Application Note No. 046

The Analysis of Organotin Compounds in Water by GC-MS using Large Volume Injection

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Introduction

Tributyl and triphenyl tin are both EC Red List compounds which must be determined in water down to levels of 0.1 ppb. Existing methods, involving the use of Grignard reagents for derivatisation, are complex, labour intensive and, as a consequence, costly.

This new approach utilises sodium tetraethyl borate as the derivatisation reagent which makes it considerably simpler. The Optic programmable injector is used to perform large volume injections of the derivatised sample, this increases the sensitivity and therefore eliminates the need for a pre-concentration step.

Extraction Method

- Place 200 ml of the water sample in a 250 ml volumetric flask
- Add tripentyl tin to a concentration of 100 ng/l
- Add 10 g of sodium chloride
- Add 1 ml of 1% sodium tetraethyl borate solution
- Swirl the sample until the salt dissolves
- Add 2.5 ml iso-octane
- Stopper the flask and shake for at least 30 minutes
- Use UHP water to top up to the mark of the volumetric flask
- Use anhydrous sodium sulphate if necessary to break the emulsion
- Remove a portion of the iso-octane layer with a pasteur pipette and transfer to a GC vial for analysis

Instrumentation

- Optic 2-200 Injector in LVI mode
- HP5890 Gas Chromatograph
- HP5972 MSD in SIM mode

Conclusions

The use of sodium tetraethyl borate as the derivatisation agent along with large volume injection simplifies and shortens the analysis of organotin compounds. Detection limits of 5 ng/L are achievable and good quality data is produced.

Results

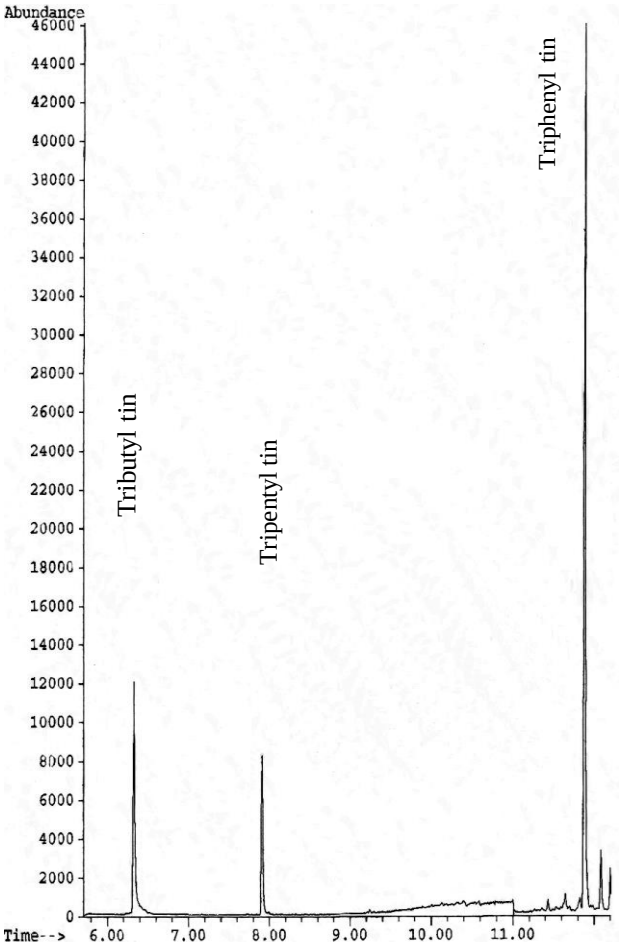

Figure: *50 µL injection of derivatised organotins at a concentration of 20 µg/L*

Acknowledgements

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Appendix

Optic 2-200 Injector Conditions:

Liner:	ATAS 'A' Type	
Injection volume:	50 µL	
Gas Flows:	Split:	50 ml/min
	Vent:	300 ml/min
Equilibration Time:		0:30 m:s
Initial Temperature:		80 °C
Vent Time:		0:24 m:s
Ramp Rate:		16 °C/s
Final Temperature:		300 °C
Split Open Time:		2:30 m:s

Gas Chromatograph Conditions:

Column: DB1-MS 30m x 0.25mm i.d. x 0.25 μm film

Initial Temperature:	90 °C
Initial Time:	2 mins
Ramp Rate:	20 °C/min
Final Temperature:	300 °C
Final Time:	0.5 mins

HP 5972 MSD

Mode:	SIM