MBT
DBT
TBT

Application Note for Speciation Analysis

Species : Monobutyltin, dibutyltin and tributyltin (MBT, DBT and TBT)
Sample Type : Marine Sediment (SOPH-1)
Sample Category : Environmental
Analysis : Species-Specific-Isotope Dilution Analysis+ GC-MS

Sample Preparation Procedure

Sample Weight : 0.05g
Isotopic Spike[1] : ^{119}Sn enriched butyltinmix
Reagent(s) : 4mL HAcO/MeOH (3:1)

1 **Microwave Heating Program**
Temperature Target : 70°C
Ramp Time : 1 minute
Heating Time : 4 minutes

2 **Derivatization Procedure**
0,5mL Extract + 4mL buffer HAcO/AcO 0,1 M at pH 4,9
Adjust pH to 4.9[2]
Add 0,5mL Hexane
Add 0,5mL $NaBEt_4$ (2%w/v) — **5 minutes mechanical shaking**

3 **Clean Up Procedure**
Separate Organic Layer
Pass Through Florisil Column — **Florisil Column**
Elute with 4 mL hexane and collect
Preconcentration with N_2 or Ar — **N-evap or equivalent**

4 **Chromatographic Separation**
T Injector - 250 °C / Splitless 0.5 min — **Tr-5 Column**
60 °C(0.5min) to 280 °C (4min) — **5% Phenyl Methyl Siloxane**
Ramp Rate : 30 °C / min — **30m x 0.25mm i.d. x 0.25 µm coating**

Recommended Equipment

Explorer 24

Explorer ‘S’ Class

File: sediment1.doc

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Marine Sediment

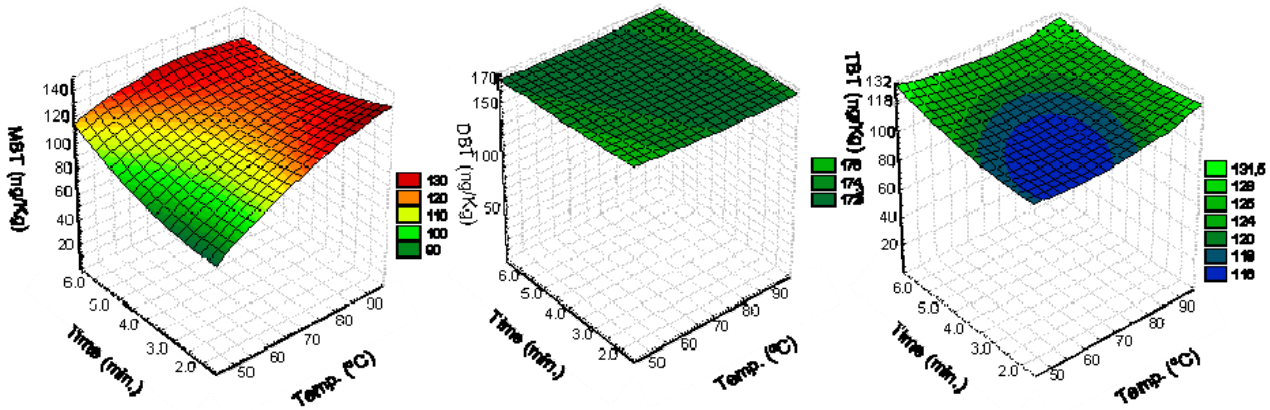
Detection and Analysis

Equipment Used

GC-MS: JEOL JMS-Q1000GC

Optimization of Sample Preparation[3]

MBT **DBT** **TBT**



Conditions: 70 ºC / 4 minutes

MBT 126 ± 6 ng Sn/g DBT 176 ± 6 ng Sn/g TBT 122 ± 4 ng Sn/g

Certified Value :
MBT (Not Certified) **DBT 174 ± 9 ng Sn/g** **TBT 125 ± 7 ng Sn/g**

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[1] Commercially available through ISC-Science (Spain) the addition of the ^{119}Sn-enriched species to the sample was done in a range of 1-10 times the expected concentration of the endogenous butyltin species in the tissue.
[2] Adjust pH with HCl conc. NH_4OH conc.
[3] Be aware that the use of species-specific isotope dilution analysis prevents the degradation of TBT during the extraction.