MBT
DBT
TBT

Application Note for Speciation Analysis

Species : Monobutyltin, dibutyltin and tributyltin (MBT, DBT and TBT)
Sample Type : Mussel tissue (CRM-477)
Sample Category : Biological Tissue
Analysis : Species-Specific-Isotope Dilution Analysis+ GC-ICP-MS or GC-MS

Sample Preparation Procedure

Sample Weight : 0.1g
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**
0,4mL 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

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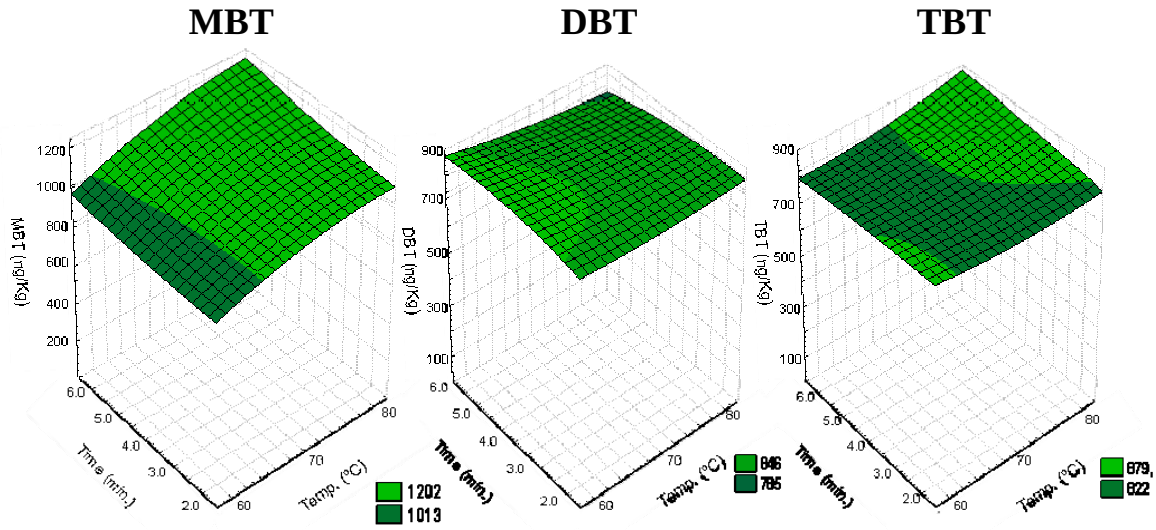
Detection and Analysis

GC-MS: JEOL JMS-Q1000GC

Equipment Used

GC-ICP-MS: Focus GC & Thermo X series

Optimization of Sample Preparation[3]



Conditions: 70 ºC / 4 minutes

MS	MBT 1071 ± 79 ng Sn/g	DBT 844 ± 22 ng Sn/g	TBT 823 ± 9 ng Sn/g
ICP	MBT 1030 ± 9 ng Sn/g	DBT 792 ± 6 ng Sn/g	TBT 841 ± 9 ng Sn/g
	Certified Value :		
	MBT 1013 ± 189 ng Sn/g	**DBT 785 ± 61 ng Sn/g**	**TBT 900 ± 78 ng Sn/g**

[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.