DEVELOPMENT AND APPLICATION OF A COMPREHENSIVE ANALYTICAL METHOD FOR SEMI-VOLATILE ORGANIC COMPOUNDS IN SEDIMENT SAMPLES USING A GAS CHROMATOGRAPHY-MASS SPECTROMETRY DATABASE SYSTEM

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September 2014

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Contents

Abstract

A novel gas chromatography-mass spectrometry (GC/MS) database (AIQS-DB) for identification and quantification of micro-pollutants is described. Their basic principle, basic structure, basic features, advantages and its application status are explained. Samples are screened and determined for organic pollutants using AIQS-DB without the use of standards. Therefore, this method is simple and easy to perform. The database is applicable for various uses, especially investigation of the causes of environmental pollution incidents, and finding a special feature of environmental pollution by chemicals at sampling sites.

A comprehensive analytical method for semi-volatile organic compounds (SVOCs) in sediment samples with the combination of the AIQS-DB and a pre-treatment method comprising of extraction and column clean-up has been developed. In the development of the comprehensive method, 119 model compounds (MCs) that are representative SVOCs registered in the AIQS-DB are used. Recoveries of them by liquid-liquid extraction with dichloromethane which are one of the pre-treatment steps for sediment samples showed that most SVOC, except for polar substances, can be extracted quantitatively. For the clean-up of extracts, silica-gel column chromatography using acetone–hexane solution as an eluent was the most suitable clean-up method for sediments. From the overall recovery tests, it was confirmed

that the developed comprehensive method can quantitatively analyze most SVOCs in sediments except for polar substances. In addition, the results of the overall recovery tests provided useful information for predicting the recoveries of substances registered in the database. The information was as follows: (1) generally, the overall recovery decreases with decreasing Log Kow, (2) substances whose Log Kow are below 1 cannot be analyzed quantitatively by the developed method, (3) if substances dissociate in water, even though their Log Kow values are larger than 1 (e.g. pentachlorophenol), their recoveries are below 60%, (4) substances that have hydroxyl functional groups and/or amino functional groups show low recoveries, and substances that have a Log Kow below 3 and also have functional groups that contain active hydrogen cannot be recovered more than 60%, (5) the recovery rates decrease with the increase in the number of active hydrogen functional groups. The method detection limits of the developed method were 4 μg/kg when measuring by TIM and 0.4 μg/kg by SIM. From the analysis of certified reference materials, it was confirmed that although the accuracy and precision of the developed method were slightly lower than those of conventional methods that are used for targeted analysis, its performance is sufficient for environmental surveys.

Finally the developed comprehensive method was applied to real sediment samples in Tokyo Bay. As a result, we detected 195 SVOCs in

the sediments; the sum of concentrations of compounds detected varied from 6095 to 39 140 μg/kg dry wt. From the kinds of the detected chemicals as well as their concentration patterns, we were able to obtain a more holistic pollution picture in Tokyo Bay. Since their concentrations increased with proximity to the innermost part of the bay, their sources seem to be mainly sewage treatment plants (STPs) and rivers flowing to this area. Additional confirmation comes from the nature of the identified pollutants, which are characteristic of chemicals used in households as well as fecal matter, business activities and urban run-off. From these results, it was confirmed that sediments in Tokyo Bay are still polluted with a wide range of chemicals, particularly domestic chemicals, despite nearly 100% of wastewater from household and business activities being treated by STPs, indicating that the developed method using the AIQS-DB is a useful tool for grasping a whole pollution picture of the environment.

In addition, it seems to be the most suitable method for confirming the safety of the environment after environmental accidents and natural disasters because a large number of chemical substances can be measured rapidly at relatively low cost. Moreover, the whole pollution picture, which is difficult to obtain by conventional methods, is also useful to find emission sources in survey areas.

Keywords: GC-MS, SVOCs, Simultaneous analysis, Tokyo Bay, AIQS-DB

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Acknowledgements

The thesis would not have been possible without the support and collaboration of my leaders, colleagues, family members and friends.

I would like to express my deepest gratitude to my supervisor Prof. Dr. Kiwao Kadokami for his guidance, support throughout the research and writing this dissertation. Moreover, the encouragements of professor Kadokami gave me more confidence in my accomplishment of the thesis

I greatly appreciate the support from my institute -- Environmental Monitoring Center of Ningbo. Especially, Thanks for my leaders for giving me such an opportunity. And I also thank my colleagues for helping me so much!

I would like to thank the members of the Kiwao Kadokami lab for greatly enhancing my learning experience over the past three years.

I also thank the international supporting services teachers who help me so much and the students who are in the same office with me, for their assistance.

I am very thankful for my parents, family and friends who have supported my decisions and encouraged me in all aspects of my life.

Finally, I would specially like to express heartfelt thanks to all my family, for understanding, support to my study in Japan.

Kitakyushu, September 2014

Abbreviations

AIQS-DB: Automated Identification and Quantification System using a Database

DDTs: Dichlorodiphenyltrichloroethane and its metabolites

EDCs: Endocrine Disrupting Chemicals

ERL: Effects Range Low

ERM: Effects Range-Medium

GC : Gas Chromatography/Gas Chromatograph

GC/MS: Gas Chromatography/Mass Spectrometry

GC-MS: Gas Chromatograph-Mass Spectrometer

HCHs: Hexachlorocyclohexanes

HMW: High Molecular Weight

LLE: Liquid-liquid Extraction

LMW: Low Molecular Weight.

Log Kow: Octagon-water Partition Coefficient

MCs: Model Compounds

MS: Mass Spectrometry/Mass Spectrometer

NaCl: Sodium Chloride

OCPs: Organochlorine Pesticide

POPs: Persistent Organic Pollutants

PPCPs: Pharmaceutical and Personal Care Products

PAHs: Polycyclic Aromatic Hydrocarbon

PCB: Polychlorinated Biphenyls

PCS: Performance Check Standards

RSD: Relative Standard Deviation

SVOCs: Semi-volatile organic compounds

SIM: Selected Ion Monitoring

STPs: Sewage Treatment Plants

SPE: Solid-phase Extraction

TIM: Total Ion Monitoring

1 Introduction

1.1 Chemical substance

For the past few decades, industrialized societies have been both developing new chemical substances and producing large quantities of existing chemicals every year. These chemicals serve many useful purposes and are to be found in all aspects of modern life. They have contributed and continue to contribute significantly, to the improvement of human health, welfare and lifestyle. However, many chemicals are released into the environment during production, transportation, utilization, or disposal, and once there produce harmful side effects, particularly on human health and ecosystems, through the persistence of significant residues in the environment, or by bioaccumulation (bioconcentration) in food chains. Environmental pollution issues, such as organo-mercury and PCB pollution, groundwater pollution with trichloroethylene and tetrachloroethylene, marine pollution by organo-tin compounds, and dioxin emissions from garbage incineration, are not just scientific problems, but have also become significant social issues.

The recent increase in the number of chemicals produced has seen a concomitant increase in the range of related environmental problems. Environmental pollution by harmful chemicals is of concern not just to Japan, but also to many countries worldwide. At UNCED (the United Nations Conference on Environment and Development held in Rio de Janeiro in June 1992) it was determined in “Agenda 21” that the management of harmful chemicals in the environment is an international theme, and the IFCS (the International Forum of Chemical Safety) was established as an inter-governmental forum in an effort to provide active international follow-up to the Conference’s resolutions.

In December 1994, concern over chemicals threatening the maintenance or preservation of the environment saw the formulation of

Japan's "Basic Environment Plan." This Plan, based on the "Basic Environment Law" (November 1993) set out as policy that a knowledge of the environmental risk posed by chemicals was one of the basic requirements for environmental preservation. The Plan aims to both quantitatively evaluate environmental risk, and at the same time eliminate the risk by several means e.g. in order to decrease environmental risk during the production, use, and disposal of chemicals, the Plan set out (i) regulations for the manner and methods of discharge of harmful chemicals, (ii) regulations for the management of chemical production and use appropriately to the degree of harmfulness (toxicity) of the chemical, (iii) regulations for the development and spread of substitute technology and products, and (iv) regulations for the appropriate treatment of recovered harmful chemicals.

In order to be able to control and manage appropriately the environmental risk posed by chemicals (decrease the risk), it is first important to determine and quantitatively evaluate the environmental risk. Such risk evaluation is based on a chemical "risk assessment" (an evaluation of the toxicity of the chemicals in question to humans or ecosystems) and an "exposure assessment" (an evaluation of the degree of chemical exposure experienced by humans or ecosystems based on determinations of the environmental concentrations of the chemicals).

After passing the Resolution accompanying the Law Concerning the Examination and Regulation of Manufacture etc. of Chemical Substances (the Chemical Substances Control Law) in 1973, the Japanese Parliament (the Diet) amended the law to make the national government perform a safety check of chemicals produced in Japan or imported, and in use at that time (of which over 20,000 were listed). This was followed by the Environmental Agency beginning a survey of residues of these chemicals in the general environmental. Later the objectives of these investigations

were expanded to include newly registered chemicals and unintentionally produced chemicals (by-products or contaminants). While the Chemical Substances Control Law, by controlling production (or import) and regulating the use of harmful chemicals to control their environmental toxicity and residues, is an important method for environmental risk management, and investigations into the actual levels of environmental contamination by chemicals provides the type of important basic information required for exposure evaluation. Furthermore, such investigations provide, and are expected to provide, information required in the performance of other environmental risk management functions, such as discharge regulation. The Chemical Substances Control Law was enacted in October 1973 amid concern over environmental pollution by PCBs, and enforced from April 1974. According to the Chemical Substances Control Law new chemicals were to be investigated before production or import (pre-examination of new chemicals) if the chemicals were (i) chemically inert under natural conditions, (ii) if they were easily accumulated in the living tissue (high bio-accumulation potential), (iii) if they have posed a significant risk to human health when ingested / absorbed on a continuous basis (chronic toxicity), and (iv) to be registered as 'first grade' and the production, import, and use etc. regulated, specified chemicals having all these characteristics. By the end of December 1995, 5,879 new chemicals (4,404 produced locally, 1,475 imported) were reported, of which the safety of 4,679 chemicals (3,591 produced locally, 1,088 imported) had been investigated.

Existing chemicals are investigated by three government organizations: chemical decomposition by microorganisms and bioaccumulation in fish and other marine products is investigated by the Ministry of International Trade and Industry (MITI), human toxicity is investigated by the Ministry of Health and Welfare, and general

investigations into actual environmental residue levels and ecological effects are performed by the Environmental Agency. By December 1996, nine chemicals had been designated as Class 1 Specified Chemical Substances -- PCBs, HCBs, PCNs, aldrin, dieldrin, endrin, DDT, chlordanes, and bis(tributyl tin)=oxide.

The Chemical Substances Control Law was revised in May 1986 (enforced from April 1987) after detection of trichloroethylene contamination of groundwater in Japan. This revision covered the production and import of Designated Chemical Substances which do not significantly bioaccumulate, are slightly degradable and have indeterminate chronic toxic effects. Where there were strong concerns over damage to human health caused by environmental contamination by such chemicals, the chemical manufacturers were required to investigate and report chemical toxicity data. If such chemicals showed significant toxicity, their production and import were regulated. To date 257 chemicals, such as chloroform, 1, 2-dichloroethane, has designated as Designated Chemical Substances. Since April 1989, tetrachloromethane, tetrachloroethylene, and trichloroethylene were designated as Class 2 Specified Chemical Substances, now 23 of such Class 2 Specified Chemical Substances are listed.

In 1974, after enactment of the Chemical Substances Control Law, the Environmental Agency began the survey for chemical substances (determining the real state of the environment), and other related projects. In order to systematically investigate the large number of chemicals, a temporary toxic substances list was prepared based on known human health effects. Chemicals on the list and those that received priority for investigation (i) are highly toxic and are regulated under the Law, (ii) are known to be difficult to degrade (persistent chemicals), and (iii) have

structures similar to PCBs, or are industrial substitutes for such chemicals.

As the number and types of matrices under investigation have increased, new chemical analytical methods have had to be developed for most chemicals. As a result, since 1977 research has been classified into three categories, namely the development of chemical analytical methods, general environmental research, and highly-detailed specific environmental research projects. Research bodies were organized with local government environmental research institutes to these research areas.

As such research was systematized, the first general investigation of chemical environmental safety was conducted from 1979 to 1988 (first 10-Year Plan) using as its starting point in the above mentioned temporary toxic substances list. Since 1988, a second general investigation of chemicals environmental safety has been conducted (second 10-Year Plan). Hereafter follows a summary of chemical monitoring in Japan.

1.2 Semi-volatile organic compounds (SVOCs)

A semi-volatile organic compound (SVOC) has a boiling point higher than water and may vaporize when exposed to temperatures above room temperature.

SVOCs originate from a broad range of human activities, including combustion for energy production and transportation, industrial processes, and agricultural uses of pesticides. SVOCs are characterized by the increased usage of complex hydrophobic organic chemicals for various purposes such as organochlorine pesticides (OCPs) in agriculture; polychlorinated biphenyls (PCBs) in transformers, capacitors, paints; polybrominated diphenyl ethers (PBDEs) as flame retardants, etc.

Furthermore, phthalic acid esters (PAEs) can nowadays be found everywhere as a result of the usage of plastics. Polycyclic aromatic hydrocarbons (PAHs) have also been detected in all kinds of environmental compartments because of the usage of fossil fuels and of the various combustion processes. In addition, municipal solid was teincineration or uncontrolled burning (e.g., plastics, crop residues), the common practices for waste elimination, has resulted in emissions of polychlorinated dibenzo-p-dioxinsand dibenzofurans (PCDD/Fs) into the environment.

A part of SVOCs are highly toxic, recalcitrant to degradation processes and have a potential for long-range transport [1]. Many casualties among humans and animals by SVOCs such as persistent organic pollutants (POPs) and pesticides have been reported. The health effects can vary greatly from those that are highly toxic, to those which at present, have no known health effects. Many are suspected to be cancer causing in humans and some are known to be carcinogenic in animals and humans e.g benzo(a)pyrene. Other SVOCs have also been shown to be reproductive toxicants based on animal studies e.g. phthalates and have been linked to asthma, allergies, and other bronchial irritations.

In addition, recently it has been newly reported that some SVOCs alter the activity of hormones in humans and wildlife and are therefore known as endocrine disrupting chemicals (EDCs). EDCs are suspected to contribute to the occurrence of neurodevelopment and behavioral problems (e.g., mental retardation or attention deficit disorder), reproductive abnormalities (e.g., decreased fertility or hypospadias), metabolic disorders (e.g., obesity, diabetes), and cancer (e.g., breast, prostate, and testicular cancers).

PAHs contain two or more fused benzene rings and are one of the most important classes of environmental pollutants. PAHs have been

detected at varying levels throughout the world, even in pristine places far from where they were generated such as high-altitude lake sediments, arctic and snow[2]. PAH are generally major components of the total extractable organic matter present in sediments. Due to the persistent, mutagenic and carcinogenic characteristics of PAHs [3], some of them are on the USA list of priority pollutants. PAH contamination has been attracting considerable scientific and political interest due to its potential impact (mutagenic and carcinogenic) on human health.

Organochlorine pesticides (OCPs) are insecticides composed primarily of carbon, hydrogen, and chlorine. Most of them break down slowly (persistent) and can remain in the environment long after application and in organisms long after exposure. Nine of the Stockholm Convention POPs are OCPs: aldrin, toxaphene, DDTs, chlordanes (CHLs), dieldrin, endrin, heptachlor, mirex and hexachlorobenzene, which is also classified as an industrial chemical [4].

OCPs represent an important group of POPs that have caused worldwide concern as toxic environmental contaminants [5, 6, 7 and 8]. Many POPs are believed to be possible carcinogens or mutagens and are of considerable concern to human and environmental health. Although most of these compounds are no longer in use, the persistence of many OC compounds in the environment has prompted continued studies aimed at evaluating environmental quality for wildlife and humans [9].

PCBs are a group of toxic organochlorine compounds. They are an important group of POPs exhibiting significant toxicity and of widespread occurrence in the environment[10]. PCBs were first synthesized in 1881 and widely used in commercial products and industries starting the 1930s[11]. PCBs are synthesized by a number of processes, including direct substitution on the performed biphenyl system,

phenylation of aromatic substrates and other aryl condensation reactions [12].

PCBs are suspected human carcinogens and may cause a serious health problem due to their persistence in the environment, their potential for chronic toxicity and their accumulation in human tissues[10, 13]. Therefore, the use and the production of PCBs have been prohibited by the Stockholm convention. However people and wildlife may still be exposed to these hazardous chemicals. Capacitors and transformers containing PCBs are still in use and some manufactured equipment containing PCBs may still be imported. Workers can be exposed to PCBs when dismantling or servicing older capacitors and transformers or when cleaning up spills or leaks. Thus it is necessary to survey the quantities and temporal trends induced by PCBs as they are still of global concern.

Steroid alcohols, also known as sterols, are components of eukaryotic cells which are distributed widely in organisms and can be found in the fat storage areas of organisms [14, 15 and 16]. Sterols are hydrophobic compounds and as they are insoluble in water, they tend to settle and be absorbed by sediment [17]. Furthermore, sterols have a long residence time in the environment and do not degrade over a short period of time, if compared with other biological compounds such as amino acids and carbohydrates [18, 19]. The major sterols that can be found in the environment are cholesterol and its epimer, cholestanol, coprostanol and epicoporosanol, and phytosterol [20]; their potential sources are estuarine phytoplankton and zooplankton, terrestrial plants and sewage[21]. The most common sterol biomarkers used for establishing organic matter sources are marine markers (cholesterol), phytoplankton (cholest-5, 22-dien-ol, brassicaterol, dinosterol), terrestrial (β-sitosterol and ergosterol) and sewage (5β-coprostanol) [22, 23].

Corprostanol, produced from the hydrogenation of cholesterol by

bacteria in the digestive system, is the most abundant sterol in human feces [24]. In aquatic environments coprostanol is microbially degraded under aerobic conditions. Reported half lives are generally < 10 days at 20□. However, coprostanol is hydrophobic and associated with particulate matter in sewage and water columns[25] and therefore, coprostanol is readily incorporated into bottom sediments, where it can be preserved for a long time under anaerobic condition. It had been reported that coprostanol in anoxic sediments is persistent, and no significant degradation was observed for 450 days at 15□. Thus, coprostanol has been proposed as a powerful molecular marker for fecal pollution monitoring and had been applied to several environmental matrices [26, 27, 28 and 29].

1.3 Sediment pollution

With the rapid development of social economy, the humans produce more and more organic substances. And some organic substances such as PCBs, OCPs are frequently found in the marine environment; they have a particular affinity for suspended organic matter which will easily sink to the ocean bottom. Pollutants dissolved in water are expected to partition into the bottom sediments at a rate controlled by equilibrium constant between water and sediments. Bottom sediments are not the ultimate sink because contaminants may return to the water compartment again once water concentrations change or if sediments are re-suspended in the water column. Nevertheless, most persistent organic pollutants are lipophilic and accumulate in sediment much more than in other compartments.

Contaminant sources to sediments are known to include sewage outfalls, industrial effluents, urban runoff, rivers and atmospheric deposition. Because many organic pollutants such as POPs are not easily degraded and persist in the long time, these contaminants accumulated in

the sediments usually have potential to give adverse effects on aquatic organisms. Sediments, particularly estuarine sediments, are one of the major sinks for these contaminants in the aquatic environment [30]. The study of sediments is an important step in mapping possible exposure pathways to various aquatic organisms, since contaminants in the sediments may be bioavailable to sediment dwelling organisms. Furthermore, dated sediment profiles have been used to estimate the historical deposition of PCBs and other organic pollutants in the river bed impacted by local and regional sources.

Water pollution by organic chemicals has been reduced in recent years by the implementation of various local and national regulations. However, in many cases the quality of marine sediments has not improved, in part because of the slow degradation and dissipation of chemicals that accumulated in sediments prior to these regulations. Consequently, under some circumstances, previously contaminated sediments are a continuing, secondary source of water pollution causing adverse effects to resident benthos and other aquatic organisms. This impact can occur directly through food chain transfer, or indirectly through changed prey behavior or biodiversity. Managing the effects of such contaminants ultimately requires information on chemical concentrations.

So it is necessary to survey the pollutants in the sediments. Toxic chemicals are monitored or surveyed in many countries, and gas chromatography/mass spectroscopy (GC/MS) is the most frequently used analytical technique because of its high sensitivity, selectivity, and flexibility, even for monitoring trace amounts of chemicals. However, before actual samples can be tested, standards of target substances must be analyzed for the determination of retention times and the preparation of calibration curves, which are often affected by subtle differences in

GC–MS conditions. The necessity for standards restricts the number of chemicals that can be simultaneously analyzed by GC/MS. So a novel gas chromatography/mass spectrometry (GC/MS) database for automated identification and quantification of micro-pollutants in environmental (AIQS-DB) were developed by Kadokami et al in 2005[31]. A particular advantage of the database system is that analysts get a comprehensive picture of chemical pollution ensamples, which is difficult by conventional methods. In order to analyze the sediment samples, we develop the comprehensive analytical method for SVOCs in sediment samples using a gas chromatography/mass spectrometry database system.

1.4 Gas chromatography/mass spectrometry (GC/MS)

1.4.1 Gas chromatography (GC)

Gas chromatography (GC) is normally used for measurement of thermally stable substances that are gases at normal temperatures or are vaporized by heating. Inert gases such as nitrogen or helium are used as a carrier gas and are flowed through a column packed with the stationary phase. A mixture is separated in a short time through differences in the interaction of substance between the stationary phase and the carrier gas. The stationary phase is called the liquid phase when it is a liquid, and partitioning between the carrier gas and liquid phase separates the mixture. This method is also known as gas–liquid chromatography (GLC). Separation columns are classified as either packed columns, which are 2–3 mm in inner diameter and packed inside with fine particles, or capillary columns, which are less than 1 mm in inner diameter and coated on the inside with a liquid phase. The latter is called capillary GC. The separation capacity of the capillary column is markedly better than the packed column, and it is generally used for environmental analysis.

The GC main unit is broadly divided into the injector through which the sample is injected, the column that separates the sample, the oven in which the column is placed, and the detector that detects the components that are separated. Carrier gas is supplied to the GC from a cylinder via a pressure regulator. In addition, a data processing unit processes output data from the detector. The capillary GC is an apparatus capable of measuring ultratrace components contained in complex mixtures such as environmental samples. In order to effectively utilize this capability and obtain precise results, the followings are important: (1) High reproducibility of retention time (fine control of oven temperature); (2) Injected sample reliably enters the column (sample is neither adsorbed to nor broken down in the injector, nor is it discharged to outside of the apparatus); (3) Target component adsorption and breakdown does not occur in the column, and the required separation capacity is obtained; (4) The detector is of high sensitivity and high selectivity, and sensitivity is stable over a wide dynamic range (calibration curve is linear across a broad range); and (5) There is no contamination from carrier gas or other parts.

1.4.2 Mass spectrometry (MS)

Mass spectrometry (MS) is a method whereby various means are employed to ionize sample molecules under vacuum or under atmospheric pressure, and the ions that form are introduced into an analyzer under high vacuum and separated according to their mass-to-charge ratio (m/z) either by electrical or magnetic effects, and then detected. Identification of a substance is possible because the ions that are formed reflect the molecular mass and structure of the substance. In addition, by detecting characteristic ions in the substance, the substance can be quantified with high selectivity and high sensitivity. In particular, by combining MS with gas chromatography (GC) or liquid

chromatography (LC) and undertaking mass analysis after separating the various components of the sample, target substances in complex mixtures such as environmental samples can be positively identified and assayed. For this reason, GC/MS and LC/MS are the most effective methods for measuring trace chemical substances in the environment and foods.

A mass spectrometer comprises a sample introduction section (chromatograph, etc.), an ionization section, a mass separation section, a detector, a vacuum evacuation unit that preserves the vacuum in the mass separation section and detector, and a system control and data processing unit that controls all of these sections and processes a vast amount of data. In addition, when ionization is carried out at atmospheric pressure, an interface is located between the ionization section and the mass separation section. In the following, the ionization section, the mass separation section, and the detector are explained respectively.

