780 pg
Dibutyltin dichloride	683-18-1	970 pg
Monobutyltin trichloride	1118-46-3	1 ng
Triphenyltin chloride	668-34-8	NA
Diphenyltin dichloride	1135-99-5	920 pg
Monophenyltin trichloride	1124-19-2	NA

Note: Organotins can bond to glass surfaces, glassware must be specially treated. All glassware used in the extraction and analysis of organotins must be acid washed using the following procedure.

- Wash glassware in hot soapy water then rinse with DI water
- Prepare a pH 2 acid bath using 12 N HCl and soak glassware in acid for 24 hours
- Remove glassware from bath, then rinse with DI water followed by a methanol rinse
- Place in a 60°F oven until dry

Procedure

1. Initial Preparation

- a) Fill a 2 liter volumetric flask with sample water
- b) Adjust pH to 2.5 by adding about 600 µL of 12N HCl
- c) Stopper flask and invert several times to mix acid

2. Cartridge Conditioning

- a) Add 10 mL of methanol to the cartridge to activate

- b) Briefly turn on vacuum to draw through a small amount to top of frit
- c) Wait 1-2 minutes
- d) Add 10 mL of a methanol/1% acetic acid solution
- e) Draw about 2 mL through the cartridge then turn off vacuum
- f) Let solution sit for 1-2 minutes then draw through
- g) Add 10 mL of reagent water to cartridge and partially draw through

Note: Do not let the cartridge dry out after start of activation otherwise start over at step 2. a)

3. **Sample Extraction**

- a) Add the 2 liter sample to the cartridge and draw through at approximately 50 mL/minute (fast drip)
- b) Rinse volumetric flask and cartridge sides with 100 mL of reagent water and draw through

4. **Elution**

- a) Dry cartridge by drawing full vacuum for 10 minutes
- b) Place a clean, treated collection tube in the manifold
- c) Add a **first** portion of 10 mL of methanol/1% acetic acid solution to the cartridge, rinsing the sides during addition, then slowly draw through cartridge
- d) Add a **second** 10 mL portion of methanol/1% acetic acid solution to the cartridge, then slowly draw through
- e) Add a **third** 10 mL portion of methanol/1% acetic acid solution to the cartridge, then slowly draw through

Micro-concentration by TurboVap[®] Nitrogen Evaporation

- 1. Place the concentrator tube in the TurboVap[®] or other analytical evaporator in a lukewarm water bath at 30° C
- 2. Evaporate the solvent volume to 0.5 mL using a gentle stream of clean, dry N₂
- 3. The internal wall of the tube must be rinsed down several times with the final solvent (methanol/1% acetic acid) during the evaporation
- 4. Do not allow the extract to become dry
- 5. Transfer the extract to a 2 mL glass vial with a PTFE-lined screw-cap or crimp-top vial and store refrigerated at 4° C
- 6. Sample is ready for μ -LC-ES-ITMS analysis