3.3 Analysis of Organotin in Fish (1) - GCMS

Explanation

Organotins such as tributyltin (TBT) and triphenyltin (TPT) are widely used as antifouling paints for ships and fishing nets, which has led to seawater and marine life pollution problems.

Conventionally, such compounds are analyzed using GC-FPD, but here an analysis example for GCMS with superb qualitative accuracy will be introduced.

Though tripentyltin (TPeT) is often used as the internal standard substance, this is not the best selection because TBT, TPT and TPeT have different recovery rates. In this example, a deuterium label compound that makes full use of GCMS features was used as the internal standard substance.

The advantage of the deuterium label compound as standard substance is that it is materially identical to the target compound but does not exist in the sample.

Pretreatment

Fig. 3.3.1 shows the methods of extraction from fish and seawater.

Analytical Conditions

Instrument : GCMS-QP5000

 $\label{eq:column} \begin{array}{ll} \mbox{Column} & \mbox{:} \mbox{ DB-1 } 0.32 \mbox{mm} \times 30 \mbox{m df} = 0.25 \mbox{μm} \\ \mbox{Column} & \mbox{:} \mbox{ 50°C(2min)-140°C(20°C/min)} \end{array}$

temperature $-220^{\circ}\text{C}(7^{\circ}\text{C/min})-310^{\circ}\text{C}$

(15°C/min)(6/min)

Injection inlet : 280°C

temperature

Interface temperature: 300°C

Carrier gas: He(40kPa)

Injection method: Spitless(2min)

Component	Selected ions (m/z)			
d27-TBT	295, 293, 316			
TBT	277, 275, 291			
Tetra-BT	291, 289			
TPeT	303. 305			
d15-TPT	366, 364			
TPT	351, 349			

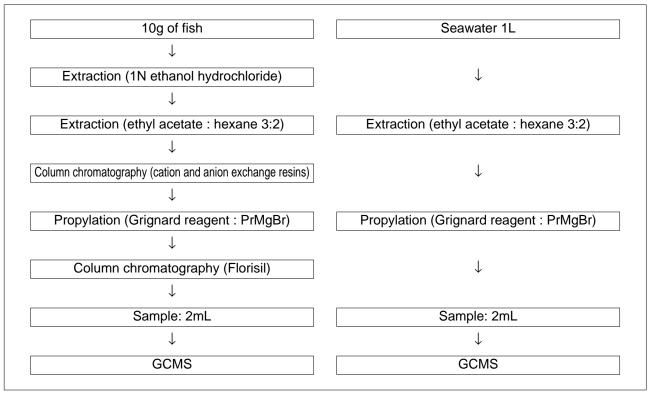


Fig. 3.3.1 Extraction methods for organotin in fish and seawater

3.3 Analysis of Organotin in Fish (2) - GCMS

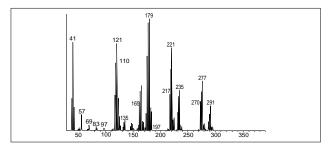


Fig. 3.3.2 TBT mass spectrum

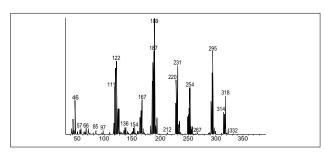


Fig. 3.3.3 d27-TBT mass spectrum

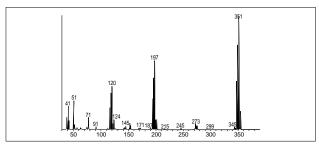


Fig. 3.3.4 TPT mass spectrum

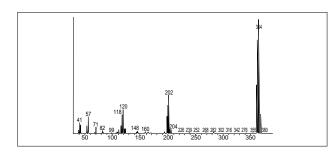


Fig. 3.3.5 d15-TPT mass spectrum

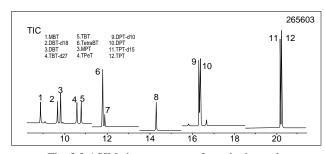


Fig. 3.3.6 SIM chromatogram of standard sample

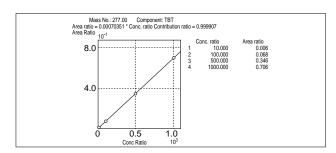


Fig. 3.3.7 TBT calibration curve (10 to 1000ppb)

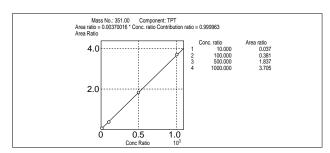


Fig. 3.3.8 TPT calibration curve (10 to 1000ppb)

5	28967	ID Type Component	: 6 Mas : Target : TBT	ss No. : 277.00
		Retention time Area Conc.	: 10.788 : 44583 : 1.021ppm	
1 1		Mass No. 1 291.00	Area 18581	Intensity ratio 42
10.5	291.0 277.0	00 00		

Fig. 3.3.9 SIM chromatogram of TBT in fish (sea bass)

Component	Sea bream (μg/g)	Sea bass (μg/g)	K port (μg/L)	W port (μg/L)
TBT	0.436	0.782	0.173	0.068
TPT	0.014	0.010	0.019	0.078

Chart 3.3.10 Quantitative results for tin in fish and seawater