The ionization methods that can be used are Electron impact ionization (EI) and chemical ionization (CI). EI is the most common means of ionization in GC/MS. Under a high vacuum of 10–8 to 10–5 mmHg, thermo electrons discharged from a filament are accelerated to 70 eV and allowed to collide with molecules of the sample, upon which the energy possessed by each electron is transferred to the molecules and electron is discharged from each sample molecule, resulting in th e formation of a molecular ion (M+). Because this ion possesses surplus energy, cleavage of aligned bonds occurs due to the energy and the positive charge, and fragment ions with smaller mass are formed. EI source can be used for any sample which can be volatilized at the reduced pressures found in the source. It provides a rich spectrum with many different fragments, ideal for identification of molecules. Most extensive libraries of mass spectra compiled of EI spectra. Sometimes, the molecule ion is nearly absent, especially when the molecule is easily fragmented.

This can make determination of the molecular weight more difficult. To obtain spectra with much larger molecular ion peaks, CI is often used.

1.4.3 Gas chromatography/mass spectrometry (GC/MS)

In this method, the components separated by GC are introduced to MS, ionized under vacuum, and detected. It can be applied to volatile substances and those that can be gasified by heating, and on this account, it is most widely used for analyzing hazardous chemical substances. The capillary GC carrier gas flow rate is only approximately 1 mL/min. For this reason, it can be directly introduced into the MS ion source, and by ionization using EI, a mass spectrum as unique as a human fingerprint can be obtained, and unknown substances can be searched for using a mass spectrum database. In addition, substances can be quantified with high sensitivity and high selectivity by using SIM and mass chromatography.

When connecting GC and MS, one must ensure that the high vacuum of the MS is not broken by the carrier gas in the GC column. In a capillary column with a low flow rate, vacuum can be retained even if the column is directly inserted into the ion source. This method is known as direct coupling, and the entire sample discharged from the column is introduced into the ion source. On the other hand, in the case of a packed column or a wide bore column, the sample is separated from the carrier gas by a glass orifice separator (jet separator, enricher). The interconnection between the GC and the MS is made with a fine gap at the joint and the outside is placed under vacuum; because the molecular mass of the carrier gas (He) is much lower than that of the sample, the carrier gas is dispersed and exhausted at right angles to the path of advance. On the other hand, the sample molecules advance directly and enter the ion source, and are thereby separated from the carrier gas. This

method has the effect of concentrating the sample by a factor of 50, and 30–40% of samples in the carrier gas can enter an ion source of the MS.

A mass spectrum is expressed as a bar graph with the vertical axis being ion intensity and the horizontal axis being m/z. The peak with the greatest ion intensity is called the base peak and is normally assigned a value of 100. Other peaks are expressed as relative strengths compared with the base peak. Figure 5 shows an EI mass spectrum of the pesticide chlorpyrifos (C9H11Cl3NO3PS; MW=349).

1.5 The objectives of the present study

As we all know that SVOCs are important to our society, they are necessary for our life. SVOCs are ingredients in cleaning agents and personal care products, and additives to materials such as vinyl flooring, furniture, clothing, cookware, food packaging and electronics. But some SVOCs produce adverse effects (toxicity) to living things including human being. Many casualties among humans and animals by SVOCs such as persistent organic pollutants (POPs) and pesticides have been reported. In addition, recently it has been newly reported that some SVOCs alter the activity of hormones in humans and wildlife and are therefore known as endocrine disrupting chemicals (EDCs).

Organic pollutants such as PCBs and OCPs are frequently found in the marine environment. They have a particular affinity for suspended organic matter that will easily sink to the ocean bottom, pollutants dissolved in water, possibly from the industrial and domestic sewage discharge as well as surface runoff, and are expected to partition into the bottom sediments at a rate controlled by an equilibrium constant between water and sediments. Nevertheless, most persistent organic pollutants are lipophilic and accumulate in sediments much more than in other compartments. Therefore sediments are considered as one of the major sinks for contaminants in the aquatic environment, and researchers

frequently use sediments as a medium for monitoring trace organic contamination in aquatic ecosystems.

To assess the impact on human health and ecosystem caused by SVOCs in sediment, it is necessary that all pollutants' concentrations are determined. On the basis of this recognition, Kadokami et al [31] have developed a novel GC/MS database system which can automatically identify and quantify nearly 1000 kinds of SVOC pollutants. The accuracy and precision using the AIQS-DB system have been confirmed. The database is applicable for various uses, not only in environmental survey but also investigating the causes of environmental pollution incidents and finding a special feature of environmental pollution. A particular advantage of the database system is that analysts get a comprehensive picture of chemical pollution in samples, which is difficult by conventional methods. Micro-pollutants in samples can be analyzed efficiently and inexpensively using AIQS-DB.

Objectives of the study are 1) to develop a novel comprehensive analytical method of SVOCs in sediment samples by utilizing features of the AIQS-DB, and 2) to confirm applicability and usefulness of the developed comprehensive method by analyzing actual sediments. We apply the method to sediment samples collected in Tokyo Bay, which seem to be heavily polluted with a lot of SVOCs due to vast population and industries.

2 Explanations of Automated Identification and Quantification System Using a Database (AIQS-DB)

2.1 Introduction

With the increasing in population and modernization of our lives, both the volume and kinds of anthropogenic chemicals have increased. More than 70,000 chemicals are currently in the usage for various purposes, e.g. agricultural, industrial and domestic use. As a result, various adverse effects, both expected and unexpected, have been reported concerning many chemicals. To take appropriate countermeasures against these effects, it is first necessary to determine the levels of chemical pollutants in the environment, and in foodstuffs etc.

Although the official methods used for the monitoring have sufficient accuracy and precision, a small number of chemical pollutants can be measured simultaneously. Anthropogenic chemicals in aquatic environment are suspected more than the regulated pollutants, and some of them may have adverse effects to aquatic lives. It suggests that the current regular monitoring may be insufficient to guarantee environmental safety. To deal with this situation, some simultaneous analytical methods such as a series of US EPA methods have been developed. However, these methods are still inconvenient and costly, because they usually need the same number of standards as their targets to identify and quantify.

Kadokami et al in 2005 have developed a novel gas chromatography-mass spectrometry (GC/MS) database for automated identification and quantification of micro-pollutants in environmental (AIQS-DB) [31]. This system is composed of three essential data sets-retention times, mass spectra, and calibration curves -- for the chemical analysis. The developed system allows the analyst to measure

nearly 1000 semi-volatile organic compounds without using target standards [31].

2.2 Basic features

2.2.1 Basic principle

The database system consists of the database, which was created with Microsoft Access, and two interface software programs: Software A (trade name: Compound Composer –database registration phase) transfers retention times, mass spectra, and calibration curves in the calibration files of the GC–MS instrument to the database; Software B (trade name: Compound Composer – method creation phase) creates calibration files for the GC–MS instrument from the database.

GC retention times, calibration curves, and mass spectra of nearly 1000 chemicals were registered in the database, and GC retention times of registered chemicals in actual samples were predicted from the retention times of n-alkanes measured before sample analysis. Differences between predicted and actual retention times were less than 3 s; an accuracy of predicted retention times is nearly identical to that obtained by analysis of standard substances. After the retention times were predicted, a calibration file for a GC-MS instrument was created from the predicted retention times, calibration curves, and mass spectra of the registered chemicals. With the resulting calibration file, automated identification of all the chemicals in actual samples was possible without the use of standards, and the identification method was as reliable as standard deviations of 20% or less for determination values could be obtained.

More than 90% of the chemicals in the database could be detected at sensitivity sufficient for all practical purposes (100pg or less). Because each chemical in the database, to which new chemicals can be easily be

added, can be determined in 1 h, micro-pollutants in samples can be analyzed efficiently and inexpensively.

2.2.2 Basic structure

Currently, 947 compounds, including 451 pesticides, are registered (Table 1). These chemicals are known to adversely affect human health, the environment, or both. We selected the chemicals from lists of compounds regulated by environmental protection laws in Japan or the United States and from lists of chemicals detected in environmental surveys by the Japanese Ministry of the Environment [41]. In addition, we registered many pesticides because a positive list system, which prohibits the use of pesticides that are not registered on the list, will be introduced for agricultural chemical residues in food in 2006 in Japan.

Table 1 Types of chemicals registered in the database

Class 1	Number	Class 2	Number
Compounds consisting of CH	194	Aliphatic compounds	31
		Benzenes	14
		Polycyclic compounds	79
		PCBs	62
		Others	8
Compounds consisting of CHO	150	Ethers	11
		Ketones	6
		Phenols	50
		Phthalates	11
		Esters	34
		Others	38
Compounds consisting of CHN (O)	113	Aromatic amines	43
		Quinoline	3
		Nitro compounds	42
		Nitrosoamines	5
		Others	20
Compounds consisting of CHS (NO)	12		12
Compounds consisting of CHP (NOS)	8	Phosphoric esters	8

		Others	0
PPCPs	14		14
Pesticides	451	Insecticides	184
		Herbicides	118
		Fungicides	116
		Others	33
Total	947		

2.2.3 Basic features

After the GC–MS conditions were set, target tuning to meet the criteria for EPA Method 625 [35] was performed. Then the PCS solution was measured, the retention times of n-alkanes were confirmed, and GC–MS performance was determined by evaluating the analytical results in terms of the criteria in Table 2. If all the criteria were met, standard solutions of a chemical were measured to prepare of a calibration curve. Then, a calibration file for the chemical, which consist of mass spectrum, retention time, quantification ion, calibration curve, and so forth, was created according to the conventional method. Finally, the calibration file data and the retention times of two n-alkanes between which the retention time of the chemical were registered in the database with Software A.

Table 2 International standards and performance check standards for GC-MS

Internal standards		
4-Chloritoluene-d4, 1,4-Dichlorobenzene-d4, Naphthalene-d8, Phenanthrene-d10, Acenaphthene-d10, Fluoranthene-d10, Chrysene-d12, Perylene-d12		
Performance check standards		
Chemicals	Check items	Criteria
Decafluorotriphenylphosphine (DFTPP)	Spectrum validity	Mass spectrum of DFTPP should meet the mass intendity criteria of EPA Method 1625
trans-Nonachlor		Mass spectrum of nonachlor should be the same as that of standard
Benzidine, pentachlorophenol	Inertness of GC column and inlet liner	Benzidine, pentachlorophenol, and 2,4-dinitroaniline should be present at their normal responses, and extreme peak tailing should not be visible
4,4’-DDT	Inertness of GC inlet	Degradation of DDT to DDD should not exceed 20%

25 n-Alkanes (n-C9H20 to n-C33H68), n-octanol,2,4-dichloroanline, 2,6-dichlorophenol, Tris(2-chloroethyl)phosphate, decafluorotriphenylphospheine,benzothiazole,2,4-dinitroaniline, benzidine, trans-nonachlor, 4,4'-DDT pentachlorophenol,2,4,6-trinitrotoluene	liner Stability of response	Determined amounts of these compounds should fall within 95% confidence limits of the mean values.

2.2.4 Advantages

2.2.4.1 SVOCs registered in the AIQS-DB in samples are screened and determined by using compound composer software without the use of standards. This method is simple and is easy to perform.

2.2.4.2 The database is applicable for various uses, especially applicable for investigating the causes of environmental pollution incidents, and finding a special feature of environmental polluted by chemicals at a sampling site.

2.2.4.3 A particular advantage of the database system is that analysts get a comprehensive picture of chemical pollution in samples, which is difficult by conventional methods.

2.2.4.4 Even though nearly 947 chemicals are registered in the database, this number is much smaller than the number of chemicals found in the environment, so the current size of the database is insufficient. However, because adding new substances to the database is easy, most of the toxic chemicals to which GC/MS is applicable will be measurable using the database without standard substances in the near future.

2.3 Application status

We applied it to various samples, such as environmental water, effluent water, sediments, soils, and foodstuffs. If chemicals registered in the database were present in the samples, the chemicals could be accurately identified and quantified.

2.3.1 Applications to pesticides residue analysis

AIQS-DB was used for analyzing the residual pesticides in vegetables and fruits in Japan. The results have proved that the sample can be analyzed by the system with high recovery rate and precision. In comparison to the traditional methods, the system has its own characteristics with fast speed, low solvent consumption and automation[32].

2.3.2 Rapid and reliable drug screening

Unique calibration-locking databases were constructed for rapid and semi-quantitative drug screening by gas chromatography-mass spectrometry (GCMS). In addition to the free-drug database of 127 drugs, a drug database with acetylating reagents was constructed to increase the number of detectable compounds in the analysis by GC/MS; 156 drugs, including 30 drugs of abuse, 42 hypnotics and their metabolites, 18 antipsychotic drugs, 15 antidepressants, and 12 antipyretic analgesic agents, were registered with parameters, such as the mass spectrum, retention time, qualifier ion/target ion percentage, and calibration curve using the novel GC/MS software NAGINATA. Among the 40 drugs examined, 38 and 30 drugs were successfully identified at the level of 1 and 0.1μg/ml respectively without using standard compounds. The time required for data analysis was less than 1 min, and semi-quantitative data were also obtained simultaneously. Because new drugs and metabolites can easily be added to the databases, it confirmed that the system is a useful tool in clinical and forensic toxicological screening [34].

2.3.3 Applicable for heating of nitrogen-containing plastics

AIQS-DB was used for analyzing characterization of SVOCs emitted during heating of nitrogen-containing plastics at low temperature [33].

2.3.4 Applicable for environmental samples

AIQS-DB was successfully applied to samples from 11 Japanese rivers, highlighting that (1) the rivers were not severely polluted and (2) the detected chemicals are mainly derived from domestic sources. The results confirmed that the comprehensive method utilizing the AIQS-DB is a powerful and useful tool for evaluating chemical pollution [51].

AIQS-DB was also used for analyzing the sediments of Dokai Bay in Japan, which is known from past surveys to have been polluted with complex mixtures of organic chemicals. The finding indicates that PAHs were those one of the causes of the adverse effects on benthic animals [37]. We analyzed 940 organic chemicals in sediments in Tokyo Bay, using AIQS-DB. From these results, it was confirmed that sediments in Tokyo Bay are still polluted with a wide range of chemicals, particularly domestic chemicals, despite nearly 100% of wastewater from household and business activities being treated by STPs [39]. Approximately 1000 chemicals were screened in surface waters from down streams of Yellow River and Yangtze River using GC/MS coupled with AIQS-DB, 95 pollutants were detected in water samples from Yellow River in Shandong Province and 121 in those from Yangtze River in Jiangsu Province. Combination of GC/MS and AIQS-DB shows high efficiency in regional pollutants survey [35]. Organic pollutants in water samples were extracted by liquid—liquid extraction and semi-quantitative by gas chromatography coupled with mass spectrometry(GC/MS) using compound composer software . The results were simultaneously confirmed by GC/MS under selected ion monitoring mode. Surface water samples were screened and determined for organic pollutants using compound composer software without any standard solutions. The results indicated that total of 47 organic pollutants were detected in the water

samples [36]. Nearly 1000 SVOCs were analyzed by a comprehensive analytical method using an automated identification and quantification system with AIQS-DB. This is the first comprehensive research on wide range SVOCs in the aquatic environment in Vietnam and showed that rivers and canals in the big cities were heavily polluted by a large number of chemicals, especially from domestic sources. The adverse effects of these chemicals on Vietnamese aquatic organisms should be examined in both urban and rural rivers (Duong Thi Hanh., 2011).

2.4 Prospects

A novel gas chromatography–mass spectroscopy (GC/MS) database for identification and quantification of micro-pollutants has been continuously developed. The relative technologies have been also developed. A comprehensive analytical method for semi-volatile organic compounds in water samples by a combination of solid-phase extraction and gas chromatography-mass spectrometry database system was developed [14]. From these results, it demonstrated that SVOCs comprised of a wide range of volatility and polarity were able to be simultaneously extracted and determined by the combination of the developed SPE method and AIQS-DB. A comprehensive analytical method for nearly 1000 SVOCs in sediments has been developed using an automated identification and quantification system with a GC/MS database [41]. From these results, it is confirmed that the method is a useful way to obtain a holistic picture of pollution by SVOC, and is a good tool for rapid screening of chemical pollution in sediments.

There are about 8,000,000 chemicals in the world. More than 70,000 chemicals are currently in use, and the amounts and types of chemicals being produced have been rapidly increasing [43]. Various adverse effects, both expected and unexpected, have been reported for many chemicals [44].

In order to decrease the adverse effects, it is necessary to determine the levels of chemical pollutants in the environment, in foodstuffs, and so on. Even though nearly 947 chemicals are registered in the database, this number is much smaller than the actual number of chemicals found in the environment, so the current size of the database is insufficient. However, because adding new substances to the database is easy, most of the toxic chemicals to which GC/MS is applicable will be measurable using the database without standard substances in the near future.

2.5 Conclusions

A novel gas chromatography-mass spectrometry (GC/MS) database (AIQS-DB) for identification and quantification of micro-pollutants is described. GC retention times, calibration curves, and mass spectra of nearly 1000 chemicals were registered in the database, and the GC retention times of registered chemicals in actual samples were predicted from the retention times of n-alkanes measured before sample analysis. Differences between predicted and actual retention times were less than 3 s, an accuracy that is nearly identical to that obtained by analysis of standard substances. After the retention times were predicted, a calibration file for the GC–MS instrument was created from the predicted retention times, calibration curves, and mass spectra of the registered chemicals. With the resulting calibration file, automated identification of all the chemicals in actual samples was possible without the use of standards, and the identification method was as reliable as conventional methods. When the GC inlet, column, and tuning conditions were adjusted using GC/MS performance check standards, relative standard deviations of 20% or less for determination values could be obtained. More than 90% of the chemicals in the database could be detected at sensitivity sufficient for all practical purposes (100pg or less). Because

each chemical in the database, to which new substances can be easily added, can be determined in 1 h, micro pollutants in samples can be analyzed efficiently and inexpensively.

3 Development of a Comprehensive Analytical Method for Semi-volatile Organic Compounds in Sediment Samples Using a Gas Chromatography-mass Spectrometry Database System

3.1 Introduction

Although it is suspected that hundreds of thousands of chemical substances of anthropogenic origin exist in the environment, the number of chemicals that are regularly examined is very limited. In Japan, only 53 substances, and in China, only 43 substances in the environmental standards are monitored regularly in the aquatic environment, which is not enough to evaluate the safety of the environment and to protect aquatic life and human health; It is particularly important is to cope with pollution caused by accidents and natural disasters, such as earthquakes. In such cases it is necessary to analyze as many chemicals as possible quickly as it can. Historically, when assessing large numbers of organic substances, many analytical methods have to be employed, which are time consuming and requires huge monetary expenditure. Preliminary screening of samples using rapid assessment tools is thus an increasingly attractive prospect for water environment managers. Consequently, Kadokami has developed AIQS-DB [31,45] that can determine the concentrations of nearly 1000 SVOCs. In addition, Miyazaki has confirmed that the accuracy and precision of measurements using the AIQS-DB system are almost the same as that obtained by the conventional internal standard methods (except for polar substances) [23].Consequently, we have developed comprehensive analytical methods for various environmental substrates by making full use of the AIQS-DB. A combination of solid-phase extraction and the AIQS-DB was successfully developed for water samples except, again, for a range of polar substances [39]. In the present study, we developed a comprehensive

analytical method for sediment samples, and evaluated the performance and the effectiveness of the method by analyzing sediments collected in Tokyo Bay in Japan.

3.2 Experiment

3.2.1 Target compounds and reagents

The AIQS-DB consists of 914 SVOCs (Appendix 1). For the most part, these chemicals are regulated by the environmental protection laws in Japan or the United States, and were detected in an environmental survey conducted by the Japanese Ministry of the Environment (MOE) [46]. The 914 chemicals include almost all of the pesticides that can be currently measured by a GC, some pharmaceuticals and personal-care products (PPCPs), and some targeted groups, e.g. compounds eluted from tires [47], such as benzothiazoles. In a pretreatment procedure examination of liquid–liquid extraction (LLE), clean-up and overall recovery, we used 119 model compounds (MCs, Table 6) that are representatives of SVOCs in the AIQS-DB, and consist of chemicals with wide range of physico-chemical properties. We also used 38 surrogates (deuterium-labeled internal standards, Table 3) for examining any problems in analysis. The model compounds and surrogate compounds were obtained from Wako Pure Chemical (Osaka, Japan), Kanto Chemical (Tokyo, Japan), Hayashi Pure Chemical (Osaka, Japan), and Cambridge Isotope Laboratories, Inc. (Andover, MA, USA).Solvents were of pesticide residue analysis grade (Wako Pure Chemical, Japan).

Table 3 Surrogate compounds for development of a comprehensive method

	Surrogate
Sr1	2-Fluorophenol
Sr2	Phenol-d5
Sr3	Bis(2-chloroethyl)ether-d8
Sr4	2-Chlorophenol-d4
Sr5	1,2-Dichlorobenzene-d4
Sr6	Acetophenone-d5
Sr7	4-Methylphenol-d8
Sr8	Nitrobenzene-d5
Sr9	2-Nitrophenol-d4
Sr10	2,4-Dichlorophenol-d3
Sr11	4-Chloroaniline-d4
Sr12	Quinoline-d7
Sr13	2-Fluorobiphenyl
Sr14	Dimethylphthalate-d6
Sr15	4-Nitrophenol-d4
Sr16	2-Aminonaphthalene-d7
Sr17	Fluorene-d10
Sr18	Diphenylamine-d10
Sr19	N -Nitrosodiphenylamine-d6
Sr20	1,2-Diphenylhydrazine-d10
Sr21	Benzophenone-d10
Sr22	2,4,6-Tribromophenol
Sr23	Simazine-d10
Sr24	Pentachlorophenol-13C6
Sr25	Dibenzothiophene-d8
Sr26	Anthracene-d10
Sr27	Fenitrothion-d6
Sr28	C20D42
Sr29	Isofenphos oxon-d6
Sr30	Benzidine-d8
Sr31	Pyrene-d10
Sr32	Bisphenol A-d14
Sr33	p -Terphenyl-d14
Sr34	Isoxathion-d10
Sr35	Tris(2-ethylhexyl)phosphate-d51
Sr36	3,3'-Dichlorobenzidine-d6
Sr37	Benzo(a)pyrene-d12
Sr38	C32D66

3.2.2 Analytical procedure

Extraction from sediment was performed with an accelerated solvent extractor (ASE 350; Japan Dionex, Osaka, Japan). After mixing a sample (10 g, wet wt) with 7 g of Hydromatrix (Varian, Palo Alto, CA, USA), surrogates were spiked into the sample. The sample was put in an extraction vessel, and was then extracted with dichloromethane/acetone (1:1) for 30 min at 100℃ at 10.3 MPa. Thereafter, the extract was concentrated to 10 ml using a rotary evaporator, and then the concentrate was added to 200 ml of a 5% sodium chloride solution. The solution was extracted with 30 ml of dichloromethane twice. After dehydration of the extract with anhydrous sodium sulfate, the extract was concentrated to 10 ml. Thereafter, the solvent was changed from dichloromethane to hexane by adding 20 ml of hexane to the concentrate and concentrating the resulting mixture to 1 ml using a rotary evaporator. This procedure was carried out twice for a complete change of the solvent. The hexane concentrate obtained was applied to a silica-gel cartridge (Sep-Pak VAC 2 g/12 ml; Waters Associates, Milford, MA, USA), and separated into 3 fractions by sequential elution with 15 ml each of hexane (Fraction 1), 5% acetone–hexane (Fraction 2), and 30% acetone–hexane (Fraction 3). Fraction 1 was treated with copper powder (reduced copper, granular, super grade; Kishida Chemical, Tokyo, Japan) to remove sulfur. Fraction 3 was passed through an activated carbon column (ENVI-carb; Supelco, Bellefonte, PA, USA) to remove colored substances (e.g. non-volatile pigments) that damage a GC column. Each fraction was concentrated to 1 ml with a rotary evaporator and a nitrogen stream, and then measured by GC/MS (QP-2010Plus; Shimadzu, Kyoto, Japan) after the addition of internal standards (Restek, Bellefonte, PA, USA). Each final test solution was measured by selected ion monitoring (SIM) and total ion monitoring (TIM) simultaneously. The GC/MS conditions for TIM were as described

in Table 4. SIM was applied to PCBs, organic chlorine pesticides, polycyclic aromatic hydrocarbons (PAHs) and sterols; detailed SIM conditions are shown in Table 5.

