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REVIEW

Organotin Speciation Analysis Based on Liquid or Gas Chromatography

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Organotin compounds have been used on a global scale for many decades now and thus have become global environmental contaminants. These compounds are highly toxic and deleterious effects on numerous organisms have been demonstrated. Several analytical methods for the determination of organotin compound in environmental (*e.g.*, water, sediment and shellfish) have been developed. Analysis of organotin species in the environment needs highly selective and sensitive methods. This paper reviews the organotin speciation analysis based on liquid or gas chromatography.

Key Words: Organotin, Liquid chromatography, Gas chromatography.

INTRODUCTION

Frankland[1] reported the synthesis of the first organotin compound. Throughout the 160 years following Frankland's initial report, a wide range of organotin compounds has been synthesized using a variety of preparatory processes. As a result, tin has more organometallic derivatives uses today than any other metal[2]. Organotin is mainly used in its forms $RnSnX_{(4-n)}$ (R = alkyl or aryl group, X = anionic group). Originally, organotin compounds were developed as stabilizers for chlorinated hydrocarbons such as poly(vinyl chloride) (PVC). They have also been used to stabilize non-chlorinated compounds such as polyamides (nylon). As knowledge of their chemistry grew, the number of their applications also grew to include catalytic agents for instance the preparation of silicone rubbers and polyurethane foams, biocides used in aquaculture (antifouling agents), agriculture as fungicides and insecticides and industry such as wood and textile preservation. Organotin compounds act as effective antifouling compounds in paints and coatings, with the primary usage of these coatings being marine vessels, both seafaring and private freshwater pleasure craft. These fouling-resistant coatings are applied to the keel or bottom, of the marine vessels to prevent growth of undesired organisms (algae, bacteria, barnacles, *etc*.), which all can contribute to the fouling of a vessel's keel.

Organotin compounds have been in use as antifouling agents since the early 1970s. As a result of the broad spectrum of usages of organotin compounds, multiple pathways are available for these compounds to enter the environment. Table-1 lists the applications of organotin compound[3].

TABLE-1
SOURCES AND POSSIBLE PATHWAYS FOR THE INTRODUCTION OF ORGANOTINS TO THE ENVIRONMENT

Compound	Application
Monobutyltin (MBT)	PVC stabilizer Catalyst Precursor for glass treatment
Dibutyltin (DBT)	PVC stabilizer Catalyst for polyurethane foams and silicones
Tributyltin (TBT, biocide, used mainly against fungi and molluscs)	Antifouling paints Wood and stone treatment Textile preservation Water paints Industrial water systems Paper industry Leather industry Breweries Anti-parasite
Triphenyltin (TPT, fungicide)	Agrochemical pesticide Antifouling paints

Triorganotin compounds are the most toxic and a decrease in the number of organic substituents linked to the tin atom is related to lower toxicity[2]. The chemical form of the organic group also determines toxic effects. An increasing order of toxicity is as follows: phenyl < butyl < propyl < methyl < ethyl[4]. As a consequence, speciation information is required to determine the risk due to the presence of organotins in the environment.

Organotin compounds are highly persistent in the environment with tributyltin compounds having half-lives ranging from 1 to 5 years when partitioned into the sedimentary layer[5].

Because of the low solubilities of organotin compound in water and due to their hydrophobic characteristics, easily cross biological membranes and accumulate in lipophilic sites, mainly in lipid tissues. This process is generally known as bioaccumulation of organometals by organisms.

In the 1980s, the use of organotin compound (OTCs) were regulated in some developed countries such as England, France and the United States, but tributyl tin has still been detected in the aquatic environment of these countries at a contamination level that causes adverse effects on the aquatic environment. In developing countries, there have been no controls on the usage of organotin compounds. In October 2001, the International Maritime Organization (IMO) adopted the Internal Convention on the Control of Harmful Antifouling Systems (AFS Convention), which prohibited the use of organotins as effective ingredients in antifouling systems for ships.

Chemical speciation analysis: Different analytical techniques have been developed for the chemical speciation of organotin compounds. However, several challenges still remain for the analytical determination of these compounds in different matrices. Some of these challenges are the improvement of detection limits. The simultaneous determination of different organotin compounds using the same analytical procedure; and the selectivity of extraction procedures to avoid interferences from the matrices, degradation and/or processing of organotin compounds[6,7].

