

Analysing Quaternary Ammonium Salts (QAS) in Seawater by Difficult Matrix Introduction (DMI)

Diane Nicholas

- *No sample preparation necessary*
- *Removes water under controlled conditions prior to analysis*
- *Retains the salt and involatiles within the microvial*

Instrumentation

- ATAS Optic 2-200 programmable injector
- Agilent 5890 GC with 5971 MSD

Principles

- Inject 2-3 μL of seawater into a microvial and place in fritted liner in Optic injector
- Vent the water, heating the injector and column oven to 100 $^{\circ}\text{C}$
- Thermally desorb the sample at high temperature to degrade the QAS, under static flow conditions
- Transfer the analytes directly onto the GC column and analyse by GC-MS

Chromatogram

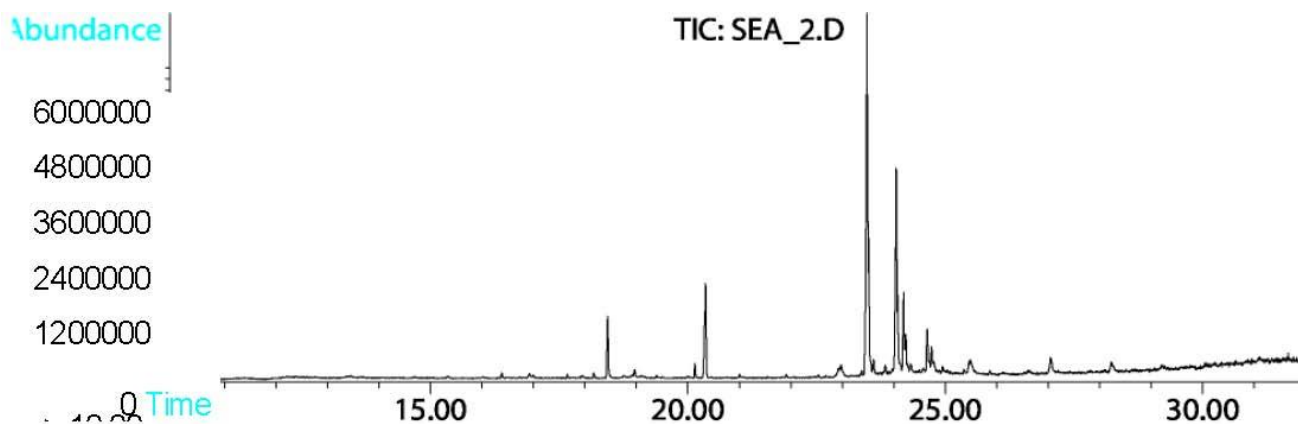


Figure 1: TIC of analysis of seawater by DMI

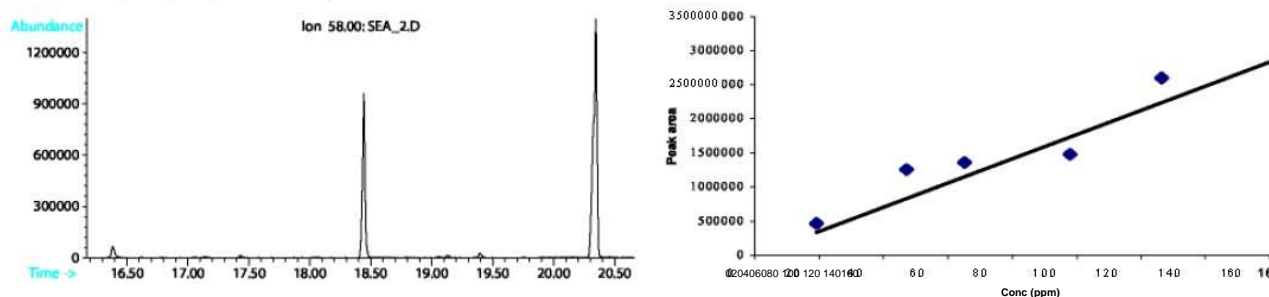
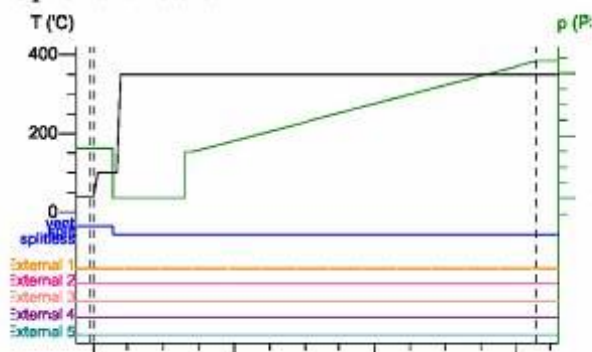


Figure 2: Selected ion for the quaternary ammonium salt Figure 3: Calibration with manual injection using the un-optimised method

We would like to thank John Sellers and Andy Dowding from AkzoNobel, UK for providing these samples

Appendix

Optic Parameters:



Liner: ATAS Fritted
 Microvial: 6 mm
 Mode: Expert
 Gas Flows: Vent: 370 ml/min
 Split: 50 ml/min
 Initial temperature: 40 °C
 Ramp rate 1: 4 °C/s
 Final temperature 1: 100 °C
 Hold time 1: 1.5 mins
 Ramp rate: 16 °C/s
 Final temperature: 350 °C
 End time: 37.5 mins
 Vent pressure: 8 psi
 Vent time: 1.5 mins
 Split open time: 1.5 mins
 Desorption pressure: 0 psi
 Desorption time: 5 mins
 Transfer pressure: 7.4 psi
 Transfer time: 0.5 mins
 Initial pressure: 7.4 psi
 Final pressure: 22 psi

GC Parameters:

Column: DB5-MS 30m x 0.25mm i.d. x 0.25um film
 Initial temperature: 45 °C
 Ramp rate 1:
 Final temperature 1: 20 °C/min
 Hold time 1: 100 °C 4.5
 Ramp rate 2: mins 10
 Final temperature 2: °C/min 350
 °C

MS Parameters:

Acquisition mode: Scan
 Low mass: High 50 m/z
 mass: Sampling 400 m/z 2
 number: Threshold: 500
 Transfer line: 280 °C
 Solvent delay: 10 mins