Table 4 GC/MS conditions for TIM

GC/MS		Shimadzu GCMS-QP 2010
Column		J&W DB-5 ms (5% phenyl-95% methylsilicone) fused silica capillary column, 30 m X 0.25 mm i.d., 0.25 μm film
Temperature		
	Column:	temperature programmed: 2 min at 40 ℃, 8 ℃/min to 310℃, 5 min at 310℃
	Injector:	250 ℃
	Transfer line:	300 ℃
	Ion source:	200 ℃
Injection method:		splitless, 1 min for purge-off time
Carrier gas:		He
Linear velocity:		40 cm/s, constant flow mode
Ionization method:		EI
Tuning method:		target tuning for US EPA method 625
Scan range:		33 amu to 600 amu
Scan rate:		0.3 s/scan

Table 5 Quantification and confirmation ions by SIM

No.	Compound	Quantification ion	Confirmation ion
1	Naphthalene	128	-
2	2-Methylnaphthalene	142	-
3	1-Methylnaphthalene	142	-
4	Acenaphthylene	152	-
5	Acenaphthene	153	-
6	PCB #1	188.05	190.05
7	Pentachlorobenzene	249.9	251.9
8	PCB #3	188.05	190.05
9	Fluorene	166	165
10	PCB #4&10	222	224
11	α-HCH	218.9	216.9
12	Hexachlorobenzene	283.8	285.8
13	PCB #8	222	224
14	PCB #19	255.95	257.95
15	β-HCH	218.9	216.9
16	γ-HCH	218.9	216.9
17	PCB #18	255.95	257.95
18	Phenanthrene	178	-
19	PCB #15	222	224
20	Anthracene	178	-
21	δ-HCH	218.9	216.9
22	PCB #54	291.9	289.9
23	PCB #28	255.95	257.95
24	PCB #33	255.95	257.95
25	Heptachlor	271.8	273.8
26	3-Methylphenanthrene	192	-
27	PCB #22	255.95	257.95
28	2-Methylphenanthrene	192	-
29	9-Methylphenanthrene	192	-
30	PCB #52	291.9	289.9
31	PCB #49	291.9	289.9
32	PCB #104	325.9	323.9
33	Aldrin	262.85	264.85
34	PCB #44	291.9	289.9
35	PCB #37	257.95	255.95
36	Heptachlor epoxide (B)	352.85	354.85
37	Oxychlordane	386.8	388.8
38	PCB #74	291.9	289.9

Table 5 Cont'd

No.	Compound	Quantification ion	Confirmation ion
39	PCB #70	291.9	289.9
40	PCB #95	325.9	323.9
41	Fluoranthene	202	-
42	PCB #155	359.85	361.85
43	trans -Chlordane	372.85	374.8
44	o,p'-DDE	246	248
45	PCB #101	325.9	323.9
46	Pyrene	202	-
47	PCB #99	325.9	323.9
48	cis -Chlordane	372.85	374.8
49	trans -Nonachlor	408.8	406.8
50	PCB #119	325.9	323.9
51	PCB #87	325.9	323.9
52	PCB #81	291.9	289.9
53	p,p' -DDE	246	248
54	Dieldrin	262.85	264.85
55	PCB #110	325.9	323.9
56	o,p' -DDD	235	237
57	PCB #77	291.9	289.9
58	PCB #151	359.85	361.85
59	Endrin	262.85	264.85
60	PCB #149	359.85	361.85
61	PCB #118	325.9	323.9
62	PCB #118	325.9	323.9
63	cis -Nonachlor	406.8	408.8
64	p,p' -DDD	235	237
65	PCB #114	325.9	323.9
66	o,p' -DDT	235	237
67	PCB #188	393.8	395.8
68	PCB #153&168	359.85	361.85
69	PCB #105	325.9	323.9
70	p,p' -DDT	235	237
71	PCB #138&158	359.85	361.85
72	PCB #178	393.8	395.8
73	PCB #126	325.9	323.9
74	PCB #187	393.8	395.8
75	PCB #183	393.8	395.8
76	PCB #128	359.85	361.85

Table 5 Cont'd

No.	Compound	Quantification ion	Confirmation ion
77	PCB #167	359.85	361.85
78	PCB #177	393.8	395.8
79	PCB #202	429.75	427.75
80	PCB #171	393.8	395.8
81	Benzo(a)anthracene	228	-
82	PCB #156	359.85	361.85
83	PCB #201	429.75	427.75
84	Chrysene & Triphenylene	228	-
85	PCB #157	359.85	361.85
86	PCB #180	323.9	325.9
87	PCB #191	393.8	395.8
88	PCB #169	359.85	361.85
89	PCB #170	323.9	325.9
90	PCB #199	429.75	427.75
91	PCB #189	393.8	395.8
92	PCB #208	463.7	461.7
93	PCB #194	429.75	427.75
94	PCB #205	429.75	427.75
95	Benzo(j&b)fluoranthene	252.1	-
96	Benzo(k)fluoranthene	252.1	-
97	PCB #206	463.7	461.7
98	Cholestane	217	372
99	PCB #209	497.7	499.7
100	Benzo(a)pyrene	252.1	-
101	Coprostanol	373	331
102	Epicoprostanol	370	316
103	Cholesterol	301	368
104	Coprostanone	316	231
105	Cholestanol	215	388
106	Indeno(1,2,3-cd)pyrene	276	-
107	Dibenzo(a,h)anthracene	277	-
108	Ergosterol	363	396
109	Campesterol	400	315
110	Benzo(ghi)perylene	276	-
111	Stigmasterol	215	416
112	β-Sitosterol	414	329

3.2.3 GC/MS analysis

The chemical substances registered in the AIQS-DB (Compound composer, Shimadzu, Kyoto, Japan) were identified and quantified by

using a combination of retention times, mass spectra, and internal standard calibration curves registered in the database. In order to obtain accurate results, a GC-MS has to be adjusted to designated conditions that are almost the same as the instrumental conditions when the database was constructed. The results obtained from performance check standards (Naginata criteria sample mix 3: Hayashi Pure Chemical, Osaka, Japan) are evaluated against three criteria [31, 45]: spectrum validity, inertness of column and inlet liner, and stability of response. When the results for performance check standards satisfy the criteria, the difference between the predicted and actual retention times is less than 3 s, and obtained chemical concentrations(excluding some highly polar compounds which are difficult to measure by GC) are comparable to those obtained by conventional internal standard methods[31, 48]. However, in the present study to make sure of quantification, we quantified MCs in extraction tests by LLE, column chromatography and overall recovery tests with the conventional internal standard method instead of quantification by the AIQS-DB.

3.3 Results and Discussion

Simultaneous analysis for a numerous compounds with various physico-chemical properties is quite different from target analysis for a small number of compounds with similar properties. In the latter case, target analysis usually removes matrix and non-target substances as much as possible to improve the detection limits, precision and accuracy of analysis, whereas in the case of simultaneous analysis for a large number of substances, an extraction method should be selected that can extract as many compounds as possible, and its clean-up procedures are selected that remove the minimum substances necessary to prevent interference with the GC measurement. Therefore, we designed analytical procedures

that are as simple as possible: extraction from sediment using a hydrophilic solvent, re-extraction with a hydrophobic solvent and adsorption chromatography to separate substances according to polarity. Although there are several extraction methods for solid samples, such as a Soxthlet or ultrasonic extraction, we chose an accelerated solvent extraction method because of its ability to extract a large number of compounds in a short period of time; the acetone/dichloromethane (1:1) mixture was used as the extraction solvent because this mixed solvent is recommended in US EPA Method 3545A [77]. We selected dichloromethane as a solvent for re-extraction because dichloromethane is the most suitable solvent for wide spectrum SVOC [59, 61]. In adsorption chromatography, we examined three adsorbents: silica-gel, florsil and alumina. The results of recovery of each pretreatment step, LLE with dichloromethane and column chromatography, and overall recovery through all the procedures, are described in the following sections.

3.3.1 Recoveries of chemicals by liquid–liquid extraction with dichloromethane

In the present study, LLE is one of the key analytical procedures because LLE is employed to extract SVOC after the extraction of sediments. We selected dichloromethane as an extraction solvent because it is usually used as a LLE solvent for SVOC, and its recoveries of SVOC have been well known [59-61]. However, there has been no systematic examination on its extraction efficiency of SVOC based on the chemical physic-chemical properties. In this context, we used 119 MCs that were selected from the AIQS-DB as being representatives of the SVOC in the AIQS-DB, and new chemicals that may be registered in the AIQS-DB in the future, to examine recovery by LLE. These MCs (Table 6) were chosen based on a compound category(e.g. see Appendix 1), and

physico-chemical characteristics (such as boiling point, Log Kow, polarity, presence of functional groups, active hydrogen). The extraction efficiencies were examined by adding each 2 μg of MCs to 200 mL of 5% NaCl water. Quantification was done by the internal standard method. Compounds whose octanol–water partition coefficient (Log Kow) are above 1 show more than 60% of recovery (Table 6). However, compounds that dissociate in water, such as pentachlorophenol, had low recovery, even though they may have a Log Kow> 1. In addition, compounds whose Log Kows are low and also have more than one polar functional group also showed low recovery: e.g. 1,4-benzenediol and 1,1,1-trichloro-2-methyl-2-propanol. The substances with low recovery were consistent with substances that are difficult to measure by the GC/MS conditions [48] used in the present study. Consequently, it was confirmed that SVOC in the AIQS-DB can be extracted by LLE with dichloromethane, except for a number of highly polar substances.

Table 6 Model compounds and their recoveries

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7) % RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery %
										Fr.1	Fr.2	Fr.3	
1	Flutolanil	1	$C_{17}H_{16}F_3NO_2$	323	66332-96-5	3.7	83	18	88			X	102
2	1,1,1-Trichloro-2-methyl-2-propanol	2	$C_4H_7Cl_3O$	176	57-15-8	2.03	21	157	9			X	94
3	2-Ethyl-1-hexanol	2	$C_8H_{18}O$	130	104-76-7	2.73	116	127	116		X	X	119
4	1-Octanol	2	$C_8H_{18}O$	130	111-87-5	3	124	23	82			X	118
5	1-Nonanol	2	$C_9H_{20}O$	144	143-08-8	3.77	61	14	110			X	61
6	3-Methoxy-1-butyl acetate	3	$C_7H_{14}O_3$	146	4435-53-4	1.01	113	7	76		X	X	101
7	Bis(2-ethylhexyl)sebacate	3	$C_{26}H_{50}O_4$	426	122-62-3	10.08	90	11	80		X		101
8	3-Chloro-1,2-dibromopropane	4	$C_3H_5Br_2Cl$	234	35407	2.96	77	10	71	X	X		94
9	n-Pentadecane	4	$C_{15}H_{32}$	212	629-62-9	7.71	140	17	100	X			143
10	n-Hexadecane	4	$C_{16}H_{34}$	226	544-76-3	8.25	143	14	102	X			196
11	n-Triacontane	4	$C_{30}H_{62}$	422	638-68-6	15.07	119	9	89	X			116
12	Dibutylamine	5	$C_8H_{19}N$	129	111-92-2	2.82	1	65	0				0
13	Caprolactam	6	$C_6H_{11}NO$	113	105-60-2	0.66	6	192	10			X	2
14	Pyrimethanil	7	$C_{12}H_{13}N_3$	199	53112-28-0	2.84	91	19	85		X	X	96
15	Aniline	8	C_6H_7N	93	62-53-3	0.94	39	36	106			X	34
16	2,6-Dimethylaniline	8	$C_8H_{11}N$	121	87-62-7	1.84	70	27	81		X	X	128
17	3,4-Dimethylaniline	8	$C_8H_{11}N$	121	95-64-7	1.84	34	58	101		X	X	59
18	m-Aminophenol	8	C_6H_7NO	109	591-27-5	0.21	1	155	0			X	2
19	p-Phenylenediamine	8	$C_6H_8N_2$	108	106-50-3	-0.3	11	38	59			X	23

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
20	m-Phenylenediamine	8	$C_6H_8N_2$	108	108-45-2	–0.33	11	38	66			X	52
21	2-Amino-5-chlorotoluene	8	C_7H_8NCl	141	95-69-2	2.27	74	20	79			X	93
22	2,4-Dichloroaniline	8	$C_6H_5NCl_2$	161	554-00-7	2.78	112	20	94		X	X	94
23	2,6-Diaminotoluene	8	$C_7H_{10}N_2$	122	823-40-5	0.16	18	65	41			X	51
24	N-Nitrosodiphenylamine(DPA)	8	$C_{12}H_{10}N_{2O}$	198	86-30-6	3.13	114	12	96		X		88
25	2,4-Dinitroaniline	8	$C_6H_5N_3O_4$	183	1997-2-9	1.84	33	41	91			X	50
26	Benzidine	8	$C_{12}H_{12}N_2$	184	92-87-5	1.34	1	42	106			X	16
27	N-Phenyl-1-naphthylamine	8	$C_{16}H_{13}N$	219	90-30-2	4.2	63	22	88		X		97
28	3,3′-Dichlorobenzidine	8	$C_{12}H_{10}N_2C_{l2}$	252	91-94-1	3.51	19	119	72			X	72
29	4,4′-Methylenebis(2-chloroaniline)	8	$C_{13}H_{12}N_2C_{l2}$	266	101-14-4	3.91	62	49	87			X	84
30	Isoprothiolane	9	$C_{12}H_{18}O_4S_2$	290	50512-35-1	2.88	76	16	91			X	109
20	m-Phenylenediamine	8	$C_6H_8N_2$	108	108-45-2	–0.33	11	38	66			X	52
21	2-Amino-5-chlorotoluene	8	C_7H_8NCl	141	95-69-2	2.27	74	20	79			X	93
22	2,4-Dichloroaniline	8	$C_6H_5NCl_2$	161	554-00-7	2.78	112	20	94		X	X	94
23	2,6-Diaminotoluene	8	$C_7H_{10}N_2$	122	823-40-5	0.16	18	65	41			X	51
24	N-Nitrosodiphenylamine(DPA)	8	$C_{12}H_{10}N_{2O}$	198	86-30-6	3.13	114	12	96		X		88
25	2,4-Dinitroaniline	8	$C_6H_5N_3O_4$	183	1997-2-9	1.84	33	41	91			X	50
26	Benzidine	8	$C_{12}H_{12}N_2$	184	92-87-5	1.34	1	42	106			X	16
27	N-Phenyl-1-naphthylamine	8	$C_{16}H_{13}N$	219	90-30-2	4.2	63	22	88		X		97

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7) % RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
28	3,3′-Dichlorobenzidine	8	$C_{12}H_{10}N_2Cl_2$	252	91-94-1	3.51	19	119	72			X	72
29	4,4′-Methylenebis(2-chloroaniline)	8	$C_{13}H_{12}N_2Cl_2$	266	101-14-4	3.91	62	49	87			X	84
30	Isoprothiolane	9	$C_{12}H_{18}O_4S_2$	290	50512-35-1	2.88	76	16	91			X	109
31	Benzyl chloride	10	C_7H_7Cl	126	100-44-7	2.3	74	10	103	X			87
32	Benzyl alcohol	10	C_7H_{8O}	108	100-51-6	1.1	39	28	102			X	47
33	Hexachlorobenzene	10	C_6C_{l6}	282	118-74-1	5.73	86	10	94	X			89
34	1,2,4,5-Tetrabromobenzene	10	$C_6H_2Br_4$	390	636-28-2	5.13	92	13	95	X			97
35	Triphenylmethane	10	$C_{19}H_{16}$	244	519-73-3	5.37	89	11	86		X		98
36	Biphenyl	11	$C_{12}H_{10}$	154	92-52-4	3.98	85	10	98	X			96
37	2,2′,4,4′,5,5-Hexabromobiphenyl	11	$C_{12}H_4Br_6$	622	59080-40-9	—	97	8	94	X			100
38	Methyl dymron	12	$C_{17}H_{20}N_2O$	268	42609-73-4	3.01	70	26	93			X	92
39	Carbofuran	13	$C_{12}H_{15}NO_3$	221	1563-66-2	2.32	75	21	82			X	90
40	Thiobencarb	13	$C_{12}H_{16}NOSCl$	257	28249-77-6	3.4	91	13	101		X		102
41	Tebufenpyrad	14	$C_{18}H_{24}ClN_3O$	333	119168-77-3	4.61	95	16	84		X	X	105
42	Iprodione	15	$C_{13}H_{13}Cl_2N_3O_3$	329	36734-19-7	3	85	22	86			X	89
43	Trifluralin	16	$C_{13}H_{16}F_3N_3O_4$	335	1582-09-8	5.34	88	13	79		X		82
44	Benfluralin	16	$C_{13}H_{16}F_3N_3O_4$	335	1861-40-1	5.29	114	12	80		X		79
45	Triclosan	17	$C_{12}H_7O_2C_{l3}$	288	3380-34-5	4.76	82	17	72		X	X	84

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
46	Pyriproxyfen	17	$C_{20}H_{19}NO_3$	321	95737-68-1	5.55	82	22	91		X		94
47	Bis(2-chloroethyl)ether	18	$C_4H_8Cl_2O$	142	111-44-4	1.29	79	7	97		X	X	103
48	Benzothiazole	19	C_7H_5NS	135	95-16-9	2.01	80	13	94		X		99
49	Dibenzothiophene	19	$C_{12}H_8S$	184	132-65-0	4.38	89	9	94	X			93
50	Bentazone	20	$C_{10}H_{12}N_2O_3S$	240	25057-89-0	–0.49	0	59	1			X	4
51	Tricyclazole	21	$C_9H_7N_3S$	189	41814-78-2	1.42	14	118	92			X	18
52	Nitrobenzene	22	$C_6H_5NO_2$	123	98-95-3	1.85	77	7	92		X		97
53	3-Chloronitrobenzene	22	$C_6H_4NO_2Cl$	157	100-00-5	2.39	78	3	76		X		88
54	2,4-Dichloronitrobenzene	22	$C_6H_3NO_2Cl_2$	191	611-06-3	3.07	108	9	87		X		95
55	2,4,6-Trinitrotoluene	22	$C_7H_5N_3O_6$	227	118-96-7	1.6	96	16	87		X	X	82
56	Pentachloronitrobenzene	22	$C_6NO_2Cl_5$	293	82-68-8	4.64	84	11	87	X			84
57	2-Nitrophenol	23	$C_6H_5NO_3$	139	88-75-5	1.79	69	15	81		X		90
58	4-Nitrophenol	23	$C_6H_5NO_3$	139	100-02-7	1.91	46	35	59			X	64
59	4-Methyl-3-nitrophenol	23	$C_7H_7NO_3$	153	2042-14-0	2.18	76	27	60			X	64
60	1-Nitronaphthalene	24	$C_{10}H_7NO_2$	173	86-57-7	3.19	110	10	93		X	X	79
61	α-HCH	25	$C_6H_6Cl_6$	288	319-84-6	3.8	93	11	91		X		97
62	β-HCH	25	$C_6H_6Cl_6$	288	319-85-7	3.78	99	9	91		X		99
63	δ-HCH	25	$C_6H_6Cl_6$	288	58-89-9	3.72	99	8	88		X		94

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
64	d-HCH	25	$C_6H_6Cl_6$	288	319-86-8	4.14	100	10	93		X		102
65	Heptachlor	25	$C_{10}H_5Cl_7$	370	76-44-8	6.1	97	12	66	X			82
66	Aldrin	25	$C_{12}H_8Cl_6$	362	309-00-2	6.5	92	9	83	X			108
67	Heptachlor epoxide	25	$C_{10}H_5Cl_7O$	386	1024-57-3	4.98	92	11	93		X		83
68	Captan	25	$C_9H_8Cl_3NO_2S$	299	133-06-2	2.8	100	17	106			X	136
69	trans-Nonachlor	25	$C_{10}H_5Cl_9$	440	39765-80-5	6.35	89	11	93	X			84
70	Dieldrin	25	$C_{12}H_8Cl_6O$	378	60-57-1	5.4	98	11	104		X		100
71	p,p′-DDE	25	$C_{14}H_8Cl_4$	316	72-55-9	6.51	89	7	86	X			108
72	Endrin	25	$C_{12}H_8Cl_6O$	378	72-20-8	5.2	100	13	82		X		85
73	p,p′-DDD	25	$C_{14}H_{10}Cl_4$	318	72-54-8	6.02	81	14	89	X	X		107
74	o,p′-DDT	25	$C_{14}H_9Cl_5$	352	789-02-6	6.79	103	10	92	X			98
75	p,p′-DDT	25	$C_{14}H_9Cl_6$	352	50-29-3	6.91	108	11	92	X			93
76	Phenol	26	C_6H_6O	94	108-95-2	1.46	32	21	70			X	111
77	Diazinon	26	$C_{12}H_{21}N_2O_3PS$	304	333-41-5	3.81	88	12	97		X	X	106
64	d-HCH	25	$C_6H_6Cl_6$	288	319-86-8	4.14	100	10	93		X		102
65	Heptachlor	25	$C_{10}H_5Cl_7$	370	76-44-8	6.1	97	12	66	X			82
66	Aldrin	25	$C_{12}H_8Cl_6$	362	309-00-2	6.5	92	9	83	X			108
67	Heptachlor epoxide	25	$C_{10}H_5Cl_7O$	386	1024-57-3	4.98	92	11	93		X		83

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log Kow[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
68	Captan	25	$C_9H_8Cl_3NO_2S$	299	133-06-2	2.8	100	17	106			X	136
69	trans-Nonachlor	25	$C_{10}H_5Cl_9$	440	39765-80-5	6.35	89	11	93	X			84
70	Dieldrin	25	$C_{12}H_8Cl_6O$	378	60-57-1	5.4	98	11	104		X		100
71	p,p′-DDE	25	$C_{14}H_8Cl_4$	316	72-55-9	6.51	89	7	86	X			108
72	Endrin	25	$C_{12}H_8Cl_6O$	378	72-20-8	5.2	100	13	82		X		85
73	p,p′-DDD	25	$C_{14}H_{10}Cl_4$	318	72-54-8	6.02	81	14	89	X	X		107
74	o,p′-DDT	25	$C_{14}H_9Cl_5$	352	789-02-6	6.79	103	10	92	X			98
75	p,p′-DDT	25	$C_{14}H_9Cl_6$	352	50-29-3	6.91	108	11	92	X			93
76	Phenol	26	C_6H_6O	94	108-95-2	1.46	32	21	70			X	111
77	Diazinon	26	$C_{12}H_{21}N_2O_3PS$	304	333-41-5	3.81	88	12	97		X	X	106
78	Isoxathion	26	$C_{13}H_{16}NO_4PS$	313	18854-01-8	3.73	119	12	100		X		109
79	Edifenphos	26	$C_{14}H_{15}O_2PS_2$	310	17109-49-8	3.48	90	23	80			X	105
80	EPN	26	$C_{14}H_{14}NO_4PS$	323	2104-64-5	4.78	91	13	112		X		95
81	2-Chloro-6-methylphenol	27	C_7H_7OCl	142	87-64-9	2.8	83	11	96		X		94
82	2,6-Dimethylphenol	27	$C_8H_{10}O$	122	576-26-1	2.36	85	10	81		X	X	102
83	3-Chlorophenol	27	C_6H_5OCl	128	108-43-0	2.5	88	7	78			X	96
84	2,6-Dichlorophenol	27	$C_6H_4OCl_2$	162	87-65-0	2.75	77	11	90		X	X	90
85	1,4-Benzenediol	27	$C_6H_6O_2$	110	123-31-9	0.59	14	52	2			X	2