As shown in Fig. 1, many critical steps are involved in the analytical procedure used for the chemical speciation of organotin compounds in different matrices (sediment, water, soil or biota). The variety steps include: sampling, sample storage, extraction of organotin compounds (transfer of the analytes of interest from a complex matrix to a simpler solution), preconcentration or clean up (removal of impurities co-extracted together with the compounds of interest), derivatization (transformation of the analytes into an extractable species such as a more volatile compound), utilization of an appropriate analytical technique for the identification and quantification and interpretation of the results.

Sampling: The first step of a chemical analysis is sampling. For the chemical speciation of tin compounds, strong caution is necessary in this step since the environmental concentrations of organotin compounds are generally very low. Thus, the choice of sample locations and sampling period should be taken into account in both biotic and abiotic samples[8].

Extraction: Different extraction techniques have been employed as the sample pretreatment techniques for the speciation of organotin compounds such as solvent extraction or liquid-liquid extraction (LLE). But, these extraction techniques are considered to be time consuming and require a large amount of organic solvents. Because of this, different microextraction techniques have been developed and solid-phase microextraction (SPME), stir bar sorptive extraction (SBSE) and liquid-phase microextraction (LPME), are the typical representatives. Liquid-phase microextraction include three different extraction modes, single drop microextraction (SDME) fiber based LPME (HF-LPME) and dispersive liquid-liquid microextraction (DLLME). Fast extraction methods (FEMs) such as microwave assisted extraction (MAE), supercritical fluid extraction (SFE) and pressurized liquid extraction (PLE).

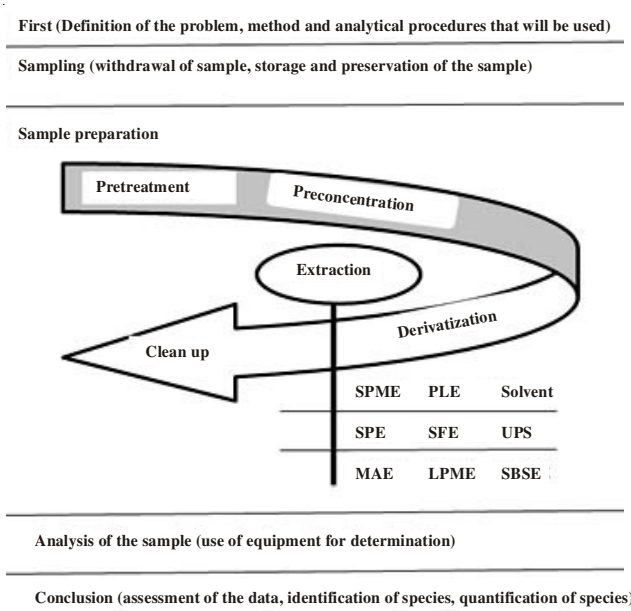

Fig. 1. Stages of a general analytical procedure used in the analysis of chemical speciation of organotin compounds

However, disadvantage of these method which employ high pressure and/or temperature is the possible loss of chemical stability of the chemical forms of the elements. On the other hand the use of ultrasonic radiation for the extraction of organotin compounds is the recent approaches in sample treatment such as ultrasound probe sonication (UPS). Molecular imprinting (MIPs) is the technique of choice for tailor made materials which use in solid phase extraction and it is appropriate mainly when a selective extraction must be performed and the commonly used sorbents lack selectivity. As well as conventional solid phase extraction, MISPE procedures could be used for off-line and on-line procedures and according to the selectivity of the polymer they could be used for compound specific or group-specific extractions.

The use of liquid-solid extraction solid phase extraction in organotin preconcentration presents undoubted advantages. Main advantages of the solid phase extraction method are the possible integration of columns and cartridges in on-line flow injection systems, less solvent consuming, ease of use and possible application as species storage device for field sampling. Commonly used methods for organotin extraction, providing preconcentration factors up to 1000, Implicate bonded silica sorbents with a variety of functional groups, such as octadecyl (C_{18})[9] and octyl (C_8)[10] to provide non polar interactions. Fullerenes C60 have also been used for retention of organotin compound extraction from Different samples[11,12].

