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ACCEPTED MANUSCRIPT

Determination of ultratrace levels of Tributyltin in waters by Isotope Dilution and Gas Chromatography coupled to tandem Mass Spectrometry

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ABSTRACT

The current EU legislation lays down the Environmental Quality Standards (EQS) of 45 priority substances in surface water bodies. In particular, the concentration of tributyltin (TBT) must not exceed 0.2 ng L-1 and analytical methodologies with a Limit of Quantification (LOQ) equal or below 0.06 ng L⁻¹ are urged to be developed. This work presents a procedure for the determination of ultratrace levels of TBT in water samples by Isotope Dilution and GC-MS/MS operating in Selected Reaction Monitoring (SRM) mode which meets current EU requirements. The method requires the monitorization of five consecutive transitions (287>175 to 291>179) for the sensitive and selective detection of TBT. The measured isotopic distribution of TBT fragment ions was in agreement with the theoretical values computed by a polynomial expansion algorithm. The combined use of Tandem Mass Spectrometry, a sample volume of 250 mL, the preconcentration of 1 mL of organic phase to 30 μL and an injection volume of 25 μL by Programmed Temperature Vaporization provided a LOQ of 0.0426 ng L⁻¹ for TBT (calculated as ten times the standard deviation of nine independent blanks). The recovery for TBT calculated in milli-Q water at the EQS level was 106.3 ± 4 %. A similar procedure was also developed for the quantification of dibutyltin (DBT) and monobutyltin (MBT) in water samples showing satisfactory results. The method was finally implemented in a routine testing laboratory to demonstrate its applicability to real samples obtaining quantitative recoveries for TBT at the EQS level in mineral water, river water and seawater.

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- **Keywords:** Tributyltin, Isotope Dilution Mass Spectrometry, GC-MS/MS, EU Water Framework Directive.
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37 The determination of TBT in water by Isotope Dilution and GC-MS/MS is proposed.

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