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log K_{ow}[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
86	4-tert-Butylphenol	27	$C_{10}H_{14}O$	150	98-54-4	3.31	88	15	86			X	93
87	2,4,6-Trichlorophenol	27	$C_6H_3OCl_3$	196	1988-6-2	3.69	76	13	89		X	X	100
88	Pentachlorophenol	27	C_6HOCl_5	266	87-86-5	5.01	9	77	41			X	68
89	4-n-Nonylphenol	27	$C_{15}H_{24}O$	220	104-40-5	5.76	332	34	90			X	612
90	Bisphenol A	27	$C_{15}H_{16}O_2$	228	1980-5-7	3.32	53	42	110			X	22
91	Tris(2-chloroethyl)phosphate	28	$C_6H_{12}O_4PCl_3$	284	115-96-8	1.78	84	7	106			X	102
92	Tributyl phosphate	28	$C_{12}H_{27}O_4P$	266	126-73-8	4	94	27	92			X	97
93	Tris(2-ethylhexyl)phosphate	28	$C_{24}H_{51}O_4P$	434	78-42-2	9.49	92	15	108		X	X	93
94	Dimethyl phthalate	29	$C_{10}H_{10}O_4$	194	131-11-3	1.6	102	14	87		X	X	66
95	Dimethyl terephthalate	29	$C_{10}H_{10}O_5$	194	120-61-6	2.25	108	13	101		X		98
96	Naphthalene	30	$C_{10}H_8$	128	91-20-3	3.3	85	8	89	X			91
97	Acenaphthylene	30	$C_{12}H_8$	152	208-96-8	3.94	85	10	94	X			101
98	Acenaphthene	30	$C_{12}H_8$	154	83-32-9	3.92	115	12	95	X			106
99	Fluorene	30	$C_{13}H_{10}$	166	86-73-7	4.18	111	11	98	X			101
100	Phenanthrene	30	$C_{14}H_{10}$	178	1985-1-8	4.46	115	13	93	X			105
101	Anthracene	30	$C_{14}H_{10}$	178	120-12-7	4.45	88	10	92	X			101
102	Fluoranthene	30	$C_{16}H_{10}$	202	206-44-0	5.16	106	15	93	X	X		95
103	Pyrene	30	$C_{16}H_{10}$	202	129-00-0	4.88	97	17	100	X			99

Table 6 Cont'd

No.	Compound	Class[a]	Molecular formula	MW[b]	CAS RN[c]	Log K_{ow}[d]	Overall recovery (n = 7)% RSD,	%	LLE extraction[e] efficiency (n = 2), %	Elution pattern			Recovery[f] %
										Fr.1	Fr.2	Fr.3	
104	Benz(a)anthracene	30	$C_{18}H_{12}$	228	56-55-3	5.76	90	13	108	X	X		95
105	Chrysene	30	$C_{18}H_{12}$	228	218-01-9	5.81	92	12	108	X	X		90
106	Benzo(k)fluoranthene	30	$C_{20}H_{12}$	252	207-08-9	6.11	91	16	98		X		89
107	Benzo(b)fluoranthene	30	$C_{20}H_{12}$	252	205-99-2	5.78	90	12	97		X		84
108	Benzo(e)pyrene	30	$C_{20}H_{12}$	252	192-97-2	6.44	90	10	95	X	X		86
109	Benzo(a)pyrene	30	$C_{20}H_{12}$	252	50-32-8	6.13	90	12	92	X	X		85
110	Indeno(1,2,3-cd)pyrene	30	$C_{22}H_{12}$	276	193-39-5	6.7	96	14	95		X		88
111	Dibenz(a,h)anthracene	30	$C_{22}H_{14}$	278	53-70-3	6.75	65	17	82		X		79
112	Benzo(ghi)perylene	30	$C_{22}H_{12}$	276	191-24-2	6.63	97	12	99		X		90
113	Tefluthrin	31	$C_{17}H_{14}ClF_7O_2$	418	79538-32-2	6.5	97	10	88		X		103
114	Buprofezin	32	$C_{16}H_{23}N_3OS$	305	69327-76-0	4.3	87	15	94		X	X	106
115	m-Terphenyl	33	$C_{18}H_{14}$	230	1992-6-8	5.52	92	9	93		X		101
116	p-Terphenyl	33	$C_{18}H_{14}$	230	92-94-4	5.6	92	10	91		X		100
117	Atrazine	34	$C_8H_{14}N_5Cl$	215	1912-24-9	2.61	79	14	82			X	103
118	Simetryn	34	$C_8H_{15}N_5S$	213	1014-70-6	2.6	52	19	69			X	99
119	Tetraconazole	35	$C_{13}H_{11}Cl_2F_4N_3O$	371	112281-77-3	3.56	71	22	92			X	82

a. 1, Acid amide pesticides; 2, alcohols; 3, aliphatic esters; 4, alkanes; 5, amines; 6, lactam; 7, anilinopyrimidine pesticide; 8, aromaticamines; 9, aromatic carbonic acid pesticides; 10, benzenes; 11, biphenyls; 12, carbaimide pesticides; 13, carbamate pesticides; 14, diazolepesticides; 15, dicarboxyimide pesticides; 16, dinitroaniline pesticides; 17, diphenylether pesticides; 18, ethers; 19, heterocyclic aromaticcompounds; 20, benzothiadiazinone; 21, heterocyclic pesticides; 22, nitrobenzenes; 23, nitrophenols; 24, nitropolycyclic aromatichydrocarbons; 25, organochlorine pesticides; 26, organophosphorus pesticides; 27, phenols; 28, phosphates; 29, phthalates; 30, polycyclicaromatic hydrocarbons; 31, pyrethroid pesticides; 32, thiazinone pesticides; 33, terphenyls; 34, triazine pesticides; 35, triazole pesticides.b. Molecular weight. c. Chemical Abstracts registry number. d. Logarithm of octanol–water partition coefficient. e. Liquid–liquid extraction with dichloromethane. f. No subtraction of blank values.

3.3.2 Examination of clean-up by adsorption chromatography with silica-gel

Since sediments usually contain a large amount of organic matrix and sulfides, a clean-up procedure is essential. In the present study, we adopted adsorption chromatography to separate SVOC according to their polarities and copper treatment to remove sulfur [50]. Although the adopted clean-up procedure cannot precisely separate chemicals, it seems to be suitable for comprehensive analysis, because our large number of target compounds have a wide range of physico-chemical properties. In the present study, we tested three types of adsorbents: silica-gel (Sep-Pak VAC 2 g/12 ml; Waters Associates, Milford, MA, USA), florsil (Waters Sep-Pac VAC 2 g) and neutral alumina (Waters Sep-Pac VAC 2 g). We selected hexane–acetone mixture as elution solvents: 1st fraction, hexane, 2nd fraction, 5% acetone–hexane and 3rd fraction, 30% acetone–hexane; elution volumes are 15 ml each. Elution tests were performed according to the developed method after adding each 2 μg of MCs to 1 ml of hexane. Although the elution patterns obtained with the three adsorbents were almost the same, the numbers of low recovery (below 5%) substances observed using silica-gel was the fewest (5) compared with florsil and alumina (12 and 20, respectively). Lower recovery substances were again polar substances, such as amines, phenols and polar pesticides. From these results, the most suitable clean-up chromatography for sediments was the combination of silica-gel and acetone–hexane solution. The elution position of each compound by silica-gel chromatography is given in Table 6. Since some polar substances that eluted in the Fraction 3 showed low recoveries due to tailing, an increase in the volume of eluent of Fraction 3 may be necessary. In the present study, since we used a commercial product (Waters Sep-Pac VAC 2 g) instead of an open column, some substances, such as 4-n-nonylphenol and n-alkanes (Table

6), were eluted from the cartridges, themselves; thus, when using commercial products, attention must be paid to the levels of substances eluted from the product in method blanks to allow for either background correction of sample data or the removal of such substances from contaminant reporting.

3.3.3 Overall recovery tests

In order to confirm the performance of the developed method, we conducted overall recovery tests. We added each 1 μg of MCs to sediment samples (10g wet wt) collected from the mouth of Dokai Bay in Kitakyushu City (total organic carbon, 0.4%), and then treated the samples according to the developed method. Quantification was performed using the internal-standard method instead of the AIQS-DB measurement. Recoveries of the MCs were calculated after background subtraction, i.e. by subtracting the amounts detected in anon-spiked sample from the amounts detected in the spiked samples. Recoveries of 80% of the MCs were in the 60 to 120% range (Table 6). LLE and/or column chromatography seems to cause low recovery (below 60% of recovery). In the overall recovery tests, the recoveries of 21 out of the 119 MCs were lower than 60%; of these 21 compounds, 10 and 15 compounds also showed low recovery (below 60%) in LLE and silica-gel column chromatography, respectively, which indicates that LLE and silica-gel column chromatography are the causes of their low recovery. Since benzidine is known to be oxidized during analytical procedures [34, 35], some amines also seem to be oxidized, and show low recoveries. Except for these polar substances, 80% of MCs showed good recovery; 70% of MCs showed 60 to 120% of recovery with a variation (repeatability) less than 20%. Some substances, such as n-alkanes, octanol and4-n-nonylphenol, had recovery > 100% due to the effects of

the contamination from a silica-gel cartridge column. From the overall recovery test, it was confirmed that the developed comprehensive method can analyze most SVOCs in sediments except for polar substances. In addition, the results of the overall recovery test provide useful information for predicting the recoveries of substances registered in the database (Table 3). The information is as follows: (1) generally, the overall recovery decreases with decreasing Log Kow, (2) substances whose Log Kows are below 1 cannot be analyzed quantitatively by the developed method, (3) if substances dissociate in water, even if their Log Kow values are larger than 5 (e.g. pentachlorophenol), their recoveries are below 60%, (4) substances that have hydroxyl functional groups and/or amino functional groups show low recoveries, and substances that have a Log Kow below 3 and also have functional groups that contain active hydrogen cannot be recovered by more than 60%, (5) the recovery decreases with the increase in the number of active hydrogen functional groups.

3.3.4 Detection limit

The AIQS-DB uses TIM to measure a large number of SVOCs, and in doing so sacrifices sensitivity. However, current GC/MS instruments have much better sensitivity compared with those of instruments manufactured one decade ago; they can measure mass spectra at pg levels. Detection limits (DL) for the target substances were estimated from concentration ratio (or, ratio of the dry weight of a sample to the volume of a final concentrate), and the instrument detection limit (IDL). For 83% of the chemicals in the AIQS-DB, the DL was ≤4 μg/kg dry wt. In addition, since the GC-MS used in this study can perform SIM/TIM measurement, we measured a sub-set of substances in the database, such as some PCBs, organochlorine pesticides, PAHs and sterols, by SIM.

Since sensitivity by SIM was ten times higher than by TIM, the DL of the PCBs, organochlorine pesticides and PAHs was ≤0.4 μg/kg dry wt.

3.3.5 Analysis of standard reference material

In order to validate the developed comprehensive method, we analyzed a standard reference material (SRM, NIST 1941b, Organics in Marine Sediment; National Institute of Standards and Technology, Gaithersburg, MD, USA). Instrumental measurements were done by TIM/SIM; PAHs were measured by TIM and quantified by the AIQS-DB and PCBs and organochlorine pesticides were measured by SIM and quantified by calibration curves in the AIQS-DB. Although the number of compounds certified in the SRM is much smaller than compounds registered in the AIQS-DB, if analytical results agree with certificated concentrations, analytical results for other compounds (except for highly polar substances) can be assumed be close to true concentrations. The results are given in Table 7. The means of the ratios of results to the certified values was from 32 to 175% (average: 90%, RSD: 29%). Although results for some of the substances differed from the certificated values, the concentrations determined for 33 out of 50 substances fell within ±30% of the certified values, which indicates that although the accuracy of the comprehensive method is slightly lower than that of the conventional methods, it is sufficient for environmental surveys.

3.4 Conclusions

We have developed a comprehensive analytical method for SVOCs in sediment samples with the combination of a pre-treatment method comprising of extraction, column clean-up and AIQS-DB. From the recovery tests of chemicals by LLE with dichloromethane using 119 MCs, it was confirmed most SVOCs, except for polar substances, can be

analyzed quantitatively. The results of examination of clean-up by adsorption chromatography with silica-gel show that the most suitable clean-up chromatography for sediments was the combination of silica-gel and acetone-hexane solution. From the overall recovery test, it was confirmed that the developed comprehensive method can analyze most SVOCs in sediments except for polar substances. From the analysis of a standard reference material (SRM), the accuracy and precision of the developed method are slightly lower than that of conventional methods that were developed for targeted analysis. Although the sensitivity of AIQS-DB using TIM may be insufficient for some chemicals that usually require a high sensitivity, such as POPs, this weak point can be overcome by using SIM/TIM mode.

Table 7 Analytical results of a standard reference material (NIST SRM 1941b)

No.	Compound	1	2	Average(A)	Certificated value (B)	Ratio (A/B), %
1	Naphthalene	355	376	365	848 ± 95	43.1
2	Fluorene	33.7	33.5	33.6	85 ± 15	39.5
3	Phenanthrene	322	303	313	406 ± 44	77
4	Anthracene	120	113	117	184 ± 18	63.4
5	3-Methylphenanthrene	53.1	59.8	56.5	105 ± 13	53.8
6	2-Methylphenanthrene	78.7	73.9	76.3	128 ± 14	59.6
7	Fluoranthene	475	419	447	651 ± 50	68.6
8	Pyrene	420	370	395	581 ± 39	68
9	Benzo(a)anthracene	217	255	236	335 ± 25	70.3
10	Chrysene & Triphenylene	268	293	281	399 ± 36	70.3
11	Benzo(j,b&k)fluoranthene	471	640	556	678 ± 39	81.9
12	Benzo(e)pyrene	472	665	568	325 ± 25	175
13	Benzo(a)pyrene	91.2	138	115	358 ± 17	32.1
14	Perylene	476	661	568	397 ± 45	143
15	Benzo(ghi)perylene	151	231	191	307 ± 45	62.2
16	Indeno(1,2,3-cd)pyrene	292	372	332	341 ± 57	97.3
17	Dibenzo(a,h)anthracene	53.3	54.4	53.8	53 ± 10	102
18	PCB #8	1.55	1.3	1.43	1.65 ± 0.19	86.4
19	PCB #18	1.97	1.99	1.98	2.39 ± 0.29	82.9
20	PCB #28	6.26	6.11	6.18	4.52 ± 0.57	137
21	PCB #44	3.2	2.88	3.04	3.85 ± 0.20	79
22	PCB #49	2.71	2.88	2.79	4.34 ± 0.28	64.4
23	PCB #52	5.34	5.7	5.52	5.24 ± 0.28	105
24	PCB #87	0.83	0.87	0.85	1.14 ± 0.16	74.7
25	PCB #95	2.96	3.18	3.07	3.93 ± 0.62	78.1
26	PCB #99	1.82	1.84	1.83	2.90 ± 0.36	63.2
27	PCB #101	3.49	3.71	3.6	5.11 ± 0.34	70.4
28	PCB #105	1.64	1.55	1.60	1.43 ± 0.10	112
29	PCB #110	3.16	3.33	3.25	4.62 ± 0.36	70.3
30	PCB #118	2.69	3.15	2.92	4.23 ± 0.19	69.1
31	PCB #128	0.76	0.92	0.84	0.696 ± 0.044	120
32	PCB #138&158	3.43	3.55	3.49	3.60 ± 0.28	96.9
33	PCB #149	3.13	3.21	3.17	4.35 ± 0.26	72.8
34	PCB #153&16	6.45	6.31	6.38	5.47 ± 0.32	117
35	PCB #156	0.44	0.54	0.49	0.507 ± 0.090	96.3
36	PCB #170	1.6	1.42	1.51	1.35 ± 0.09	112
37	PCB #180	2.57	3.09	2.83	3.24 ± 0.51	87.4
38	PCB #183	1.17	1.19	1.18	0.979 ± 0.087	121

Table 7 Cont'd

No.	Compound	1	2	Average(A)	Certificated value (B)	Ratio (A/B), %
39	PCB #187	2.84	2.89	2.86	2.17 ± 0.22	132
40	PCB #194	1.1	1.26	1.18	1.04 ± 0.06	113
41	PCB #201	0.64	0.58	0.61	0.777 ± 0.034	78.3
42	PCB #206	2.66	2.44	2.55	2.42 ± 0.19	106
43	PCB #209	7.17	6.95	7.06	4.86 ± 0.45	145
44	Hexachlorobenzene	6.97	6.76	6.87	5.83 ± 0.38	118
45	cis -Chlordane	0.94	0.8	0.87	0.85 ± 0.11	102
46	trans -Chlordane	0.68	0.72	0.7	0.566 ± 0.093	123
47	cis -Nonachlor	0.38	0.44	0.41	0.378 ± 0.053	108
48	trans -Nonachlor	0.3	0.32	0.31	0.438 ± 0.073	70.6
49	p,p'-DDE	3.57	3.78	3.68	3.22 ± 0.28	114
50	p,p'-DDD	4.21	4.29	4.25	4.66 ± 0.46	91.1

4 Confirmation of Applicability and Usefulness of the Developed Comprehensive Method for Sediments in Tokyo Bay

4.1 Introduction

Tokyo Bay is located in one of the most densely populated, industrialized and modernized areas in the world. The bay is an almost entirely enclosed sea surrounded by three prefectures including Tokyo, the capital of Japan with nearly 30 millions of people living there. In addition to the vast population, a lot of factories are located in the coast of the bay with heavy industries; the industrial output of the industrial zone amounts to 20–30% of that of Japan. Tokyo Bay receives a vast volume of wastewater from domestic sources, industrial activities and also agricultural activities. Agriculture in suburban areas of Tokyo is mainly directed towards growing vegetables. Although almost all the wastewater from households, business activities and industry are treated by wastewater treatment plants, large amounts of man-made chemicals still flow into the bay. Many contamination assessments have been carried out in Tokyo Bay, including for polycyclic aromatic hydrocarbon (PAHs) [46], dioxins [76], polychlorinated biphenyls (PCBs) [77], brominated flame reterdants [78], alkylphenols [79], and perfluorinated compounds [80]. But existing studies on chemicals have focused on a small numbers of target substances or a group of substances. It is therefore difficult to obtain a complete picture of chemical pollution of the bay.

We have developed a comprehensive analytical method for semi-volatile organic compounds in sediment samples using a gas chromatography-mass spectrometry database system. Since we can obtain concentrations of wide-variety substances using this method, it is possible to interpret the source activities of pollution (domestic, business, industrial and agricultural activities), the occurrence and sources of major

pollutants, and the occurrence of specific substances and their spatial distribution. If we were to analyze the same large number of substances by targeted analytical methods, we have to do many tests at great cost and effort. And at the same time, in order to confirm that usefulness of the developed comprehensive method, we analyzed real sediments that were taken in sediments in Tokyo Bay in Japan. In this examination, we measured samples by GC/MS-SIM/TIM and identified and quantified the substances in the AIQS-DB.

4.2 Collection of the sediments

Sediment samples used in this study for evaluation of the performance of the developed method were collected from20 sites in Tokyo Bay (Fig. 1) on August 2009 using a Smith-Mcintyre bottom sampler; 19 samples, except for Stn 15 which contained a lot of sea shells, were analyzed. The top 5 cm of sediment was used for chemical analyses. The average sedimentation rates in Tokyo Bay are 0.18 g cm^{-2} y^{-1}[69] and 0.2–0.3 g cm^{-2} y^{-1} (1.19–1.89 cm^{-2} y^{-1}) [71]. Based on these data, our samples correspond to accumulation in the last 3–5 years if no disturbance has occurred.

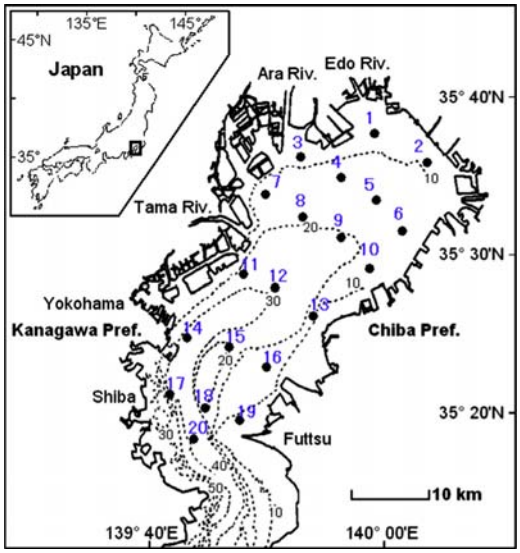

Figure 1 Sampling location.

4.3 Results and discussion

4.3.1 Recoveries of surrogates

In order to ascertain whether this developed analytical method is correct or not, we added 38 surrogates to the Tokyo Bay's sediment samples and examined their recoveries.

Since the target 947 chemicals comprise various physico-chemical groups, 38 substances, which are selected as representatives of all the targets, were examined on the recovery test with dichloromethane extraction.

Table 8 lists average recoveries and RSD of surrogate compounds spiked into sediment samples (n=19). Recoveries of surrogates distribute from 25% to 127%, except for isofenphos oxon-d6 (146%), biphenyl A-d14(136%), tris(2-ethylhexyl)phosphate-d51(131%) and benzidine-d8, 2,4-dinitrophenol-d3, 3,3'-dichlorobenzidine-d6 (which could not be extracted). Recoveries of most of the surrogates (Table 8) were at the same level as those of overall recovery tests. However, some chemicals were of low recovery. It was reported that most compounds having Log

Kow>1 were recovered at 70% or more but hydrophilic compounds (Log Kow<1) could not be recovered sufficiently [51]. But some compounds such as amino compounds and compounds having low boiling points even having Log Kow>1 cannot be recovered sufficiently due to decomposition or evaporation during analysis. Some phenol and amine compounds showed low recovery rate in spite of their relatively high Log Kow, indicating these compounds are difficult to be extracted by liquid-liquid extraction method. Benzidine-d8 and 3, 3‘-dichlorobenzindine-d6 were oxidation during extraction processes. Isofenphos oxon-d6, fenitrothion-d6 and tris(2-ethylhexyl)phosphate-d51 showed high recovery rates, it suggested that these compounds are effected by matrices. The relative standard deviations (RSD) of surrogates were mostly below 21%. From these results, we can determine that this method was applied to most of 1000 pollutants except for highly polar or easy evaporation substances.