Derivatization: For organometallic compounds, derivatization is commonly related to gas chromatography analysis in order to provide extractable species such as more volatile thermally stable compounds prior to separation. Hydride generation is the most extended technique which use tetrahydroborate, followed by alternative aqueous alkylation reactions such as those provided by sodium tetraethyl borate, tetra(*n*-propyl)borate or tetrabutylammonium tetrabutylborate. Nowadays non-aqueous alkylation, such as the use of Grignard reagents, is less used due to high time consumption, complex analytical set-up and a relatively tedious multi-step procedure.

Separation: Commonly used separation techniques, such as gas chromatography (GC) or liquid chromatography (LC), have been popular. In the last decade, advances in the methods of extraction and separation techniques have been developed in conjunction with highly sensitive and element specific detectors such as atomic absorption spectrometry (AAS), atomic emission spectrometry (AES), flame photometric detection (FPD), pulsed flame-photometric detection (PFPD), microwave-induced plasma atomic emission detector (MIP-AED) and mass spectrometry (MS). In particular, inductively coupled plasma mass spectrometry (ICP-MS) has gained popularity as a sensitive and selective detector when coupled to LC and gas chromatography for determination of butyl tins.

Analytical methods for organotin species determination: The monitoring of organotins in environmental at extremely low concentrations is an important issue, which needs highly sensitive analytical techniques. For the speciation of organotin compounds, the coupling of chromatographic separation with element-specific spectrometric detection has proven to be useful. Most of the analytical methodologies developed for the speciation of organotins are based on gas chromatography (GC) for its high resolution power and availability of sensitive detectors. However, gas chromatography requires a previous derivatization of the non-volatile compounds and this affects the duration and accuracy of the total analytical procedure, especially for the analysis of complex natural matrices. The use of liquid chromatographic (LC) methods is that does not need derivatization before analysis. Different sample treatment in chromatography-based speciation of organotin pollutants in the last 10 years are summarized in Table-2.

TABLE-2
SAMPLE PREPARATION METHODS APPLIED WITHIN A REPRESENTATIVE SELECTION OF RECENT (2000–2010) RESEARCH AND APPLICATION PAPERS DEALING WITH TIN SPECIATION ANALYSIS IN ENVIRONMENTAL AND BIOLOGICAL SAMPLES

Matrix	Analytes	Techniques of extraction	Separation	Detection	Ref.
Seawater	TBT and TPhT	Polymer-based SPE	HILIC	ESI-MS	13
Fish and mussel	TBT	UAE	GF	AAS	14
Sediments	Butyltins	C_8 SPE	GC	PFPD	10
Seawater	Butyltins	SWCNT as SPME	GC	MS	15
Shellfish	DBT, TBT, DPhT and TPhT	UAE	HPLC	ICP-MS	16
Sewage sludge	Butyl-, phenyl- and octyltins	MSE, UAE, MASE	GC	MS	17
Mussels	Butyltins	LLE	GC	MS	18
River water and seawater	Butyltins	HS-SPME	GC	MS/MS	19
Water matrices	MMT, DMT, MBT, DBT, TBT, MPhT, DPhT and TCyHT	*In situ* derivatization and LLE	GC	PFPD	20
Human urine	Butyl- and phenyltins	HS-SPME	CGC	MIP-AED	21
Urine	Butyl- and phenyltins	*In situ* derivatization and LLE	GC	MIP-AED and MS	22
Agricultural soils	Fenbutatin oxide (FBTO)	PLE	GC	AED	23
Seawater	Methyltins	–	HPLC	HG–ICP/MS	24
Fish, crustaceans, bivalves, cephalopods	Butyltins	LLE	GC	AED	25
Water	TBT, TPhT, TMeT and TPrT	SPME	HPLC	ICP-MS	26
Biota	Butyltins	MISPE	LC	ICP-MS	27
Water	Butyl and phenyltins	DLLME	GC	FPD	28
Seawater, shellfish	Butyltins	HS-SDME	GC	ICP-MS	29
Mussel tissues	Butyltins	SPE	GC	MS	30
Soil	Butyl-, phenyl- and octyltins	MSAE, ASE, MAE and UAE	GC	PFPD	31
Textile and plastics	TBT, TPhT and TET	MASE	HPLC	ESI-MS	32
Surface waters, sediments and biota	Butyltins	HS-SBSE	TD-GC	MS	33
French brandies and wines	Ethylated organotins	SPME	GC	PFPD	34
Sediments	Butyltins	HS-SPME	GC	MS/MS	35
Freshwater origin sediment and mussel	Butyltins	LLE	GC	ID-ICPMS	36
Seafood	Butyltins	UAE	GC	MIP-AES	37
Fish	Butyl- and phenyl-tins	LLE	GC	FPD	38
Fortified flour	TBT, TPhT, TrPhT, TET and TrET	MASE	NPHPLC	UV	39
Seawater	Tributyltin and 4-hydroxybutyldibutyltin	LLE	LC	APCI-MS	40
Aquatic plants	methyl-, butyl- and phenyltin	SPME	GC	PFPD	41
Mussels tissue, Oysters tissue and marine sediment	Butyltins	UPE and MISPE	GC	FPD	42
Surface sediments and mussels	Butyl-, Octyl- and phenyltins	LLE, HS-SPME	GC	PFPD	43
Water and sediments	Butyltins	C_{60} as SPE	GC	MS	11 12

