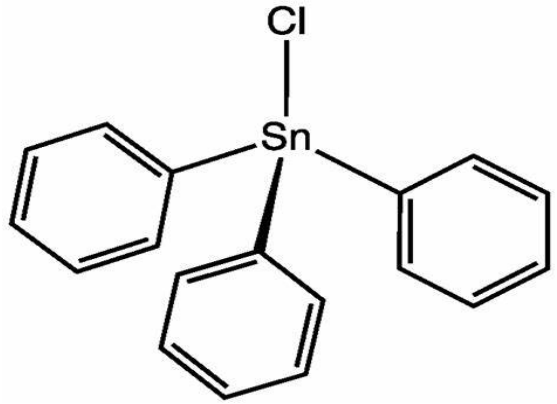
Solid-Phase Extraction and Determination of Organotin by Micro-Liquid Chromatography Electrospray Ion Trap MS



UCT Part Numbers

ECUNIC18
C18 endcapped
1100 mg/83 mL cartridge

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

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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 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

DCN-218030-185

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