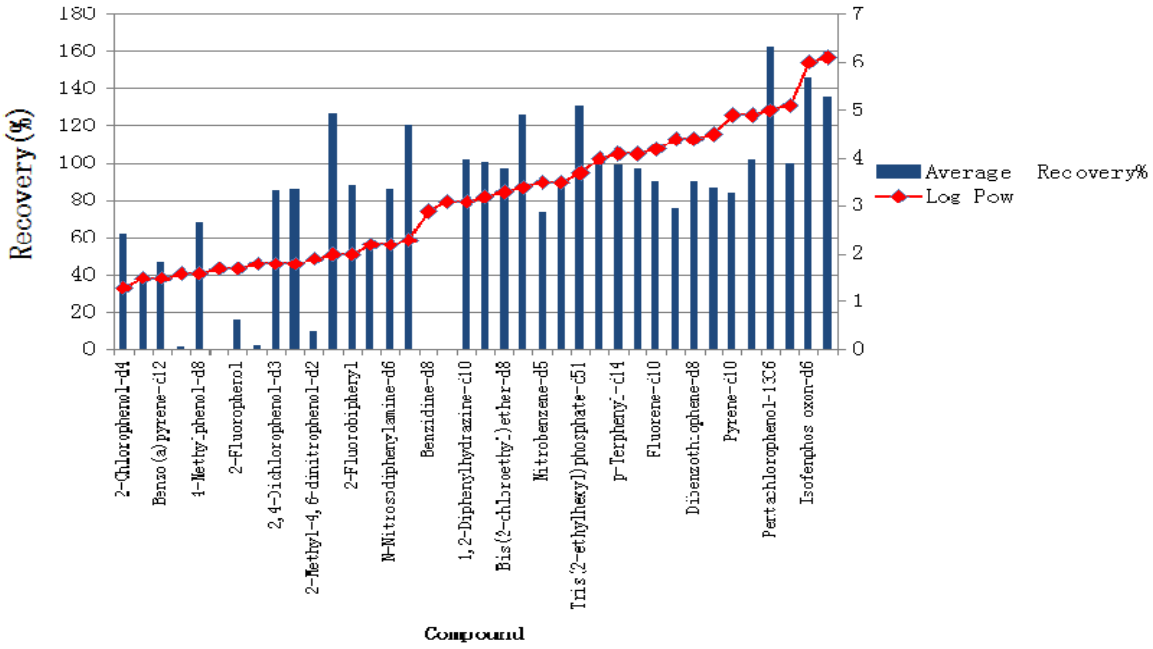

Figure 2 The correlation between recovery rate and Log Kow of surrogate compounds

Table 8 Surrogate compounds and their recoveries

Code	Surrogate	Log Kow	Average Recovery%	RSD %	Recommended surrogate
1	2-Chlorophenol-d4	1.3	62.1	6.9	
2	Phenol-d5	1.5	39.0	16.4	
3	Benzo(a)pyrene-d12	1.5	47.4	26.8	
4	4-Chloroaniline-d4	1.6	1.6	1.9	
5	4-Methylphenol-d8	1.6	68.0	7.8	
6	2,4-Dinitrophenol-d3	1.7	0.0		
7	2-Fluorophenol	1.7	16.2	4.4	
8	2-Aminonaphthalene-d7	1.8	2.4	2.7	
9	2,4-Dichlorophenol-d3	1.8	85.2	8.5	Recovery of acidic substances
10	2-Nitrophenol-d4	1.8	86.1	9.4	
11	2-Methyl-4,6-dinitrophenol-d2	1.9	9.8	25.0	
12	Isoxathion-d10	2.0	127	28.8	
13	2-Fluorobiphenyl	2.0	87.9	6.5	Recovery of acidic substances
14	1,2-Dichlorobenzene-d4	2.2	56.2	6.9	Volatilization loss
15	N-Nitrosodiphenylamine-d6	2.2	85.9	7.5	
16	Fenitrothion-d6	2.3	120	11.0	
17	Benzidine-d8	2.9	0.0		
18	3,3'-Dichlorobenzidine-d6	3.1	0.0		Recovery of basic substances
19	1,2-Diphenylhydrazine-d10	3.1	102	21.2	
20	2,4,6-Tribromophenol	3.2	101	6.7	
21	Bis(2-chloroethyl)ether-d8	3.3	97.3	14.6	
22	4-Nitrophenol-d4	3.4	126	18.5	Recovery of basic substances
23	Nitrobenzene-d5	3.5	73.6	6.4	
24	Anthracene-d10	3.5	89.3	10.7	
25	Tris(2-ethylhexyl)phosphate-d51	3.7	131	12.0	Confirmation of matrix effect
26	Dimethylphthalate-d6	4.0	101	6.9	
27	p-Terphenyl-d14	4.1	99.1	7.5	Validation of analysis itself
28	Quinoline-d7	4.1	97.0	8.9	
29	Fluorene-d10	4.2	90.8	6.3	
30	Acetophenone-d5	4.4	75.8	7.2	
31	Dibenzothiophene-d8	4.4	90.3	8.0	
32	Diphenylamine-d10	4.5	86.5	7.7	
33	Pyrene-d10	4.9	84.0	11.4	
34	Benzophenone-d10	4.9	102	20.4	Validation of analysis itself
35	Pentachlorophenol-13C6	5.0	163	27.8	Recovery of acidic substances
36	C20D42	5.1	100	8.7	
37	Isofenphos oxon-d6	6.0	146	15.6	
38	Bisphenol A-d14	6.1	136	26.7	Comfirmation of matrix effect

4.3.2 Overview of survey results of micro-pollutants

The number of chemicals detected at each site increased towards the innermost part of the bay, from 142 individual substances at Stn 19 to 177 individual substances at Stn 7 (Table 9). A total of 195 substances were detected at least once, belonging to 25 different chemical groups (Table 9; Table 10). Substances indicative of pollution in the bay were sterols and domestic substances that appear to be discharged from STPs and rivers (Table 9 and Table 10). In addition to the number of chemicals, concentrations also increase towards the innermost part of the bay (Table 9). The highest total concentration of chemicals (39140 µg kg dry wt) was found at Stn 4 located in a coastal zone near Tokyo.

Many chemicals are detected throughout the bay, such as antioxidants, fragrances, cosmetics, plasticizers, alkyl phenols, PAHs, organochlorine pesticides (OCPs) and many more (Table 9 and Table 10), indicate they are from multiple sources like rivers, STPs and atmospheric deposition. However, the number and concentrations of industrial chemicals were much lower than those in Dokai Bay [42].

Table 9 Concentrations of chemicals in sediments in Tokyo Bay

Origin	Category/number	Stn1	Stn2	Stn3	Stn4	Stn5	Stn6	Stn7	Stn8	Stn9	Stn10	Stn11	Stn12	Stn13	Stn14	Stn16	Stn17	Stn18	Stn19	Stn20
Agriculture	Insecticides	5.1	384	5.8	6	3.9	4.3	7.2	4.2	3.8	2	2.9	3.7	1.6	1.8	0.54	1.8	0.55	0.16	0.35
	187	10	6	10	10	8	8	10	7	9	4	8	9	4	7	12	4	4	4	4
	Herbicides	0.3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	117	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	Fungicides	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	112	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	Other pesticides	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	36	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	Antioxidants	663	613	1019	1391	813	904	1221	1312	1445	1394	690	1167	857	508	535	621	487	441	503
Business/	8	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Household	PPCPs	349	144	394	958	639	1474	278	276	365	198	310	305	223	106	59	137	233	1	2
/traffic	19	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	1	1
	Cosmetics and fragrances	52	41	103	138	94	105	64	81	88	69	109	86	39	50	33	54	22	39	27
	13	6	6	6	5	7	7	7	7	6	7	7	6	6	6	3	4	4	4	3
	Disinfectants and insecticidal fumigants	5.3	6.1	11	14	25	18	17	14	27	8.9	13	32	6.4	32	4.2	3.1	1.4	1.3	1.5
	2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
	Plasticizers	816	634	1169	5680	1437	2894	1295	4803	1242	1876	5999	941	578	532	78	601	972	337	479
	14	6	6	6	7	7	7	7	7	6	7	7	6	6	6	3	6	6	6	6

Low columns show the number of chemicals.

Origin	Category/number	Stn1	Stn2	Stn3	Stn4	Stn5	Stn6	Stn7	Stn8	Stn9	Stn10	Stn11	Stn12	Stn13	Stn14	Stn16	Stn17	Stn18	Stn19	Stn20

Table 9 Cont'd

	Metabolitesof reterdants	125	203	139	25	230	105	101	85	42	139	19	45	0	0	11	0	0	0	0
	3	1	1	1	1	1	1	1	1	1	1	1	1	0	0	1	0	0	0	0
Business/	Fire retardants	36	22	47	26	47	52	29	30	39	33	20	42	22	13	8	21	30	11	12
Household	13	3	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
/traffic	Compounds leached from tires	71	83	139	385	145	227	205	230	259	134	211	285	135	187	35	194	47	26	39
	21	3	5	3	4	5	6	4	4	4	4	5	4	4	4	1	4	4	3	3
	Fatty acid methy esters	14	21	24	39	30	154	72	311	540	14	29	49	0	25	2.3	5.8	0	0	0
	34	2	1	1	1	2	2	2	2	3	2	1	1	0	1	1	1	0	0	0
	Petroleum	4126	3733	7982	9272	7513	8634	3137	4063	6393	6690	4633	7373	4834	3379	4613	3890	2217	1515	1688
	26	25	25	25	25	25	25	25	25	25	25	25	25	24	24	25	25	25	25	25
	Intermediates for resins	5.3	14	22	2.6	21	8.8	12	215	10	41	10	10	11	4.1	2.6	15	13	1.4	7.7
	2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
	Other substances of domestic origin	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	27	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Industry	PCBs and PCNs	27	25	35	28	23	24	42	29	20	14	20	16	12	15	7.4	4.3	4.1	1.6	21
	86	50	41	43	51	41	49	52	51	50	43	53	51	45	54	50	54	49	39	47
	PAHs	999	1572	2506	1897	1801	1904	2091	1806	2056	1980	2598	2351	1514	1396	329	1547	522	484	485
	50	33	32	33	32	32	32	32	32	32	33	32	33	33	33	33	32	33	32	32

Low columns show the number of chemicals.
Table 9 Cont'd

Origin	Category/number	Stn1	Stn2	Stn3	Stn4	Stn5	Stn6	Stn7	Stn8	Stn9	Stn10	Stn11	Stn12	Stn13	Stn14	Stn16	Stn17	Stn18	Stn19	Stn20

Industry	Intermediates in organic synthesis	57	61	157	157	89	83	104	126	126	100	125	330	149	120	39	310	30	29	21
	96	7	8	7	8	7	9	9	7	8	8	7	7	7	7	7	6	7	7	7
	Solvents	9.9	9.5	16	17	15	19	17	14	15	14	10	13	8.8	4.8	1.7	6.3	8.6	4.7	4.9
	14	4	4	4	4	4	4	4	3	3	4	4	4	4	3	1	4	3	3	2
	Storage and transfer agents	1.9	1.9	5.2	2.9	3.1	4.3	5.3	3	0	2.3	3.1	1.9	1.2	0.8	0.7	0.9	0	0	0.3
	3	2	2	2	1	1	2	2	1	0	1	1	1	1	1	2	1	0	0	1
	Explosives	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	6	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	Other substances of industrial origin	6.1	8.8	12	15	10	16	8.7	2.4	8.4	9.2	5.3	9.3	3.9	2.3	2.1	1	11	0.01	2.9
	40	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	2	1	3
Sterols	Sterols	12795	17536	13579	19073	22409	16706	12626	12219	19117	16059	9867	12796	12516	6760	4288	4030	5020	2882	2599
	10	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9
Other	Others	20166	25116	27392	39140	35366	33304	21368	25695	31862	28835	24817	26015	21040	13345	10335	11730	9917	6348	6095
	1	173	159	163	171	162	174	177	169	169	161	173	170	156	168	168	171	156	142	151
Total 940																				

Low columns show the number of chemicals.

Table 10 Chemicals detected in the 19 sediments in Tokyo Bay

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
1	Bis(2-chloroisopropyl)ether	ND	380	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2	a-HCH	0.09	ND	0.09	0.15	ND	ND	0.19	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3	b-HCH	0.09	0.06	0.11	0.14	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.05	ND	ND	ND
4	g-HCH	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
5	Endrin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.44	ND	ND	ND	0.64	ND	ND	ND
6	Dieldrin	ND	ND	ND	ND	ND	ND	0.55	ND	ND	ND	ND	ND	ND	0.21	ND	0.48	ND	ND	ND
7	*trans*-Chlordane	0.15	ND	0.11	0.76	0.22	0.26	0.11	0.18	0.09	ND	0.09	0.09	ND	0.10	ND	ND	ND	ND	ND
8	*cis*-Chlordane	0.12	ND	0.27	0.40	0.25	0.21	0.12	0.19	0.22	ND	0.07	0.08	ND	ND	ND	0.02	ND	ND	ND
9	*trans*-Nonachlor	0.10	ND	0.39	0.24	ND	0.22	0.06	0.15	0.10	ND	0.05	0.02	ND	ND	ND	ND	ND	ND	ND
10	*cis*-Nonachlor	0.10	ND	0.14	0.05	0.19	0.11	0.08	ND	0.21	ND	0.06	0.05	ND	0.04	ND	ND	ND	ND	ND
11	Heptachlorepoxide(B)	ND	ND	ND	ND	0.22	ND	ND	ND	0.71	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
12	*o,p'*-DDE	0.64	0.99	0.74	0.63	0.23	0.36	0.79	0.42	0.22	0.10	0.27	0.09	0.21	0.14	0.02	0.03	0.07	ND	0.02
13	*p,p'*-DDE	2.97	2.32	3.06	2.77	1.92	2.45	3.98	2.51	1.56	1.45	1.61	1.30	0.95	0.84	0.36	0.38	0.30	0.09	0.17
14	o,p'-DDD	0.43	0.18	0.18	0.35	0.31	0.36	0.65	0.35	0.35	0.15	0.33	0.29	0.12	0.23	0.07	0.11	0.08	0.03	0.09
15	*o,p'*-DDT+*p,p'*-DDD	0.46	0.54	0.70	0.49	0.54	0.35	0.68	0.35	0.34	0.33	0.37	0.32	0.34	0.26	0.10	0.09	0.10	0.03	0.08
16	Pebulate	0.3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
17	*n*-C10H22	30	24	42	46	45	49	26	36	45	36	26	40	25	16	47	18	11	11	13
18	*n*-C11H24	312	325	558	685	511	562	313	408	501	433	326	554	325	210	234	266	157	163	143
19	*n*-C12H26	13	15	32	30	21	23	19	19	25	21	11	33	32	21	48	25	16	15	18
20	*n*-C13H28	99	92	158	214	187	189	101	145	168	128	102	183	118	78	28	92	60	56	50
21	*n*-C14H30	31	42	83	123	60	85	34	32	81	71	40	85	19	6	33	7	8	2	9

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
22	*n*-C15H32	36	31	72	184	53	109	50	42	77	57	41	69	86	56	12	68	46	39	41
23	*n*-C16H34	109	128	235	372	196	247	113	80	218	178	133	202	97	42	160	72	40	29	52
24	*n*-C17H36	496	366	637	1347	684	856	361	492	661	441	367	558	305	239	132	262	209	129	139
25	*n*-C18H38	192	202	362	535	324	379	159	199	359	249	198	345	182	107	69	154	108	72	103
26	*n*-C19H40	206	20	69	563	65	102	39	46	74	38	49	60	33	33	20	33	66	9	9
27	*n*-C20H42	122	164	277	376	248	260	147	191	304	238	168	281	153	98	152	135	130	62	84
28	*n*-C21H44	36	18	61	70	63	81	33	29	50	70	53	85	54	43	67	36	103	11	11
29	*n*-C22H46	246	274	491	381	452	456	153	210	499	143	185	185	101	112	140	139	91	38	84
30	*n*-C23H48	70	55	125	123	114	177	63	46	77	108	69	106	72	52	47	46	142	17	15
31	*n*-C24H50	123	131	232	291	212	272	154	169	216	274	161	250	222	118	299	162	97	59	104
32	*n*-C25H52	168	116	236	165	204	470	105	192	305	308	122	188	138	66	89	104	61	42	25
33	*n*-C26H54	147	115	330	272	347	433	79	158	160	259	144	291	189	152	135	182	52	57	46
34	*n*-C27H56	211	243	431	424	427	512	184	180	300	440	281	462	317	226	213	231	124	70	71
35	*n*-C28H58	163	172	358	465	325	397	89	201	315	355	250	340	254	178	151	204	38	76	98
36	*n*-C29H60	347	325	765	645	684	837	281	241	497	791	483	784	554	396	593	411	247	141	130
37	*n*-C30H62	219	222	504	593	561	573	162	252	378	376	312	390	293	215	226	261	119	104	109
38	*n*-C31H64	422	377	903	798	851	966	345	304	535	953	547	1067	732	481	734	469	181	159	149
39	*n*-C32H66	147	123	460	332	394	88	35	288	197	272	233	265	195	167	350	158	11	40	84
40	*n*-C33H68	177	148	547	222	469	496	75	88	334	442	315	539	337	269	630	347	97	112	98
41	Squalane	347	142	391	954	634	1469	274	273	360	196	307	302	220	104	53	135	230	ND	ND
42	1,4-Dichlorobenzene	5	6	11	14	25	18	17	14	27	9	13	32	6	32	4	3	1	1	1

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
43	Diphenyl ether	ND	ND	ND	ND	2	2	2	ND	ND	1	2	ND	1	ND	ND	ND	ND	ND	ND
44	2,6-Di-*tert*-butyl-4-benzoquinone	30	35	34	27	32	37	47	73	44	68	33	60	44	31	ND	65	28	32	32
45	Anthraquinone	20	13	35	31	31	35	20	20	26	24	37	29	19	18	11	19	11	5	16
46	Acetophenone	1	1	5	4	4	8	7	6	7	2	4	4	1	1	ND	ND	1	ND	ND
47	3- & 4-*tert*-Butylphenol	2	2	3	4	13	6	2	3	16	3	2	3	2	1	ND	7	2	0	1
48	Nonylphenol	125	203	139	25	230	105	101	85	42	139	19	45	ND	ND	11	ND	ND	ND	ND
49	Bisphenol A	5	14	22	3	21	9	12	215	10	41	10	10	11	4	3	15	13	1	8
50	2,6-Di-*t*-butyl-4-ethylphenol	630	576	982	1361	768	862	1172	1236	1385	1323	655	1105	810	476	535	548	457	408	470
51	Bis(2-ethylhexyl)phthalate	666	510	889	5318	1180	2493	1020	3900	897	1468	5695	683	461	429	61	473	771	275	417
52	Butyl benzyl phtalate	19	11	50	40	27	53	28	32	32	74	35	35	12	10	7	10	3	2	8
53	Dicyclohexyl phthalate	ND	ND	ND	3	4	10	4	25	ND	4	6	ND	ND	ND	ND	ND	ND	ND	ND
54	Di-*n*-butyl phthalate	18	14	45	22	73	72	117	703	107	202	47	70	40	47	ND	47	136	11	9
55	Di-*n*-octyl phthalate	18	13	32	35	24	64	37	45	30	25	42	19	8	8	ND	12	6	19	9
56	*cis*-11,14,17-Eicosatrienoic acid methyl ester	10	21	24	39	27	151	67	300	456	14	29	49	ND	25	ND	6	ND	ND	ND
57	Methyl palmitate	ND	ND	ND	ND	ND	ND	ND	ND	21	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
58	Stearic acid methyl ester	4	ND	ND	ND	3	2	4	11	64	0	ND	ND	ND	ND	2	ND	ND	ND	ND
59	Octanol	2	4	4	ND	2	8	2	5	7	2	3	ND	2	3	20	ND	1	2	ND
60	Benzyl alcohol	17	17	38	71	34	38	20	21	32	25	51	23	13	15	3	15	9	12	10
61	Phenylethyl alcohol	ND	ND	ND	ND	ND	ND	ND	14	ND	ND	ND	16	ND	13	ND	15	ND	ND	ND
62	1-Nonanol	4	2	6	7	9	9	2	1	2	4	3	3	3	1	ND	4	ND	20	1
63	2-Ethyl-1-hexanol	13	12	25	24	19	21	14	12	20	17	14	21	11	4	ND	7	8	6	7

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
64	Ethanol, 2-phenoxy-	33	39	62	79	61	71	61	61	72	54	49	80	18	22	ND	25	10	8	16
65	alpha-Terpineol	7	5	16	25	12	6	10	14	14	12	9	10	ND	ND	ND	ND	ND	ND	ND
66	Di(2-ethylhexyl)adipate	83	74	127	239	110	135	74	86	155	86	160	114	45	33	10	51	47	25	28
67	Acetamide, *N*-(2-phenylethyl)-	15	12	29	23	17	13	24	32	32	19	26	26	14	13	35	49	30	16	21
68	2(3H)-Benzothiazolone	ND	19	ND	ND	ND	ND	ND	ND	ND	ND	33	ND	ND	ND	ND	ND	ND	ND	ND
69	2-(Methylthio)-benzothiazol	ND	ND	ND	ND	ND	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
70	2-Acetyl-5-methylthiophene	ND	ND	ND	43	6	6	5	8	8	2	3	3	1	2	ND	1	ND	ND	ND
71	Benzothiazole	ND	0	ND	ND	1	0	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0	ND	ND
72	Dibenzothiophene	6	5	14	15	15	17	17	15	17	11	15	13	ND	ND	4	9	3	3	5
73	2-Methylbenzothiazole	23	12	48	239	60	128	115	129	146	58	99	176	101	150	ND	119	6	2	3
74	Tributyl phosphate	24	20	37	19	29	35	22	24	32	23	15	28	17	10	3	18	13	10	11
75	Tricresyl phosphate	7	ND	ND	ND	ND	6	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
76	Tris(2-ethylhexyl) phosphate	5	1	9	7	18	10	7	6	8	10	5	14	5	3	5	3	17	1	1
77	L-Menthol	2	3	3	4	5	4	4	3	5	2	3	3	3	2	6	2	3	1	2
78	4-Cymene	1	2	3	3	2	4	2	ND	3	0	1	3	2	1	2	1	5	1	1
79	1,2,4-Trichlorobenzene	ND	4	ND	ND	ND	3	5	ND	3	3	ND	ND	ND	ND	1	ND	ND	ND	ND
80	1,2-Dichlorobenzene	1	2	3	3	3	3	2	2	3	2	1	2	1	1	ND	1	0	1	ND
81	Pentachlorobenzene	0.13	0.04	0.36	0.21	0.12	0.14	0.21	0.21	0.13	0.02	0.08	0.10	0.04	0.06	0.01	0.01	ND	ND	0.01
82	Hexachlorobenzene	0.27	0.06	0.36	0.28	0.24	0.19	0.30	0.24	0.36	0.13	0.21	0.15	0.19	0.34	0.05	0.11	0.07	0.01	0.02
83	1,2-Dimethylnaphthalene	4	ND	6	ND	ND	ND	ND	ND	ND	3	ND	7	4	4	2	ND	9	ND	ND
84	1,3-Dimethylnaphthalene	4	3	14	12	8	11	12	11	9	10	11	12	7	7	5	5	12	3	3

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
85	1,4-&2,3-Dimethylnaphthalene	4	2	9	7	7	10	9	8	9	6	9	10	4	7	4	5	21	3	4
86	1-Methylphenanthrene	4	3	7	11	8	10	7	7	9	5	10	6	7	10	3	9	8	3	3
87	1-Phenylnaphthalene	7	6	18	17	9	8	7	10	10	16	8	11	6	5	3	7	2	3	3
88	2,3-Benzofluorene	49	44	163	93	99	90	94	93	94	101	146	118	5	52	8	61	13	15	13
89	2,6-Dimethylnaphthalene	22	7	65	57	40	54	25	31	29	32	33	47	6	23	17	20	16	5	24
90	2-Methylphenanthrene	16	5	12	20	15	26	14	16	13	11	14	15	58	9	4	10	4	2	3
91	2-Phenylnaphthalene	24	19	48	44	43	35	36	38	42	44	49	46	35	26	11	27	11	10	11
92	4,5-Methylene-phenanthrene	19	10	57	45	46	39	19	19	25	16	44	27	8	35	7	42	3	6	3
93	Acenaphthene	4	3	13	10	7	9	9	9	11	9	11	13	33	4	1	6	2	1	1
94	Acenaphthylene	2	2	8	6	6	6	6	7	6	4	9	9	13	4	1	5	2	2	2
95	Anthracene	11	7	25	24	19	23	18	22	24	16	26	25	7	15	6	17	6	6	5
96	Benzo(*a*)anthracene	34	24	109	76	64	56	77	75	87	83	127	98	5	62	7	79	22	26	20
97	Benzo(*a*)pyrene	35	34	112	70	72	61	87	73	88	92	136	109	15	64	8	82	21	23	20
98	Benzo(*c*)phenenthrene	5	4	10	7	8	7	11	7	10	8	12	9	68	11	3	12	4	4	4
99	Benzo(*e*)pylene	107	85	313	234	213	184	264	215	273	271	346	312	72	171	26	212	61	62	56
100	Benzo(*ghi*)perylene	34	31	109	72	76	62	112	86	105	106	121	124	10	67	7	75	21	20	21
101	Benzo(*j*&*b*)fluoranthene	60	45	165	123	112	97	133	120	137	143	191	170	207	94	23	115	31	31	29
102	Benzo(*k*)fluoranthene	63	17	56	45	37	32	53	41	51	53	70	56	78	33	8	42	11	11	11
103	Chrysene & Triphenylene	33	23	105	71	65	66	79	77	87	79	129	95	112	59	6	73	22	25	22
104	Dibenzo(*a*,*h*)anthracene	8	8	29	22	21	19	26	22	25	30	39	36	39	21	6	23	6	6	6
105	Fluoranthene	56	32	163	133	112	110	125	134	150	115	182	159	66	148	25	115	37	44	40

