Solid-Phase Extraction and Determination of Organotin by Micro-Liquid Chromatography Electrospray Ion Trap MS

(Part A Water Samples)

UCT Part Number:
ECUNIC18 (C18 endcapped, 1100mg/83mL cartridge)

EPA Method 8323

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
   a) Briefly turn on vacuum to draw through a small amount to top of frit
   b) Wait 1-2 minutes

c) Add 10 mLs of a methanol/1% acetic acid solution
d) Draw about 2 mL through the cartridge then turn off vacuum
e) Let solution sit for 1-2 minutes then draw through
f) Add 10 mLs 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 a 100 mLs 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 mLs 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 N_2
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

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