

IONSET SELECTION FOR SENSITIVITY OPTIMISATION OF ORGANOTIN ANALYSIS BY GC-MS

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Organotin analysis by GC-MS was carried out over a wide range of concentrations, from 1 ng/ml to 1000 mg/ml using standard compounds to test the sensitivity and detection limits of tributyltin (TBT), dibutyltin (DBT), and monobutyltin (MBT) compounds on a Shimadzu GC-MS 5050A. This study was essential for the characterisation of TBT sorption on sand, silt and clay particles. Tetraethyltin and phenylated derivatives of TBT, DBT and MBT were tested, using phenyl magnesium bromide as the derivatising agent, three different capillary columns, and both full mass scan and selected-ion-monitoring (SIM) modes.

The detection under the full mass scan mode resulted in much higher levels of background noise than under the SIM mode. The noise generated by the full mass scan mode varied from 150,000 to 350,000 counts, while the SIM mode produced a background varying from 35,000 to 45,000 counts. The extremely reactive Grignard reagent caused the derivatisation of other sample components producing trace amounts of many other phenyl compounds, which, under the full mass scan mode, generated peaks interfering with those of the organotin compounds at low level concentrations. The absolute detection limit of these organotin compounds achieved by GC-MS was 1 – 10 ng as Sn under full mass scan mode, and 10 – 100 pg as Sn under the SIM mode, respectively, with 1-ml injection and a split-ratio 10.

The SIM mode was further refined by careful selection of the ion-set for detection. Only ions that were typical of the organotin compounds were included in the mass detection list. Noise levels were hence reduced to less than ± 1000 counts. The GC-MS sensitivity and detection limits were significantly improved without the need for further cleaning during the sample preparation stage. Less than 1 pg as Sn of TBT, DBT, and MBT, respectively, can be reliably detected.