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
106	Fluorene	5	4	15	17	12	17	12	14	14	10	17	14	9	7	4	7	4	3	3
107	Indeno(1,2,3-*cd*)pyrene	40	37	130	91	68	70	107	90	117	111	131	138	81	73	18	81	18	22	22
108	Naphthalene	10	9	39	36	23	25	24	33	41	17	31	32	21	18	12	20	14	12	8
109	Perylene	222	1021	386	278	381	542	451	281	300	338	384	363	237	178	55	196	62	62	53
110	Phenanthrene	30	16	88	79	61	65	70	79	87	70	92	85	56	50	17	66	22	23	33
111	Pyrene	60	44	184	135	123	123	150	142	154	133	161	165	102	95	21	115	38	45	41
112	*m*-Terphenyl	2	1	4	3	3	3	3	3	ND	ND	3	ND	ND	ND	1	ND	ND	ND	ND
113	*p*-Terphenyl	0	1	2	ND	ND	1	2	ND	ND	2	ND	2	1	1	ND	1	ND	ND	0
114	1-Methylnaphthalene	5.1	5.6	8.7	9.8	9.2	9.5	11.8	10.2	9.4	9.4	11.4	7.2	5.4	9.9	0.9	3.1	3.3	1.0	2.8
115	2-Methylnaphthalene	11.1	12.8	17.1	22.3	21.4	23.0	26.1	22.8	23.3	22.5	27.1	16.4	13.9	20.5	2.8	3.0	3.6	1.1	2.2
116	3-Methylphenanthrene	8.4	4.9	14.4	25.0	12.3	8.3	11.1	8.8	10.9	10.0	13.7	11.5	5.3	8.7	6.1	12.5	8.9	5.5	11.4
117	9-Methylphenanthrene	3.6	3.4	4.7	5.0	4.8	5.3	7.3	5.7	5.1	4.8	6.5	5.5	4.7	5.4	1.5	3.3	2.6	0.5	4.1
118	PCB#1	ND	0.03	0.04	0.04	0.04	0.05	0.10	0.10	0.06	0.06	0.09	0.05	0.05	0.06	0.03	0.03	0.04	0.02	0.04
119	PCB#3	0.04	ND	0.04	0.03	ND	0.03	0.05	0.03	0.04	ND	0.05	0.02	ND	0.02	ND	0.01	0.01	ND	ND
120	PCB#4&10	0.03	0.03	0.05	0.03	0.02	0.01	0.03	0.03	0.03	0.03	0.03	0.02	0.02	0.03	0.03	0.02	0.02	0.02	0.03
121	PCB#8	0.30	0.33	0.62	0.34	0.34	0.21	0.44	0.38	0.24	0.18	0.31	0.28	0.31	0.27	0.26	0.12	0.18	0.11	0.13
122	PCB#15	0.32	0.26	0.49	0.40	0.23	0.28	0.43	0.59	0.36	0.14	0.46	0.39	0.15	0.25	0.07	0.10	0.12	0.05	0.04
123	PCB#18	0.99	1.15	1.99	0.82	0.83	0.64	1.16	0.83	0.45	0.30	0.50	0.31	0.25	0.33	0.20	0.12	0.14	0.10	0.12
124	PCB#19	0.03	ND	ND	ND	ND	ND	0.06	ND	0.03	ND	0.02	ND	ND	0.03	0.01	0.01	ND	ND	ND
125	PCB#22	0.44	0.39	0.78	0.46	0.34	0.38	0.56	0.41	0.25	0.21	0.24	0.17	0.15	0.12	0.03	0.04	0.04	0.01	0.02
126	PCB#28	3.74	3.81	6.55	4.18	3.44	3.20	4.69	3.63	2.32	2.08	2.29	1.82	1.47	1.31	0.55	0.42	0.45	0.18	126

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
127	PCB#33	0.84	0.82	1.57	0.87	0.62	0.55	1.04	0.71	0.42	0.35	0.41	0.37	0.31	0.28	0.13	0.08	0.09	0.04	0.05
128	PCB#37	0.72	0.61	1.11	0.83	0.71	0.63	1.04	0.73	0.53	0.48	0.47	0.35	0.33	0.28	0.10	0.08	0.12	0.04	0.04
129	PCB#44	1.16	1.20	1.80	0.97	1.07	0.91	1.48	1.03	0.62	0.11	0.49	0.36	0.31	0.27	0.08	0.06	0.07	0.01	0.07
130	PCB#49	1.35	0.92	1.38	1.19	0.83	1.09	1.90	1.28	0.86	0.50	0.84	0.67	0.37	0.48	0.20	0.15	0.17	0.09	0.12
131	PCB#52	1.40	1.85	2.35	1.30	1.35	1.16	2.27	1.36	0.79	0.75	0.78	0.65	0.56	0.45	0.17	0.14	0.12	0.04	0.11
132	PCB#54	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.01	ND	ND	ND	ND	ND
133	PCB#70	1.57	1.96	2.95	1.90	1.45	1.66	2.64	1.78	1.18	1.31	1.02	0.73	1.03	0.54	0.27	0.16	0.14	0.04	0.10
134	PCB#74	1.11	0.95	1.46	1.23	0.94	1.08	1.54	1.06	0.70	0.60	0.66	0.56	0.44	0.37	0.15	0.12	0.13	0.05	0.08
135	PCB#77	0.54	0.38	0.45	0.40	0.27	0.23	0.56	0.55	0.28	0.18	0.30	0.20	0.08	0.17	0.03	0.05	0.09	0.01	0.03
136	PCB#81	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.01	ND	ND	ND
137	PCB#87	0.44	0.52	0.51	0.70	0.32	0.44	0.91	0.60	0.42	0.19	0.35	0.26	0.04	0.15	0.02	0.06	0.04	ND	0.09
138	PCB#95	0.98	0.91	1.14	0.94	0.88	0.99	1.41	1.02	0.62	0.54	0.57	0.39	0.33	0.38	0.18	0.11	0.11	0.04	0.60
139	PCB#99	1.01	0.95	1.11	1.00	0.85	0.96	1.63	1.06	0.83	0.67	0.73	0.61	0.57	0.50	0.24	0.18	0.18	0.07	0.11
140	PCB#101	1.52	1.63	1.75	1.59	1.28	1.49	2.69	1.82	1.29	0.83	1.17	0.92	0.81	0.72	0.28	0.23	0.19	0.06	0.72
141	PCB#105	0.44	0.48	0.43	0.50	0.38	0.43	0.87	0.55	0.37	0.29	0.36	0.24	0.20	0.20	0.06	0.06	0.07	0.02	0.06
142	PCB#110	1.13	1.13	1.47	1.42	1.06	1.17	2.08	1.51	0.97	0.73	0.86	0.64	0.36	0.46	0.13	0.15	0.14	0.04	0.28
143	PCB#114	0.01	ND	ND	ND	ND	ND	0.02	0.02	ND	ND	0.01	ND	ND	0.01	ND	ND	ND	ND	ND
144	PCB#118	1.70	1.54	1.79	2.11	1.57	1.68	2.95	2.31	1.40	0.84	1.49	1.09	0.75	0.89	0.29	0.31	0.26	0.11	0.25
145	PCB#119	0.05	0.06	0.07	0.07	0.04	0.04	0.09	0.05	0.05	0.03	0.05	0.03	0.01	0.02	0.01	0.01	ND	ND	ND
146	PCB#123	0.03	ND	ND	0.17	ND	0.20	0.10	0.04	ND	ND	0.03	0.04	ND	0.04	0.17	0.02	0.01	ND	ND
147	PCB#126	0.01	ND	ND	0.01	ND	ND	ND	ND	0.03	0.03	0.02	0.03	ND	ND	0.02	0.01	ND	ND	ND

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
148	PCB#128	0.23	0.03	0.03	0.29	ND	0.17	0.43	0.30	0.21	0.10	0.20	0.15	0.10	0.14	0.02	0.06	0.05	ND	0.11
149	PCB#138&158	0.55	0.53	0.62	0.63	0.60	0.52	1.08	0.75	0.58	0.40	0.56	0.45	0.38	0.44	0.24	0.16	0.13	0.05	0.93
150	PCB#149	1.07	0.80	0.68	1.04	0.93	1.03	1.92	1.39	0.99	0.44	1.11	0.94	0.50	0.97	0.58	0.26	0.21	0.08	2.84
151	PCB#151	0.31	0.24	0.23	0.26	0.31	0.35	0.66	0.30	0.31	0.17	0.23	0.18	0.09	0.30	0.15	0.06	0.05	0.02	1.36
152	PCB#155	0.13	0.03	0.36	0.30	0.10	0.09	0.13	0.19	0.07	0.07	0.10	0.05	0.07	0.02	0.02	0.01	0.01	ND	ND
153	PCB#153&168	0.53	0.49	0.53	0.57	0.51	0.56	1.06	0.74	0.59	0.38	0.58	0.50	0.40	0.53	0.26	0.15	0.13	0.05	1.23
154	PCB#156	0.08	0.04	0.07	0.09	0.06	0.08	0.18	0.08	0.06	0.03	0.07	0.05	0.03	0.05	0.02	0.03	0.02	ND	0.07
155	PCB#157	0.02	ND	0.01	0.02	0.02	0.02	0.06	0.02	0.02	ND	0.02	0.02	0.00	0.02	0.01	0.01	0.01	ND	0.01
156	PCB#167	0.03	0.03	0.03	0.06	ND	0.04	0.07	0.03	0.03	0.02	0.02	0.03	0.10	0.02	0.01	0.01	0.01	ND	0.03
157	PCB#170	0.22	0.13	0.16	0.23	0.16	0.23	0.48	0.34	0.27	0.13	0.34	0.24	0.21	0.45	0.24	0.11	0.06	0.03	1.66
158	PCB#171	0.05	0.03	0.02	0.04	0.03	0.06	0.11	0.09	0.08	ND	0.08	0.07	0.04	0.10	0.06	0.03	0.02	0.00	0.34
159	PCB#177	0.08	0.06	0.04	0.10	0.11	0.09	0.19	0.18	0.13	0.05	0.12	0.14	0.08	0.21	0.11	0.04	0.04	0.01	0.76
160	PCB#178	0.03	ND	ND	0.03	0.01	0.03	0.07	0.04	0.03	0.03	0.05	0.04	0.02	0.08	0.04	0.01	0.01	ND	0.31
161	PCB#180	0.41	0.34	0.29	0.43	0.40	0.42	0.86	0.60	0.62	0.35	0.62	0.58	0.45	1.00	0.59	0.19	0.16	0.05	3.70
162	PCB#183	0.10	0.06	0.07	0.09	0.12	0.11	0.22	0.16	0.14	0.09	0.17	0.14	0.13	0.24	0.14	0.05	0.04	0.01	0.88
163	PCB#187	0.22	0.15	0.17	0.29	0.29	0.28	0.51	0.37	0.34	0.23	0.37	0.31	0.29	0.59	0.34	0.12	0.12	0.04	1.88
164	PCB#189	ND	ND	ND	ND	ND	ND	0.01	ND	ND	ND	0.01	ND	ND	0.01	ND	ND	ND	ND	0.03
165	PCB#191	0.31	ND	ND	ND	0.27	0.30	0.67	ND	ND	0.25	0.01	0.44	0.33	0.02	0.49	ND	ND	0.04	0.04
166	PCB#194	0.09	0.07	0.06	0.09	0.06	0.08	0.20	0.07	0.07	0.07	0.10	0.08	0.12	0.20	0.17	0.03	0.03	0.01	0.53
167	PCB#199	0.08	0.05	0.05	0.08	0.07	0.10	0.18	0.13	0.12	0.05	0.14	0.12	0.08	0.27	0.14	0.06	0.04	0.01	0.95
168	PCB#201	0.01	ND	ND	0.02	ND	0.00	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01	ND	ND	ND	0.09

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
169	PCB#202	ND	ND	ND	0.01	ND	0.01	0.03	0.02	0.02	ND	0.03	0.01	ND	0.04	0.01	ND	ND	ND	0.13
170	PCB#205	ND	ND	ND	0.08	ND	0.07	ND	0.06	0.06	ND	ND	0.07	ND	0.01	0.02	0.03	0.03	ND	0.03
171	PCB#206	0.02	ND	ND	0.02	ND	0.02	0.04	0.03	0.03	ND	0.04	0.04	0.03	0.09	0.03	0.02	0.02	ND	0.16
172	PCB#208	0.01	ND	ND	0.01	ND	ND	0.01	0.01	ND	ND	0.01	0.01	ND	0.03	ND	ND	0.01	ND	0.03
173	PCB#209	0.05	0.02	0.05	0.06	0.03	0.05	0.08	0.10	0.04	0.02	0.08	0.06	0.04	0.09	0.02	0.03	0.02	0.01	0.01
174	Longifolene	6	9	11	14	10	16	8	2	8	9	5	9	4	2	2	1	11	ND	3
175	Biphenyl	4	3	9	8	7	7	7	8	8	6	8	8	7	6	8	5	3	3	3
176	Dibenzylether	3	2	3	2	3	3	4	4	ND	3	2	1	1	ND	ND	1	ND	ND	ND
177	Benzanthrone	12	8	25	22	19	17	20	20	24	27	32	30	18	18	11	20	7	6	7
178	Isophorone	4	4	8	9	8	8	8	9	9	8	6	7	4	3	ND	3	3	3	4
179	3,5-Dimethylphenol	ND	ND	ND	11	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
180	3-&4-Methylphenol	17	5	60	64	19	18	30	51	44	18	25	247	99	77	ND	269	11	14	5
181	4-*tert*-Octylphenol	9	35	18	11	19	14	9	9	7	20	9	13	5	3	3	3	2	1	0
182	Dibenzofuran	7	4	18	17	12	13	12	15	16	10	18	15	8	5	8	ND	4	3	2
183	Diphenylamine	ND	ND	ND	ND	ND	ND	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
184	Quinoline	4	1	7	8	5	5	6	7	7	4	14	6	5	4	3	5	2	2	2
185	Carbazole	5	2	20	16	9	5	11	17	16	13	20	11	7	7	5	8	2	1	2
186	4-Bromophenol	4	4	27	14	17	13	35	71	67	57	143	157	129	209	287	289	299	574	202
187	beta-Sitosterol	1926	2666	2722	3662	4071	3170	2644	2240	3588	3061	2011	2821	2499	1193	806	772	932	473	277
188	Campesterol	1822	4377	2185	3844	4358	2999	2022	1657	2976	2523	1312	1781	1634	759	453	461	642	285	246
189	Cholestane	5	9	5	7	11	9	11	10	12	10	9	10	9	7	4	3	4	2	3

Table 10 Cont'd

No	Compound	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20
190	Cholestanol	1711	3053	2618	3634	4436	2996	2465	2160	3795	3153	1866	2776	2585	1402	889	822	1059	470	377
191	Cholesterol	4943	3507	2234	2885	4234	2720	2592	2070	3219	2627	1511	1658	1916	1187	726	695	1447	817	1172
192	Coprostanol	189	791	896	757	946	569	511	350	784	602	278	306	411	211	91	101	133	56	42
193	Coprostanone	17	522	400	503	665	399	9	331	540	423	299	4	296	233	127	104	162	64	12
194	Epicoprostanol	497	301	301	684	512	1230	174	855	1168	1093	791	620	943	627	399	371	415	558	234
195	Stigmasterol	1686	2311	2216	3098	3175	2614	2199	2548	3035	2568	1791	2819	2223	1141	793	701	227	157	236
TOC, %		1.60	1.11	2.78	3.25	2.79	2.68	2.83	3.29	3.28	2.56	2.75	2.87	2.22	2.22	-	1.41	0.68	0.30	0.47
Total concentration		20166	25116	27392	39140	35366	33304	21368	25695	31862	28835	24817	26015	21040	21040	10335	11730	9917	6348	6095
Concentration nomarized by TOC, µg/kg C		1260397	2262725	985311	1204319	1267584	1242696	755039	780995	971414	1126348	902424	906431	947767	947767	-	831914	1458340	2115987	1296745
Number of compounds detected		173	159	163	171	162	174	177	169	169	161	173	170	156	156	151	171	156	142	151

4.3.3 Sources and spatial distribution of sterols

Sterols had the highest concentration compared to the other types of contaminants (Table 9). Coprostanol, which is an indicator of fecal pollution [49], increased in concentration towards the innermost part of the bay, although the average concentration of the 19 sites was 68% of the concentration in 1970[52] when the sewerage coverage ratio in Tokyo was 48%. The ratio of copostanol to cholesterol (sterol ratio) can be used to indicate sewage contamination (>0.2) [53]. In this study, the sterol ratio increased towards the innermost part of the bay (0.04 at Stn 20 to 0.40 at Stn3), where a large number of STPs are located, so it was with the coprostanol concentration. The sterol ratios at 9 sites in the innermost part of the bay were higher than 0.2, suggesting that the innermost part of the bay is contaminated by sewage. Nearly 100% of sewage in Tokyo metropolitan area is treated by the activated sludge method. Epicoprostanol is produced as a by-product of sewage treatment and is only present in treated sewage discharges or old samples [54]. A plot of the ratio of epicoprostanol/coprostanol against coprostanol/cholesterol can indicates the likely treatment or age in sediments in the bay [55]. A cross-plot of these ratios indicates that sewage is treated and/or is old (Figure 3). From these results, we hypothesize that although sediments in the bay are affected by sewage, most of human feces load comes from STP discharges.

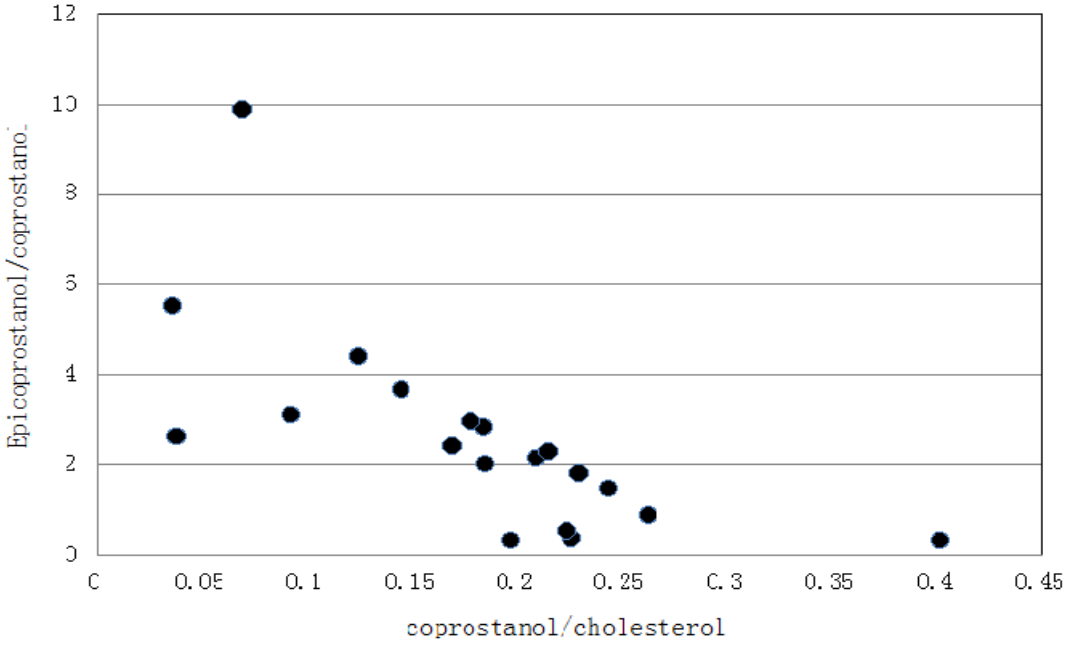

Figure 3 The relationship between epicoprostanol/coprostanol and coprostanol/cholesterol for all sediments.

4.3.4 Sources and spatial distribution of PAHs and n-alkanes

PAHs increased in concentration towards the innermost part of the bay (Table 10). A large number of vehicles and factories surround the bay, thus it was expected that PAH concentrations would be high. The PAHs levels are, however, relatively low compared with the enclosed seas in other developed countries. For instance, the average concentration of PAHs in estuary, harbor and coastal zone reported by Zakaria et al (2002) [56] is 6112 ng g^{-1} dry wt. Strict regulation for exhaust gas of vehicles and the effluents of factories is probably the reason for the relatively low pollution of the bay by PAHs. However, sediment PAHs concentrations in this study are similar to those observed throughout the bay in 1980[57], which suggests emissions of PAHs around the bay have not changed since 1980.

There are two anthropogenic sources of PAHs in the aquatic environment: petroleum products and pyrogenic processes. Several PAHs indices have been proposed to distinguish petrogenicand pyrogenic

sources of PAHs. The ratio of methyphenanthrenes(1-, 2-, 3- and 9-methyphenanthrene) to phenanthrene (MP/P ratio)[56,58] is one of the most useful indices. MP/P ratios in combustion mixtures are generally<1, whereas unburned fossil PAHs mixtures typically display a range of values from 2 to 6[56, 58]. Since the MP/P ratios obtained in this study were from 0.42 at Stn 13 to 1.07 at Stn 1 (average 0.64; SD 0.22), the ratios indicate that the origin of PAHs is pyrogenic. Another useful index is a ratio of fluoranthene to fluoranthene plus pyrene (Fl/ (Fl + Py)); ratios between 0.40 and 0.50 show liquid fossil fuel (vehicle and crude oil) combustion [59]. The ratios obtained from Tokyo Bay range from 0.42 at Stn 2 to 0.61 at Stn 14 (average 0.49; SD 0.04), again suggesting that source of PAHs is the combustion of liquid fossil fuel, which is consistent with Takada et al (1984)[61].

The composition ratio of PAHs at Stn 2 is different from those atother sites because of a high concentration of perylene. It is known that perylene originates from terrestrial organic matter [60], and the spatial distribution of perylene is quite different from those of other PAHs. From these findings, Stn 2 seems to be affected by perylene that flows into the bay through a river near Stn 2. To examine the source of PAHs, cluster analysis using all the PAHs excluding perylene was carried out. The dendrogram (Figure 4) obtained shows that the 19 sites were separated into 3 well defined groups. In addition, one group consisting of 14 sites covers three quarters of the bay, reinforcing the suggestion that the main source of PAHs is non-point source. The n-Alkanes also increased in concentration towards the innermost part of the bay (Table 10). It is known that there are two sources of n-alkanes in sediments: biogenic and petrogenic sources. Several indices were used to distinguish them [58]. The data used for distinguishing source are summarized in Table 11. All of the results obtained shows that main source of n-alkanes is terrestrial plants,

consistent with Takada et al. (1984) [61].

Table 11 n-Alkane indices for sediment samples

Index	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20	Average	SD
Major hydrocarbon[a]	C17	C31	C31	C17	C31	C31	C17	C17	C17	C31	C31	C31	C31	C31	C31	C31	C29	C11	C31	-	-
Sum of <C21/sum of >C20[b]	0.66	0.61	0.46	0.94	0.47	0.50	0.77	0.72	0.65	0.39	0.46	0.49	0.40	0.37	0.25	0.41	0.62	0.63	0.65	0.55	0.17
Sum of all *n*-alkanes/C16[c]	38	29	34	25	38	35	28	51	29	38	35	36	50	81	29	54	55	53	33	40	14
2(C27 + C29)/ (C26 + 2C28 + C30)[d]	1.6	1.7	1.5	1.2	1.4	1.5	2.2	1.0	1.4	1.8	1.6	1.8	1.8	1.7	2.4	1.5	3.0	1.4	1.1	1.7	0.5

[a] n-Alkanes around C18 are dominant for oily samples; C15, C17 or C19 is dominant for marine algae; C27, C29 or C31 is dominant for vascular land plants.

[b] The ratio of sum of <21 to sum of >20 for algae, plankton and crude oil is close to 1.0; the ratio for sedimentary bacteria, marine animals, higher plants and sediments shows lower value.

[c]Biogenic samples show large value (i.e., 50); oily samples are small value (i.e., 15).

[d]Around 1 is petrogenic hydrocarbons; 3 to 6 is vascular plants.