Matrix	Analytes	Techniques of extraction	Separation	Detection	Ref.
Activated sludge batch reactors	TBT, DBT, MBT and TPhT	UAE	GC	FPD	44
Water and sediments	Butyl- and phenyltins	Automated HS-SPME	RTL-GC	MS	45
Water and sediments	DBT and TBT	LLE, SLE	PTV-GC	SIM-MS	46
Fresh and canned mussels,	Butyltins, MPhT and DPhT	SLE	HPLC	HG-AAS	47
Waters and marine sediments	Methyl-, butyl- and phenyltins	UPE	PT- CGC	MIP-AED	48
Harbour sediment	Butyltins and phenyltins	SPME	GC	PFPD	49
Water	TBT	HS-SDME	GC	MS	50
Sediments and seawater samples	MBT, DBT, TBT and TPhT	MIP	GF	AAS	51
Water samples	MBT, DBT, TBT and TPhT	SLMP	GC	FID	52
Seawater samples	TBT and TPhT	LPME	GC	MS-MS	53
Fresh Waters and Fish	DBT and TPhT	C_{18} -SPE	µLC	ES-ITMS	54
Polychlorinated biphenyl-based transformer oil samples	MPhT, DPhT and TrPhT	Solvent extraction	GC	MS, FPD and AED	55
Biological samples	Methyl-, butyl-, phenyl- and octyltins	HS- SPME	GC	PFPD	56
Seawater and sediment	Butyltins	LLE, SLE	GC	IL-ICP-MS	57
Natural waters	Butyltins	LLE	GC	QFAAS	58
Mussel and oyster tissue	DBT, TBT and TPhT	C_{18} -SPE	HPLC	ICP-MS	59
Water, sediments and mussels	TeBT, TBT, DBT, MBT, TPhT, DPhT, MPhT and TePhT	SPE, SLE	LPGC	MS/MS	60
Sediment, tissue and water samples	TeBT, TBT, DBT, MBT, TPhT, DPhT, MPhT, DCyT and TCyT	SLE, LLE	GC	HRMS	61
Water samples	Butyltins	SPME	MCGC	AED	62
Wines	Butyltins	HS-SPME	GC	MS	63
Water samples	butyltin and phenyltin	LLE	GC	MS–MS	64
Shellfish	butyltin and phenyltin	C_{18} -SPE	LC	FD	65
Standard Solution	TeET and TeMT	–	GC	SLEI, TLEI and LIAF	66
Sediments	Butyltin and phenyltin	PLE	LC	ICP-MS	67
Aqueous standard solutions, harbor water and mussels	TBT and TPhT	SBSE	CGC	ICP-MS	68
Water and sediment	TeET,TeBT	HS- SPME	GC	FID	69
Water samples	Tetraalkyltin, Tetraarltin and butyltins	MMLLE	GC	MS	70

