Organotin Speciation in Environmental Matrices by Automated *On-line* Hydride Generation-Programmed Temperature Vaporization-Capillary Gas Chromatography-Mass Spectrometry Detection

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In the present contribution, a new automated *on-line* hydride generation methodology was developed for dibutyltin and tributyltin speciation at the trace level, using a programmable temperature-vaporizing inlet followed by capillary gas chromatography coupled to mass spectrometry in the selected ion-monitoring mode acquisition (PTV-GC/MS(SIM)). The methodology involves a sequence defined by two running methods, the first one configured for hydride generation with sodium tetrahydroborate as derivatising agent and the second configured for speciation purposes, using a conventional autosampler and data acquisition controlled by the instrument's software.

From the method-development experiments, it had been established that injector configuration has a great effect on the speciation of the actual methodology, particularly, the initial inlet temperature (-20ºC; He: 150 ml/min), injection volume (2 µl) and solvent characteristics using the solvent venting mode. Under optimized conditions, a remarkable instrumental performance including very good precision (RSD < 4%), excellent linear dynamic range (up to 50 µg/ml) and limits of detection of 0.12 µg/ml and 9 ng/ml, were obtained for dibutyltin and tributyltin, respectively. The feasibility of the present methodology was validated through assays upon in-house spiked water (2 ng/ml) and a certified reference sediment matrix (Community Bureau of Reference - CRM 462, Nr. 330 dibutyltin: 68 ± 12 ng/g; tributyltin: 54 ± 15 ng/g on dry mass basis), using liquid-liquid extraction and solid phase extraction sample enrichment and multiple injections (2 × 5 µl) for sensitivity enhancement. The methodology evidenced high reproducibility, is easy to work-up, sensitive and

showed to be a suitable alternative to replace the currently dedicated analytical systems for organotin speciation in environmental matrices at the trace level.

References

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