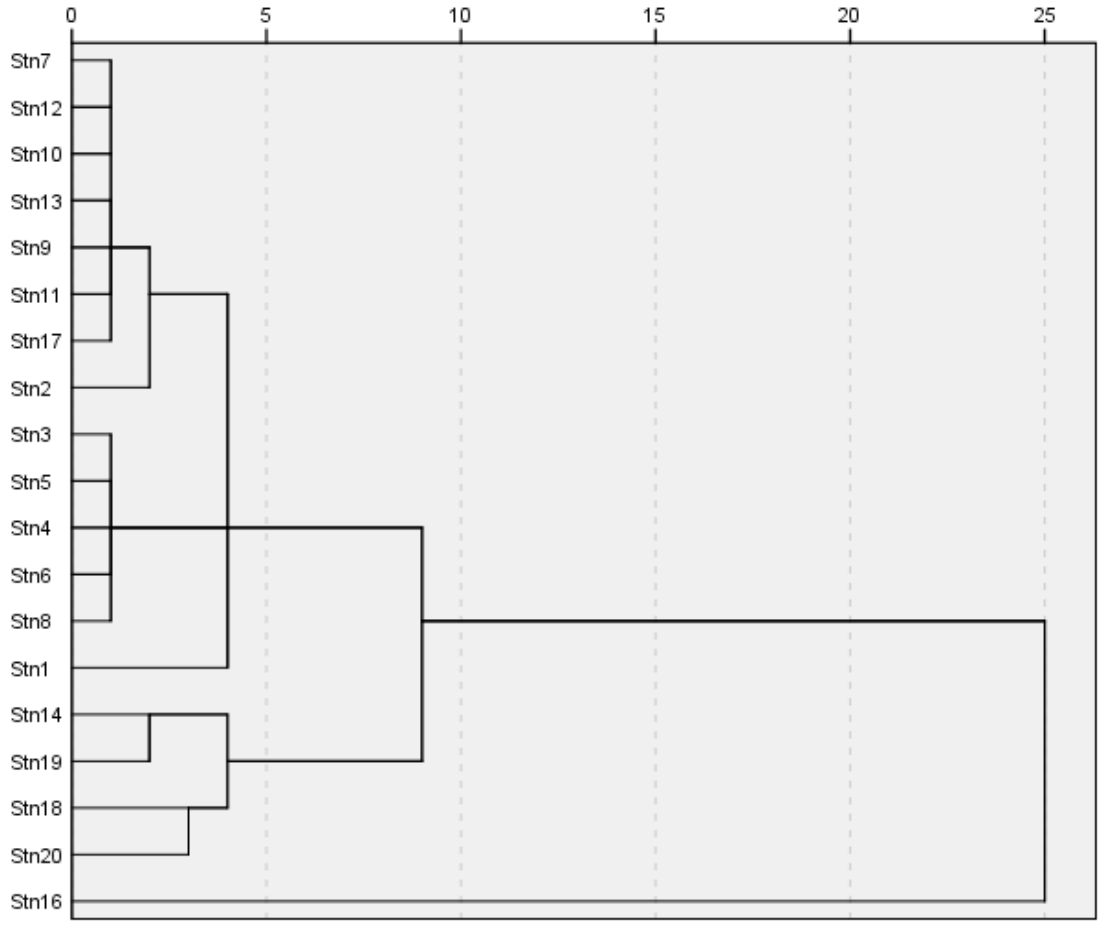

Figure 4 Dendrogram of all the sampling sites by cluster analysis using normalized PAHs (excluding perylene) concentrations.
(Ward method and squared Euclidean distance)

4.3.5 Spatial distribution of persistent organic pollutants

Among the OCPs examined in this study, DDTs, chlordanes, HCHs, nonachlor, heptachlorepoxide, dieldrin and endrin were detected (Table 10). Their concentrations are similar to those reported by Shimizu et al. (2005) [62], indicating that the amount of inflow to the bay did not change between 2000 and 2009. DDTs exhibited the highest concentrations followed by HCHs and chlordanes. The sites located on the west side in the innermost part of the bay (Stn 3, 4 and 7), had relatively high concentrations of OCPs. Assuming that these highly hydrophobic chemicals are adsorbed to suspended solids and settle to the bottom near their emission sources, they seem to flow into the bay from rivers. PCBs

were found at all sampling sites (Table 9 and 10). Relatively high concentrations were found in the innermost part of the bay, especially Stn 3 and 7 in the west side. However, relatively high concentrations were also found at Stn 20, a site that otherwise had the lowest total concentration of all contaminants across the 19 sampling sites (Table 9 and 10). In addition to the high concentration, the congener profile at Stn 20 was different from those of other sites. A number of PCB products (Kanechlor, KC-300, 400,500, 600 and 1000) have been used in Japan. Takasuga et al (2006) [74] reported their congener profiles, and so we performed cluster analysis using composition ratios of 20 congeners of KC-products and the 19 sites. The dendrogram obtained is shown in Figure5. Eighteen sites except for Stn 20 are classified into the same group of KC-MIX, and Stn 20 was in the KC-600 group, which indicates that Stn 20 was affected by a specific source (probably point source) nearby.

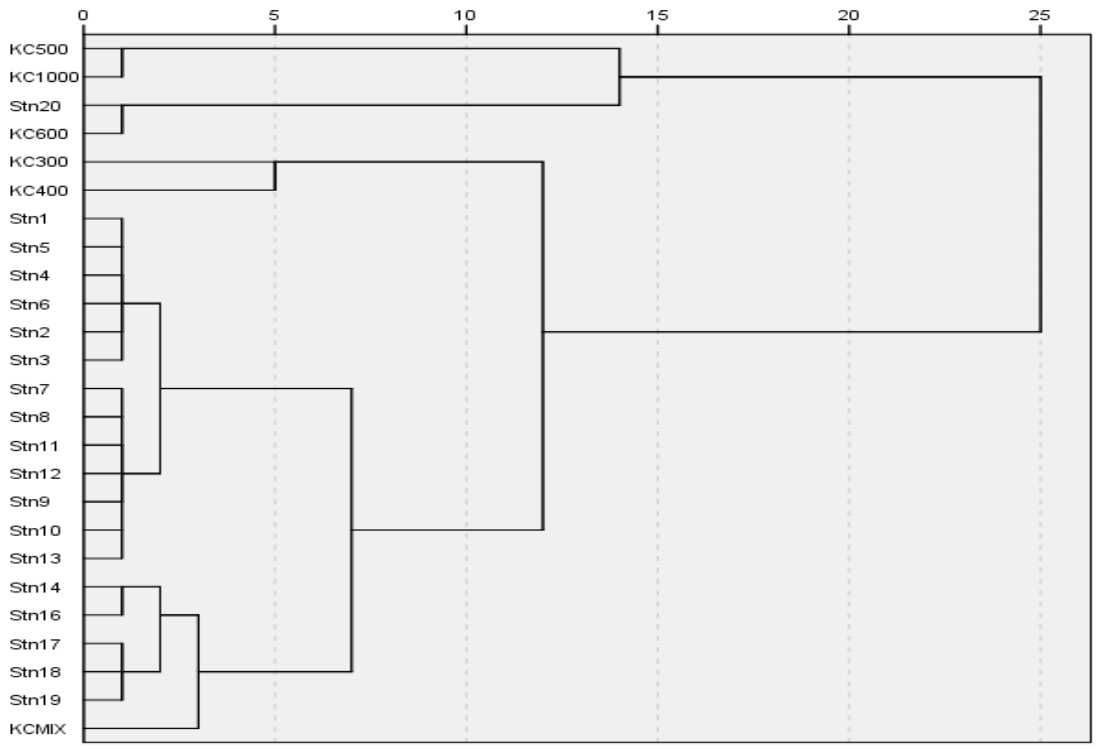

Figure 5 Dendrogram of all the sampling sites and PCB products by cluster analysis using normalized concentrations of 20 PCB congeners.
(Wardmethod and squared Euclidean distance)

4.3.6 Domestic chemicals

The chemicals that seem to be discharged from domestic sources, such as antioxidants, PPCPs, fire retardants, plasticizers, decomposed products of non-ionic detergent and substances leaching from tires [63], comprise a large proportion of the total contaminant concentration (Figure 6). Nonylphenol, octylphenol and bisphenol A, which are endocrine disrupting chemicals, were also detected at most sampling sites at concentrations at the same level as previous studies [64, 65]. Although nearly 100% of domestic wastewater (except for road run-off) is treated in STPs, many of these plants are not designed for the specific treatment of all these chemicals, and, moreover, when their maximum flow is exceeded, untreated water can enter the bay. Substances originating from tires via road run-off enter the bay without treatment. In Norway, Arp et al (2011)[65] compared the concentration of flame retardants entering and exiting water treatment plants and found no significant differences in concentration; the hypothesis they provided was that these chemicals were present on low density plastic residues that were not efficiently removed by flocculation, centrifugation or filtration. If their hypothesis is correct, hydrophobic substances that are easily absorbed on plastic are little decomposed by physical treatments. Further study on treatment efficiencies of these substances by the conventional STPs is necessary for improving sediment quality.

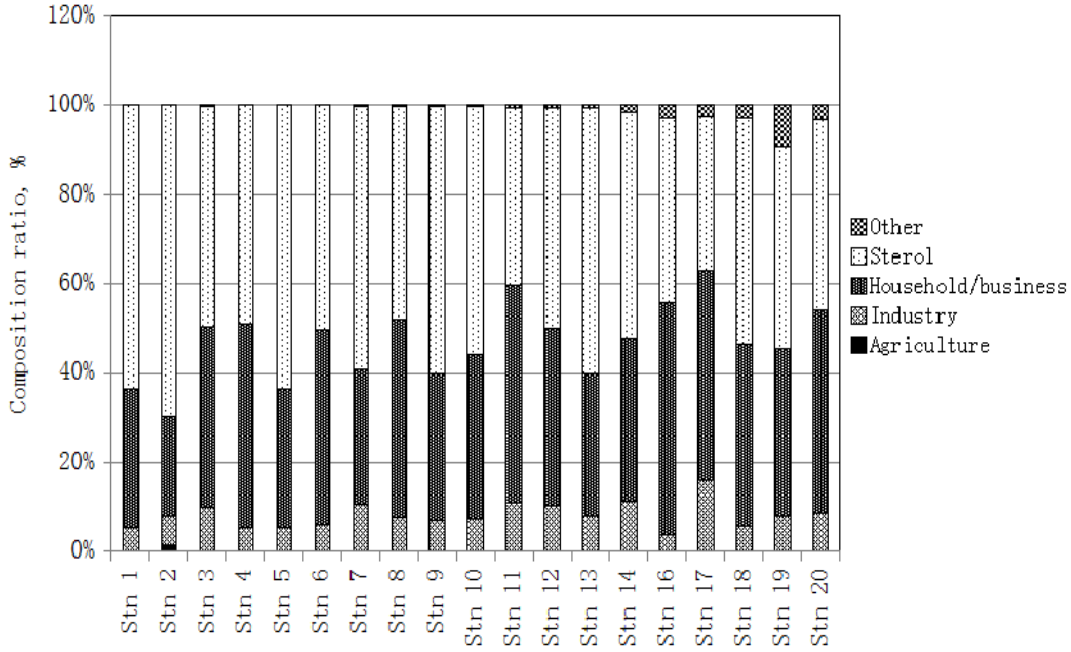

Figure 6 Composition ratios of emission sources.
Composition ratio of each source, which is categorized in Table 9, is the ratio of sum of detected concentrations of each source to the total detected concentration.

4.3.7 Specific substances found in the study

Bis(2-chloroisopropyl)ether was found only at Stn 2, though at a relatively high concentration (380 μg kg^{-1} dry wt). As far as we know, this is the first finding of this compound in sediments in Japan. This substance is used as an insecticide (nematocide) in Japan, and appeared to be discharged from a river near Stn 2. 4-Bromophenol was found at all the sampling sites and its concentration increased towards the mouth of the bay, i.e. in an opposite direction to all other substances, which indicates that 4-bromophenol does not enter from the land, but rather seems to enter the bay from the Pacific Ocean. 4-Bromophenol is synthesized by seaweeds, e.g. brown algae [67, 68], which supports the hypothesis of an oceanic source.

4.3.8 Differences in contamination levels between sampling sites

Tokyo Bay is a semi-closed bay with a relatively large surface area (960 km^2) [69]. There are likely regional differences in emission sources and amounts of micro-pollutants.

The volume of domestic wastewater discharged from Tokyo and Kanagawa Prefecture is much larger than that from Chiba Prefecture, due to population differences and the location of the three major rivers running through Tokyo. On the other hand, industrial factories are located in the both coastal areas: between Stn 11 and14 in the Tokyo side and between Stn 2 and 10 in the Chiba side. Therefore, since some differences in spatial distribution were expected, a cluster analysis was performed to classify the 19 sites. The 19 sites were classified into 4 groups: 1st, Stn 1, 3, 7, 12 and 13; 2nd, Stn 2, 5, 6, 9 and 10; 3rd, Stn 4, 8 and 11; 4th, Stn 14, 16, 17, 18, 19 and 20 (Figure 7).

Groups 2, 3, and 4 are comprised of sites physically closest to each other, indicating that sediments are affected by nearby emission sources. This corresponds with the results obtained in the Dokai Bay study [41]. Hydrophobic chemicals such as PAHs are readily sorbed to suspended solids and settle quickly to the bottom of the bay near emission source [75]. The west side and the east side from the center to the head of the bay are separated by currents throughout the year [70]. A clear anticlockwise circulation is found in the westside of the center of the bay in spring and summer. On the other hand, clockwise circulation is found in the mouth of the bay throughout the year. Since the areas of the coast separated by these currents are the same as the areas classified by the cluster analysis, the currents may also contribute to spatial distribution and diffusion of chemical substances.

Target chemicals were classified into five groups based on their main uses/sources: sterol, domestic (traffic, business or household),

industrial, agricultural and other (Table 9). We determined the contribution from each group at each site (Figure 6). The results confirm that the sediment in the bay was strongly affected with sterols and chemicals discharged from domestic sources. It takes 1.6 months for a complete exchange of seawater in the bay with outside seawater[71], so the effects from STPs and rivers, which are the emission sources of the both substances groups and are located in the innermost part of the bay, are diluted and/or removed by this exchange. This type of dilution, however, will have less of an impact on strongly sediment sorbed substances (like PAHs and PCBs) than it would on less strongly sorbed substances (like polar or ionic contaminants). As a result, both overall concentrations and the contribution from emission sources declined approaching the mouth of the bay, particularly for strongly sorbing substances.

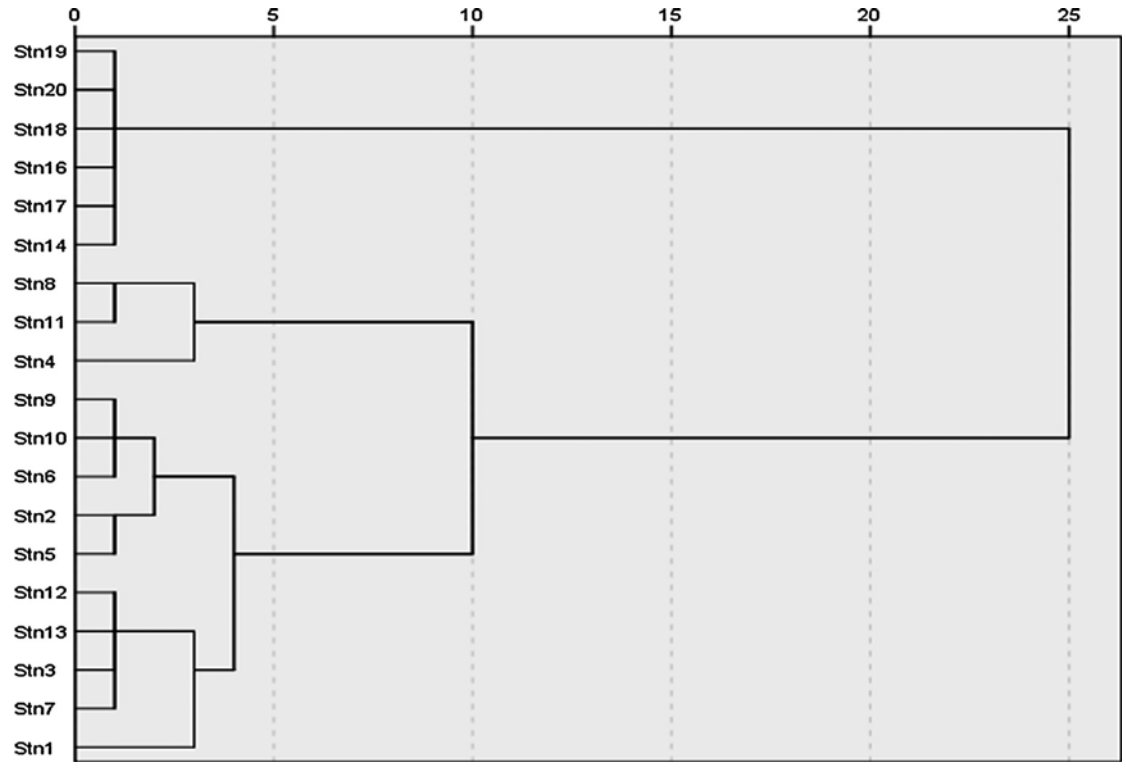

Figure 7 Dendrogram of all the sampling sites by cluster analysis using concentrations of all the detected compounds.

4.3.9 Comparison between the detected concentration and sediment quality guidelines

In order to evaluate the effect of the detected substances on benthic animals, we compared the detected concentration to the sediment quality guideline published by National Oceanic Atmospheric Administration [72]. Except for the sites located in the mouth of the bay, concentrations of PCBs and some OCPs are higher than the ‘‘effects range low’’ (ERL) threshold (Table 12). On the other hand, mean the ERM-Quotient, which is calculated by summing individual quotients (concentration/ERM) and dividing by the number of quotients, of all the sites are much smaller than one. Therefore micro-pollutants listed in the sediment quality guideline by this assessment are not predicted to produce substantial adverse effects on benthic animals in the bay. However, since a large number of micro-pollutants discharged by domestic and industrial activities other than those in the sediment quality guideline pollute sediments of the bay, their adverse effects cannot be ignored. In order to elucidate the effects by micro-pollutants on benthic animals, a detailed biological survey and/or bioassay in a laboratory using benthic animals, which have been found in the bay including in the past, is needed.

Table 12 Comparison between measured concentrations and sediment quality assessment guidelines of the National Oceanic and Atmospheric Administration (NOAA).

Substance	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20	ERL[a]	ERM[b]
Naphthalene	9.9	8.5	39	36	23	25	24	33	41	17	31	32	21	18	12	20	14	12	8.2	160	2100
Acenaphthylene	2.4	2.3	8.2	6.3	5.7	6	5.6	7.1	5.8	3.8	9.5	9.1	5.1	4.4	1.4	4.6	1.7	1.5	2	44	640
Acenaphthene	4.2	3.0	13	10	6.8	8.7	8.8	8.7	11	8.9	11	13	6.6	4.3	1	5.7	2	1.5	0.7	16	500
Fluorene	5.0	4.0	15	17	12	17	12	14	14	10	17	14	9	6.5	4	7.2	4.2	3.1	2.8	19	540
Phenanthrene	30	16	88	79	61	65	70	79	87	70	92	85	56	50	17	66	22	23	33	240	1500
Anthracene	11	6.9	25	24	19	23	18	22	24	16	26	25	15	15	6.1	17	6.2	6.1	5	85.3	1100
2-Methylnaphthalene	11	13	17	22	21	23	26	23	23	23	27	16	14	21	2.8	3.0	3.6	1.1	2.2	70	670
Sum LMW[c]-PAHs	73	54	206	195	148	168	165	187	206	148	214	194	127	119	44	123	54	48	54	552	3160
Fluoranthene	56	32	163	133	112	110	125	134	150	115	182	159	103	148	25	115	37	44	40	600	5100
Pyrene	60	44	184	135	123	123	150	142	154	133	161	165	102	95	21	115	38	45	41	665	2600
Benzo(a)anthracene	34	24	109	76	64	56	77	75	87	83	127	98	68	62	7	79	22	26	20	261	1600
Chrysene and Triphenylene	33	23	105	71	65	66	79	77	87	79	129	95	66	59	6	73	22	25	22	384	2800
Benzo(a)pyrene	35	34	112	70	72	61	87	73	88	92	136	109	72	64	8	82	21	23	20	430	1600
Dibenzo(a,h)anthracene	7.8	7.7	29	22	21	19	26	22	25	30	39	36	22	21	5.8	23	5.6	5.8	6	63.4	260
Sum HMW[d]-PAHs	226	165	703	508	458	435	545	522	591	532	773	662	432	449	72	487	146	169	149	1700	9600
Total PAHs	299	219	909	703	606	603	709	709	797	681	987	856	559	568	116	610	200	217	204	4022	44792
Total PCBs[e]	35	36	50	38	32	32	56	40	27	19	26	21	17	20	9.6	5.7	3.2	2.1	28	22.7	180
Chlordane	0.28	ND	0.38	1.2	0.47	0.47	0.23	0.37	0.31	ND	0.16	0.17	ND	ND	ND	0.026	ND	ND	ND	0.5	6
Dieldrin	ND	ND	ND	ND	ND	ND	0.55	ND	ND	ND	ND	ND	ND	0.21	ND	0.48	ND	ND	ND	0.02	8

Table 12 Cont'd

Substance	Stn 1	Stn 2	Stn 3	Stn 4	Stn 5	Stn 6	Stn 7	Stn 8	Stn 9	Stn 10	Stn 11	Stn 12	Stn 13	Stn 14	Stn 16	Stn 17	Stn 18	Stn 19	Stn 20	ERL[a]	ERM[b]
p,p0-DDE	3.0	2.3	3.1	2.8	1.9	4.4	4	2.5	1.6	1.5	1.6	1.3	0.95	0.84	0.36	0.38	0.3	0.09	0.17	2.2	
p,p0-DDD	0.46	0.54	0.70	0.49	0.54	0.35	0.68	0.35	0.34	0.33	0.37	0.32	0.34	0.26	0.1	0.091	0.1	0.03	0.078	2.2	27
p,p0-DDT	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1	7
Sum total DDTs	3.4	2.9	3.8	3.3	2.5	2.8	4.7	2.9	1.9	1.8	2	1.6	1.3	1.1	0.46	0.47	0.4	0.12	0.24	1.58	46.1
Mean ERM-Quotient[f]	0.03	0.03	0.06	0.05	0.04	0.04	0.06	0.05	0.04	0.04	0.05	0.04	0.03	0.03	0.01	0.03	0.01	0.01	0.01	–	–
TOC,% by weight	1.6	1.11	2.78	3.52	2.79	2.68	2.83	3.29	3.28	2.56	2.75	2.87	2.22	1.58	nm[g]	1.41	0.68	0.3	0.47	–	–

[a] EFL = Effects range-low.
[b] EFM = Effects range-medium.
[c] LMW = Low molecular weight.
[d] HMW = High molecular weight.
[e] A total PCBs concentration is calculated by dividing the concentration of 20-PCB congeners by their contribution to the total PCBs: KC MIX, 0.54 for Stns 1–19 and KC 600,
0.58 for Stn 20.
[f] A mean ERM-Quotient is calculated by summing individual quotients (concentration/ERM) and dividing by the number of quotients.
g nm = not measure.

4.4 Conclusions

In order to confirm that usefulness of the developed comprehensive method, we analyzed real sediments that were taken in sediments in Tokyo Bay in Japan. In this examination, we measured samples with GC-MS-SIM/TIM and identified and quantified the substances in the AIQS-DB. One-fifth of the target substances were observed in sediment extracts, with high concentrations in the innermost part of the bay. Chemicals associated with domestic activities (household, businesses and traffic) were found at relatively high concentrations even though nearly 100% of wastewater discharged from domestic and business activities is treated with STPs. This finding shows that detailed survey focusing on domestic substances by target analysis is necessary. On the other hand, industrial chemicals with high concentrations were not found in this study even though many factories are operating along the coast of the bay, indicating that industrial factories make efforts to reduce their emission amounts through cleaner-production and proper wastewater treatment. The results of the initial risk assessment, comparing detected concentrations and sediment quality assessment guidelines, suggested possibility that micro-pollutants continue to produce adverse effects on benthic animals. However, since a large number of micro-pollutants other than those in the sediment quality guideline were found in sediments of the bay, their potential adverse effects should not be ignored. In order to further improve sediment quality in the bay, countermeasures to reduce their inflow amounts would be needed after clarification of their emission sources and/or inflow routes to the bay. It was confirmed that the developed method using the AIQS-DB is a useful tool for grasping a whole pollution picture of the environment. Moreover, the whole pollution picture, which is difficult to obtain by conventional methods, is also useful to find emission sources in survey areas.

5 Conclusions

We have developed a comprehensive analytical method for SVOCs in sediment samples with the combination of a pre-treatment method comprising of extraction, column clean-up and AIQS-DB. From the recovery tests of chemicals by LLE with dichloromethane using 119 MCs, it was confirmed most SVOCs, except for polar substances, can be analyzed quantitatively. The results of examination of clean-up by adsorption chromatography with silica-gel show that the most suitable clean-up chromatography for sediments was the combination of silica-gel and acetone-hexane solution. From the overall recovery test, it was confirmed that the developed comprehensive method can analyze most SVOCs in sediments except for polar substances. From the analysis of a standard reference material (SRM), the accuracy and precision of the developed method are slightly lower than that of conventional methods that were developed for targeted analysis. Although the sensitivity of AIQS-DB using TIM may be insufficient for some chemicals that usually require a high sensitivity, such as POPs, this weak point can be overcome by using SIM/TIM mode; in the present study we applied SIM to POPs in SRM and sediments in Tokyo Bay in Japan and obtained good results. It is sufficient for environment surveys.