AAS: Atomic absorption spectroscopy; ASE: Accelerated solvent extraction; APCI-MS: Atmospheric pressure chemical ionization mass spectrometry; C-GC: Capillary gas chromatography; DBT: Dibutyltin; DMT: Dimethyltin; DPhT: Diphenyltin; DLLME: Dispersive liquid-liquid micro extraction; ESI-MS: Electro spray ionization mass spectrometry; ES-MS: Electrospray mass spectrometry; ES-ITMS: Electrospray/ion trap mass spectrometry; FID: Flame ionization detector; FPD: Flame photometric detection; GC: Gas chromatography; GC-ICP-MS: Gas chromatography inductively coupled plasma mass spectrometry; GF-AAS: Graphite furnace atomic absorption spectroscopy; HS-SDME: Head space single drop micro extraction; HS-SPME: Head space solid phase micro extraction; HILIC: Hydrophilic interaction chromatography; HPLC: High performance liquid chromatography; HG-ICP-MS: Hydride generation inductively coupled plasma mass spectrometry; HGPT-GC-ICP-MS: Hydride generation purge and trap gas chromatography inductively coupled plasma mass spectrometry; HG: Hydride generation; HRMS: High-resolution mass spectrometry; IL-ICP-MS: Isotopically-labelled inductively coupled plasma mass spectrometry; IDMS: Isotope dilution mass spectrometry; IT-MS: Ion trap mass spectrometry; LLE: Liquid-liquid extraction; LPME: Liquid phase micro extraction; LC: Liquid chromatography; LPGC: Low pressure gas chromatography; LIAF: Flame laser-induced atomic fluorescence; MMT: Monomethyltin; MBT: Monobutyltin; MPhT: Monophenyltin; MIP: Molecularly imprinted polymer; MSE: Mechanical stirring extraction; MSAE: Mechanical stirring acid extraction; MISPE: Molecularly imprinted solid-phase extraction; MASE: Microwave assisted solvent extraction; MMLLE: Microporous membrane liquid–liquid extraction; MC-GC: Multicapillary gas chromatography; MIP-AED: Microwave induced plasma atomic emission detector; MIP-AES: Microwave induced plasma atomic emission spectrometry; MS/MS: Tandem mass spectrometry; NP-HPLC: Normal phase high performance liquid chromatography; PFPD: Pulsed flame photometric detection; PLE: Pressurized liquid extraction; PT: Purge and trap; PTV-GC: Programmable temperature-vaporizing inlet gas chromatography; QFAAS: Quartz furnace atomic absorption spectroscopy; RP-HPLC: Reversed phase high performance liquid chromatography; RTL-GC: Retention time locked gas chromatography; SLMP: Supported liquid membrane probe; SFE: Supercritical fluid extraction; SWCNT: Single-walled carbon nano tubes; SLEI: Single-step flame laser-enhanced ionization; SIM-MS: Selected ion-monitoring mass spectrometry; SPE: Solid phase extraction; SPME: Solid phase micro extraction; SBSE: Stir bar sorptive extraction; TD-GC: Thermal desorption gas chromatography; TLEI: Two-step flame laser-enhanced ionization; TBT: Tributyltin; TeBT: Tetrabutyltin; TCyHT: Tricyclohexyltin; TPhT: Triphenyltin; TePhT: Tetrphenyltin; UPE: Ultrasonic probe extraction; UPS: Ultrasound probe sonication; UAE: Ultrasonic assisted extractions; UV: Ultra violet.

Conclusion

Organotins have been shown to have a harmful effect on the environment at reasonably low concentrations and bioaccumulation can occur in real samples. Consequence, it is necessary to monitor the environment for these compounds. The determination of the long-term cycling and toxicity of the organotin compounds present in the environment requires the combination of a separation technique with a sensitive detector. There is a lot of research has been carried out to develop analytical techniques that are able to quantitatively

determine the chemical form of trace elements in a wide variety of sample matrices. The combination of two analytical techniques (separation and detection) can give useful methods with enough sensitivity to quantify organotin species as well as supply important structural information, as a result allowing comprehensive identification of unknown species. Of the separation techniques, gas chromatography enables the separation of most species in a single run with excellent resolution. Regrettably as most organotins are not volatile, it is necessary to introduce a time consuming and potentially problematic derivatization step. Whereas liquid chromatographic approaches obviate the need for a derivatization step, resolution is frequently poorer.

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