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## **Novel solid phase micro extraction (SPME) Method for GC-MS analysis of Tributyltin**

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Tributyltin (TBT) is an organotin compound belongs to the group of Persistent Organic Pollutants (POPs) and one of the active ingredients in biocides used to control a broad spectrum of organisms. Antifouling paints which are used for boat hulls, docks, fishnets contain tributyltin to prevent the growth of aquatic fouling organisms. Increasing concentration of TBT in the environment, due to anthropogenic activities cause toxicological impact on target and non-target organisms having sexual disorders like sex changes at ultra-trace level called imposex. According to WHO, the No Observed Effective Level (NOEL) of TBT is below 1 ng L<sup>-1</sup>, thus a highly specific and sensitive analytical methods are needed to detect TBT in parts per trillion (ppt) level. In the present study, a new ultra-trace quantification method to detect TBT using Solid Phase Micro Extraction (SPME) followed by Gas Chromatography-Mass Spectrometry (GCMS) was optimized. TBT derivatization to tributyltinhydride was carried out using potassium borohydride (KBH<sub>4</sub>). Medium polar PDMS/DVB, fused silica (65 μm, 24 Ga) SPME fiber were found to be the most appropriate conditions for extraction and pre-concentration of derivatized TBT hydride compound. The best conditions for the extraction were optimized and analyses were done by using GC-MS under 270 °C desorption temperature and 1 mL min<sup>-1</sup> of helium gas flow rate. Field samples were collected from highly polluted and heavy boat traffic area along the coastal belt from Dikkovita to Mirissa, Sri Lanka. The optimal extraction conditions were found to be 10 minutes extraction time, pH 4.9 and the ambient temperature 30 °C. The highest TBT concentration was detected in Colombo port (303±4.7 ng L<sup>-1</sup>) following the TBT was recorded in the fishery harbors at Beruwala (124±4.1 ng L<sup>-1</sup>), Galle (110±4.1 ng L<sup>-1</sup>), Dikkovita (97±4.3 ng L<sup>-1</sup>), Hikkaduwa (77±5.3 ng L<sup>-1</sup>) and Ambalangoda (57±5.2 ng L<sup>-1</sup>). TBT concentrations in Dehiwala, Wellawatta, Madu, Benthota and Gin ganga river mouths were recorded as 268±4.1 ng L<sup>-1</sup>, 240±3.4 ng L<sup>-1</sup>, 214±3.3 ng L<sup>-1</sup>, 145±2.7 ng L<sup>-1</sup> and 50±2.1 ng L<sup>-1</sup> respectively. The recovery of the TBT extraction was 87±2.1 % for the artificial sea water while the Minimum Quantification Level (MQL) was 1 ng L<sup>-1</sup>. The Minimum Detection Level (MDL) of the method was calculated as 0.3 ng L<sup>-1</sup>. The optimized method is a solvent free and the use of SPME is highly sensitive method to detect TBT in parts per trillion (ppt) levels.

**Keywords:** Tributyltin, Solid Phase Micro Extraction (SPME), Gas Chromatography Mass Spectrometry (GCMS), Imposex.

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