Thesis Title	Sample Handling of Biological Samples for Gas Chromatographic Determination of Organotin Compounds
Name	Tinnakorn Tiensing
Degree	Master of Science (Applied Analytical and Inorganic Chemistry)

Thesis Supervisory Committee

Chatchavalee	Kalambaheti	Ph.D
Juwadee	Shiowatana	Ph.D
Pornsawan	Visoottiviseth	Ph.D
Prapin	Wilairat	Ph.D

Date of Graduation 7 May B.E. 2540 (1997)

ABSTRACT

The removal of interferences and organic matrices for butyltin and phenyltin analysis in biological samples was reported with solid phase extraction (SPE) method. The determination of butyltin and phenyltin compounds was performed by gas chromatography with flame photometric detector (GC-FPD) equiped with sulphur mode (393 nm) on HP-5 capillary column (25 m×0.2 mm i.d. × 0.11μm film thickness). The limit of dectection of propylated product of di- and tributyltin were 13.04 and 20.45 ppb, and propylated product of di- and triphenyltin were 32.01 and 46.86 ppb, respectively. Butyltin and phenyltin compounds in fish and oyster samples were extracted by 0.1% tropolone in ether. Lipid extract solution could be destroyed by addition of NaOH solution in a saponification step. These solutions or extracted solutions

were further derivatized with ethylmagnesium bromide or n-propylmagnesium bromide to prepare the volatile derivatives. Derivatized solution were passed through SPE clean-up column packed with silica gel or florisil. The optimum packing which gave high efficiency to trap lipid was 10-15 g of florisil. The eluent for eluting organotin derivatives was 20-30 ml of ether. The recovery of the clean-up method attained 100 %. Adsorption technique could also be used to remove interferences by collecting the interested fraction when eluting with hexane and THF. The minimum amount of non-polar packing materail should be 30 g of XAD-4 and XAD-16.

Preconcentration technique was conducted to enhance the sensitivity of TBT and TPT analysis in seawater sample by using silica gel or C_{18} column. The column was conditioned with 1%(v/v)HCl in MeOH, followed by 0.1% tropolone in MeOH. Elution of TBT and TPT was performed using 10 ml of ether or 0.03% tropolone in ether with 90-100% recovery. The maximum sample volumes that can be loaded on a 200 mg of silica gel or a 200 mg of C_{18} column were 500 ml and 1000 ml, respectively. This preconcentration procedure could enhance the sensitivity up to 1000 times from the original.