By applying the method to actual sediment samples in Tokyo Bay in Japan, we found the pollution by domestic chemicals and POPs that had been banned to use in the 1990s. From the pattern of detected chemicals as well as their concentration patterns, we can obtain a more holistic pollution picture in sediments. From these results, it is confirmed that the developed method using the AIQS-DB is a useful tool for grasping a whole pollution picture of the environment. In addition, it seems to be the most suitable method to confirm the safety of the environment after environmental accidents and natural disasters because a large number of

chemical substances can be measured rapidly at relatively low cost. Moreover, the whole pollution picture, which is difficult to obtain by conventional methods, is also useful to find emission sources in survey areas.

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7 Publications

[1]Development of Comprehensive Analytical Method for Semi-Volatile Organic Compounds in Sediments by Using Automated Identification and Quantification System with GC-MS Database, Kiwao KADOKAMI, Shuangye PAN, Duong Thi HANH, Xuehua LI, and Terumi MIYAZAKI, *Analytical Sciences*, 28, pp1183-1189, 2012.

[2]Screening Analysis of Hundreds of Sediment Pollutants and Evaluation of Their Effects on Benthic Organisms in Dokai bay, Japan, Kiwao Kadokami, XuehuaLi, Shuangye Pan, Naoko Ueda, Kenichiro Hamada, Daisuke Jinya, and Tomomi Iwamura, *Chemosphere*, 90, pp721-728, 2013.

[3]Target and Screening Analysis of 940 Micro-Pollutants in Sediments in Tokyo Bay, Japan, Shuangye Pan, Kiwao Kadokami, Xuehua Li, Hanh Thi Duong, and Toshihiro Horiguchi, *Chemosphere*, 99, pp 109-116, 2014.

[4]Screening and Analysis of 940 Organic Micro-pollutants in River Sediments in Vietnam Using an Automated Identification and Quantification Database System for GC-MS, Duong Thi Hanh, Kiwao Kadokami, Pan Shuangye, Naoki Matsuura, Nguyen Quang Trung, *Chemosphere*, 107, pp 462-472, 2014.

[5]A Rapid Analytical Method for Screening Organic Micro-pollutants by Gas Chromatography and Application, Shuangye Pan, Kiwao Kadokami, Modern Scientific Instruments, 3, pp 183-187, 2014(in Chinese).

8 Presentations

8.1 International symposiums

[1]Development of a Comprehensive Analytical Method Using a Novel GC-MS Database for Grasping the Whole Picture of Chemical Pollution, Kiwao Kadokami, Daisuke Jinya, Pan Shuangye, Hanh Duong, Xuehua Li, Terumi Miyazaki, SETAC Asia/Pasific, 2012, Kumamoto.

[2]Target and Screening Analysis of 1000 Substances in Substances in Sediments in Tokyo Bay, Japan, Pan Shuangye, Kiwao Kadokami, Xuehua Li, Duong Thi Hanh and Toshihiro Horiguchi, Dioxin 2013, 2013, Daegu, Korea.

8.2 Domestic symposiums

[1]Occurrence of Micro-pollutants in Sediments in Tokyo Bay, Pan Shuangye, Kiwao Kadokami, The 45th Annual Conference of Japan Society on Water Environment 2011, Sapporo.

[2]Distribution and Origin of Organic Micro-pollutants in Sediments in Tokyo Bay, Pan Shuangye, Kiwao Kadokami, Li Xuehua，Toshihiro Horiguchi, 20th Symposium on Environmental Chemistry, 2011, Kumamoto.

Appendix 1 Compounds registered in the database

No.	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
1	Dibutylamine	C8H19N	111-92-2	129	86	7.93	0.01	intermediate in organic synthesis	industry
2	3-Hexanol, 4-ethyl-	C8H18O	19780-44-0	130	59	8.091	0.01		industry
3	Phenol	C15H16O2	80-05-7	228	213	8.211	0.01	intermediate for resin/antioxidant	business/household
4	Aniline	C6H7N	62-53-3	93	93	8.233	0.01	intermediate in organic synthesis/leaching from tire	business/household
5	3-Methoxy-1-butyl acetate	C7H14O3	4435-53-4	146	71	8.302	0.01	intermediate for resin/solvent	industry
6	Pentachloroethane	C2HCl5	197-61-7	200	167	8.35	0.01		industry
7	1,1,1-Trichloro-2-methyl-2-propanol	C4H7Cl3O	57-15-8	176	125	8.352	0.01	plasticizer	business/household
8	Bis(2-chloroethyl)ether	C4H8Cl2O	111-44-4	142	93	8.482	0.01	intermediate in organic synthesis/solvent	industry
9	2-Chlorophenol	C6H5ClO	95-57-8	128	128	8.485	0.01	by-product of chlorination/ intermediate in organic synthesis	industry
10	Hymexazol	C4H5NO2	10004-44-1	99	99	8.685	0.01		
11	Butanoic acid, butyl ester	C8H16O2	109-21-7	136	89	8.692	0.01	fragrance	business/household
12	n-C10H22	C10H22	124-18-5	142	85	8.802	0.01	petroleum	business/household
13	1,3-Dichlorobenzene	C6H4Cl2	541-73-1	146	146	8.922	0.01	solvent/intermediate in organic synthesis	industry
14	Nicotinonitrile	C6H4N2	100-54-9	104	104	8.96	0.01	intermediate for pesticide	industry
15	Benzyl chloride	C7H7Cl	100-44-7	126	91	9.072	0.01	intermediate in organic synthesis	industry
16	1,4-Dichlorobenzene	C6H4Cl2	106-46-7	146	146	9.127	0.01	insecticidal fumigant	business/household
17	4-Cymene	C10H14	99-87-6	134	119	9.327	0.03	solvent	industry
18	Dicyclopentadiene	C10H12	77-73-6	132	66	9.375	0.03	intermediate for resin	industry
19	2-Ethyl-1-hexanol	C8H18O	104-76-7	130	83	9.379	0.01	intermediate in organic synthesis/fragrance/leaching from tire	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
20	Benzyl alcohol	C7H8O	100-51-6	108	108	9.476	0.01	cosmetics/fuel additive/solvent/leaching from tire	business/household
21	1,2-Dichlorobenzene	C6H4Cl2	95-50-1	146	146	9.493	0.01	solvent	industry
22	2-Methylphenol	C16H26O	4130-42-1	234	219	9.796	0.03	antioxidant	business/household
23	Bis(2-chloroisopropyl)ether(DCIP)	C6H12Cl2O	108-60-1	170	121	10.018	0.01	insecticide	agriculture
24	Methomyl oxime	C5H11N3O2S	13749-94-5	105	105		0.01	other pesticide	agriculture
25	trans-Decahydronaphthalene	C10H18	493-02-7	138	138	10.073	0.03	solvent	industry
26	N-Nitrosopyrrolidine	C4H8N2O	930-55-2	100	100	10.094	0.01	reagent	business/household
27	N-Methylaniline	C7H9N	100-61-8	107	106	10.148	0.03	intermediate in organic synthesis	industry
28	Acetophenone	C8H8O	98-86-2	120	105	10.156	0.01	solvent/leaching from tire	business/household
29	N-Nitrosomorpholine	C4H8N2O2	59-89-2	116	116	10.232	0.01	solvent/intermediate in organic synthesis	industry
30	3-&4-Methylphenol	C10H14O	88-18-6	150	135	10.232	0.01	intermediate in organic synthesis	industry
31	Octanol	C8H18O	111-87-5	130	56	10.247	0.01	cosmetics/fragrance/solvent	business/household
32	2-Methylaniline	C7H9N	95-53-4	107	106	10.247	0.01	intermediate for dyes	industry
33	Hexachloroethane	C2Cl6	67-72-1	234	201	10.389	0.01	intermediate in organic synthesis	industry
34	3-Toluidine	C7H9N	108-44-1	107	106	10.39	0.03	intermediate for dyes	industry
35	2-Chloro-6-methylphenol	C7H7ClO	87-64-9	142	107	10.424	0.01		industry
36	2-Methoxyphenol	C8H10O	576-26-1	122	122		0.01	intermediate for resin	industry
37	Nitrobenzene	C6H5NO2	98-95-3	123	123	10.588	0.01	intermediate in organic synthesis	industry
38	N,N-Dimethylaniline	C8H11N	121-69-7	121	120	10.636	0.03	intermediate for dyes	industry
39	1,2-Dibromo-3-chloropropane	C3H5Br2Cl	96-12-8	234	157	10.665	0.03	intermediate in organic synthesis	industry
40	3-Bromochlorobenzene	C6H4BrCl	108-37-2	190	192	10.774	0.03		industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
41	n-C11H24	C11H24	1120-21-4	156	85	10.872	0.01	petroleum/plant	business/household
42	2,6-Dimethylphenol	C18H30O	732-26-3	262	247	10.973	0.01		industry
43	N-Nitrosopiperidine	C5H10N2O	100-75-4	114	114	11.011	0.01	reagent	business/household
44	Phenylethyl alcohol	C8H10O	60-12-8	122	122	11.1	0.01	fragrance/leaching from tire	business/household
45	2-Bromochlorobenzene	C6H4BrCl	694-80-4	190	192	11.299	0.03		industry
46	Isophorone	C9H14O	78-59-1	138	82	11.301	0.03	paint/solvent	industry
47	Methyl octanoate	C9H18O2	111-11-5	158	87	11.315	0.01	fatty acid methy ester	business/household
48	2-Chloroaniline	C6H6cLN	95-51-2	127	127	11.395	0.03	intermediate for dyes	industry
49	Aldoxycarb (deg)	C7H14N2O4S	1646-88-4	222	68	11.404	0.01	insecticide	agriculture
50	N-Ethylaniline	C8H11N	103-69-5	121	106	11.426	0.03	intermediate for dyes	industry
51	2-Nitrophenol	C6H5NO3	88-75-5	139	139	11.459	0.01	intermediate in organic synthesis/ exhaust gas of automobile	business/household
52	1,3,5-Trichlorobenzene	C6H3Cl3	108-70-3	180	182	11.514	0.03	intermediate in organic synthesis/solvent	industry
53	Urea, N,N-diethyl-	C5H12N2O	634-95-7	116	116	11.709	0.01	leaching from tire	business/household
54	2,4-Dimethylphenol	C10H8O	90-15-3	144	144	11.714	0.01	intermediate for dyes	industry
55	Bis(2-chloroethoxy)methane	C5H10Cl2O2	111-91-1	172	93	12.04	0.01	intermediate in organic synthesis	industry
56	2-Nitrotoluene	C7H7NO2	88-72-2	137	120	12.042	0.01	intermediate in organic synthesis	industry
57	3,5-Dimethylphenol	C10H14O	585-34-2&98-54-4	150	135	12.144	0.01	intermediate for resin	industry
58	2,4-Dichlorophenol	C6H4Cl2O	120-83-2	162	162	12.146	0.01	reagent, by-product of chlorination	business/household
59	2,6-Dimethylaniline	C8H11N	87-62-7	121	121	12.15	0.01	intermediate in organic synthesis	industry
60	Clofentezine	C14H8Cl2N4	74115-24-5	302	137	12.19	0.01	other pesticide	agriculture
61	2,5-Dichlorophenol	C6H4Cl2O	583-78-8	162	162	12.196	0.01	intermediate for pesticides	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
62	1-Nonanol	C9H20O	143-08-8	144	83	12.243	0.01	intermediate for fragrance	business/household
63	2,5-Dimethylaniline	C8H11N	95-78-3	121	121	12.272	0.03	intermediates in the synthesis of dyes	industry
64	2,3-Dichlorophenol	C6H4Cl2O	576-24-9	162	162	12.284	0.01	reagent	business/household
65	2-Anisidine	C7H9NO	90-04-0	123	123	12.31	0.03	intermediate for dyes	industry
66	3,5-Dimethylaniline	C8H11N	108-69-0	121	121	12.341	0.03	intermediate for dyes	industry
67	1,2,4-Trichlorobenzene	C6H3Cl3	120-82-1	180	180	12.41	0.01	intermediate in organic synthesis/solvent	industry
68	L-Menthol	C10H20O	2216-51-5	156	95	12.416	0.01	PPCPs	business/household
69	Naphthalene	C10H8	91-20-3	128	128	12.573	0.01	PAH	industry
70	3-&4-Chlorophenol	C6H5ClO	108-43-0&106-48-9	128	128	12.621	0.02	by-product of chlorination/intermediate in organic synthesis	industry
71	alpha-Terpineol	C10H18O	10482-56-1	154	136	12.751	0.03	perfumes/solvent	business/household
72	3-Nitrotoluene	C7H7NO2	99-08-1	137	137	12.758	0.01	intermediate in organic synthesis	industry
73	2,3-&3,4-Dimethylaniline	C8H11N	87-59-2&95-64-7	121	121	12.765	0.05	intermediate in organic synthesis	industry
74	n-C12H26	C12H26	112-40-3	170	85	12.81	0.01	petroleum	business/household
75	4-Chloroaniline	C6H6ClN	106-47-8	127	127	12.817	0.01	intermediate for dyes and pesticides	industry
76	2,6-Dichlorophenol	C6H4Cl2O	87-65-0	162	162	12.825	0.01	by-product of chlorination/intermediate for trichlorophenol	industry
77	Hexachloropropylene	C3Cl6	1888-71-7	246	213	12.878	0.01	solvent	industry
78	2-Acetyl-5-methylthiophene	C7H8OS	13679-74-8	140	125	12.913	0.01	leaching from tire	business/household
79	1,2,3-Trichlorobenzene	C6H3Cl3	87-61-6	180	180	13.011	0.03	intermediate in organic synthesis/solvent	industry
80	Hexachlorobutadiene	C4Cl6	87-68-3	258	225	13.045	0.01	solvent	industry
81	4-Nitrotoluene	C7H7NO2	99-99-0	137	137	13.115	0.01	intermediate in organic synthesis	industry
82	4-Anisidine	C7H9NO	104-94-9	123	108	13.137	0.03	intermediate for dyes	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
83	Ethanol, 2-phenoxy-	C8H10O2	122-99-6	138	94	13.137	0.01	leaching from tire/solvent/intermediate in organic synthesis	business/household
84	Methamidophos	C2H8NO2PS	10265-92-6	141	94	13.214	0.05	insecticide	agriculture
85	Benzothiazole	C7H5NS	95-16-9	135	135	13.335	0.01	leaching from tire	business/household
86	3-Chloronitrobenzene	C6H4ClNO2	121-73-3	157	157	13.338	0.01	intermediate in organic synthesis	industry
87	3-Anisidine	C7H9NO		123	123	13.493	0.03		industry
88	Quinoline	C9H7N	91-22-5	129	129	13.55	0.03	intermediate in organic synthesis	industry
89	DDVP	C4H7Cl2O4P	62-73-7	220	185	13.583	0.01	insecticide	agriculture
90	4-Chloronitrobenzene	C6H4ClNO2	100-00-5	157	157	13.625	0.01	intermediate in organic synthesis	industry
91	e-Caprolactam	C6H11NO	105-60-2	113	113	13.753	0.01	intermediate for fiber	industry
92	Formamide, N-cyclohexyl-	C7H13NO	766-93-8	127	84	13.85	0.01	leaching from tire	business/household
93	p-Phenylenediamine	C6H8N2	106-50-3	108	108	13.893	0.01	intermediate for dyes/developing fluid	industry
94	N-Nitroso-di-n-butylamine	C8H18N2O	924-16-3	158	84	13.924	0.01	reagent	business/household
95	1,4-Benzenediol	C6H6O2	123-31-9	110	110	13.965	0.01	developing fluid	business/household
96	2-tert-Butylphenol	C10H14O	89-72-5	150	121	14.048	0.01	intermediate in organic synthesis	industry
97	Novaluron-deg	C17H9ClF8N2O4	116714-46-6	492	335	14.113	0.01	insecticide	agriculture
98	2-sec-Butylphenol	C10H8O	135-19-3	144	144	14.133	0.01	intermediate in organic synthesis	industry
99	Nereistoxin oxalate deg.					14.151			
100	m-Aminophenol	C6H7NO	591-27-5	109	109	14.229	0.01	intermediate for dyes	industry
101	Pentamethylbenzene	C11H16	700-12-9	148	133	14.276	0.03		industry
102	4-Bromophenol	C6H5BrO	106-41-2	172	172	14.285	0.03		industry
103	Allidochlor	C8H12ClNO	93-71-0	173	138	14.318	0.01	herbicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
104	4-Chloro-3-methylphenol	C7H7ClO	59-50-7	142	142	14.35	0.03	fungicide, paint	agriculture
105	Thymol	C10H14O	89-83-8	150	135	14.423	0.01	PPCPs	business/household
106	3- & 4-tert-Butylphenol	C11H16O2	121-00-6	180	165	14.425	0.01	antioxidant	business/household
107	m-Phenylenediamine	C6H8N2	108-45-2	108	108	14.452	0.01	intermediate for dyes	industry
108	Safrole	C10H10O2	94-59-7	162	162	14.478	0.01	PRESERVATIVE/intermediate in organic synthesis	industry
109	2-Methylbenzothiazole	C8H7NS	120-75-2	149	149	14.589	0.03		industry
110	5-Chloro-2-methyl aniline	C7H8ClN	95-79-4	141	106	14.599	0.03	intermediate for dyes	industry
111	2-Methylnaphthalene	C11H10	91-57-6	142	142	14.621	0.01	PAH	industry
112	n-C13H28	C13H28	629-50-5	184	85	14.627	0.01	petroleum/plant	business/household
113	1,2,3-Trimethoxybenzene	C9H12O3	634-36-6	168	168	14.727	0.03		industry
114	4-sec-Butylphenol	C11H16O	14938-35-3	164	107	14.781	0.01		business/household
115	3-&4-Nitroanisole	C7H7NO3	555-03-3& 100-17-4	153	153	14.989	0.05	intermediate in organic synthesis	industry
116	Methyl decanoate	C11H22O2	110-42-9	186	87	15.015	0.01	fatty acid methy ester	business/household
117	2,4-Dichloroaniline	C6H5Cl2N	554-00-7	161	161	15.134	0.01	reagent/intermediate in organic synthesis	business/household
118	1,2,4,5-Tetrachlorobenzene	C6H2Cl4	95-94-3	214	216	15.135	0.01	intermediate in organic synthesis	industry
119	2,3,5-Trichlorophenol	C6H3Cl3O	933-78-8	196	196	15.136	0.01	intermediate for pesticides/preservative	industry
120	Hexachlorocyclopentadiene	C5Cl6	77-47-4	270	237	15.141	0.01	intermediate in organic synthesis	industry
121	Dichlobenil	C7H3Cl2N	1194-65-6	171	171	15.27	0.01	herbicide	agriculture
122	Tribenuron-methyl	C15H17N5O6S	101200-48-0	395	154	15.34	0.01	herbicide	agriculture
123	Isosafrole	C10H10O2	120-58-1	162	162	15.347	0.01	perfumes	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
124	Chlorimuron-ethyl	C15H15ClN4O6S	99283-00-8	414	159	15.354	0.01	herbicide	agriculture
125	Phenol, 2,6-dimethoxy-	C15H24O	25154-52-3	220	135	15.41	0.01	nonionic detergent metabolite	business/household
126	2-Nitroanisole	C7H7NO3	91-23-6	153	123	15.42	0.03	intermediate in organic synthesis	industry
127	Nicotine	C10H14N2	54-11-5	162	84	15.452	0.01	PPCPs	business/household
128	2,3-Dichloroaniline	C6H5Cl2N	608-27-5	161	161	15.474	0.03	reagent	business/household
129	2,4,6-Trichlorophenol	C6H3Cl3O	88-06-2	196	196	15.489	0.01	by-product of chlorination/intermediate for pesticides	industry
130	2,4,5-Trichlorophenol	C6H3Cl3O	95-95-4	196	196	15.562	0.01	intermediate for pesticides/preservative	industry
131	4-n-Butylphenol	C8H10O	108-68-9	122	122	15.584	0.03	intermediate in organic synthesis	industry
132	EPTC	C9H19NOS	759-94-4	189	128	15.601	0.01	herbicide	agriculture
133	2,5-Dichloronitrobenzene	C6H3Cl2NO2	89-61-2	191	191	15.722	0.01	intermediate in organic synthesis	industry
134	2,3,4-Trichlorophenol	C6H3Cl3O	15950-66-0	196	196	15.724	0.01	intermediate for pesticides/preservative	industry
135	Acetamide, N-phenyl-	C8H9NO	103-84-4	135	135	15.85	0.01	leaching from tire	business/household
136	2,4-Dichloronitrobenzene	C6H3Cl2NO2	611-06-3	191	191	15.866	0.01	intermediate in organic synthesis	industry
137	2-Chloronaphthalene	C10H7Cl	91-58-7	162	162	15.971	0.01	PCN	industry
138	2,3,6-Trichlorophenol	C6H3Cl3O	933-75-5	196	196	16.026	0.01	intermediates in the synthesis of dyes, pigments, and phenolic resins	industry
139	1-Chloronaphthalene	C10H7Cl	90-13-1	162	162	16.029	0.01	PCN	industry
140	2,6-Diaminotoluene	C7H10N2	823-40-5	122	122	16.059	0.01	intermediate in organic synthesis	industry
141	3,5-Dichlorophenol	C6H4Cl2O	591-35-5	162	162	16.06	0.01	reagent	business/household
142	Biphenyl	C12H10	92-52-4	154	154	16.06	0.03	heat-transfer oils/intermediate in organic synthesis	industry
143	Propamocarb	C22H17N3O5	131860-33-8	403	344	16.209	0.01	fungicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
144	2,3-Dichloronitrobenzene	C6H3Cl2NO2	3209-22-1	191	191	16.224	0.03	intermediate in organic synthesis	industry
145	n-C14H30	C14H30	629-59-4	198	85	16.335	0.01	petroleum	business/household
146	2-Nitroaniline	C6H6N2O2	88-74-4	138	138	16.401	0.01	intermediate in organic synthesis	industry
147	Diphenyl ether	C12H10O	101-84-8	170	170	16.413	0.03	fragrance	business/household
148	3,4-Dichlorophenol	C6H4Cl2O	95-77-2	162	162	16.459	0.01	intermediate in organic synthesis	industry
149	2,4,6-Trichloroaniline	C6H4Cl3N	634-93-5	195	195	16.469	0.03	reagent/intermediate in organic synthesis	business/household
150	2,6-Dimethylnaphthalene	C12H12	581-42-0	156	156	16.502	0.03	PAH	industry
151	Longifolene	C15H24	475-20-7	204	161	16.63	0.03		industry
152	Mevinphos 1	C7H13O6P	7786-34-7	224	127	16.669	0.01	insecticide	agriculture
153	Cyclohexanamine, N-cyclohexyl-	C12H23N	101-83-7	181	138	16.69	0.01	leaching from tire	business/household
154	Methyl undecanoate	C12H24O2	1731-86-8	200	87	16.701	0.01	fatty acid methy ester	business/household
155	1,3-Dimethylnaphthalene	C12H12	575-41-7	156	156	16.733	0.03	PAH	industry
156	Mevinphos 2	C7H13O6P	7786-34-7	224	127	16.734	0.01	insecticide	agriculture
157	Quinoline, 2,7-dimethyl-	C11H11N	93-37-8	287	157	16.76	0.01		industry
158	3,4-Dichloroaniline	C6H5Cl2N	95-76-1	161	161	16.793	0.03	intermediate for dyes and pesticides	industry
159	Butylate	C11H23NOS	2008-41-5	217	146	16.797	0.01	herbicide	agriculture
160	Acephate	C4H10NO3PS	30560-19-1	183	136	16.808	0.05	insecticide	agriculture
161	1,4-Dinitrobenzene	C6H4N2O4	100-25-4	168	168	16.886	0.01	intermediate in organic synthesis	industry
162	Diphenylmethane	C13H12	101-81-5	168	167	16.909	0.03	PAH	industry
163	Chlormephos	C5H12ClO2PS2	24934-91-6	235	234	16.945	0.01		
164	1,4-&2,3-Dimethylnaphthalene	C12H12	571-58-4&581-40-8	156	156	17.061	0.05	PAH	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
165	1,3-Dinitrobenzene	C6H4N2O4	99-65-0	168	168	17.085	0.01	intermediate in organic synthesis	industry
166	Etridiazole (Echlomezol)	C20H23NO3	71626-11-4	325	148	17.109	0.01	fungicide	agriculture
167	Dimethyl phthalate	C10H10O4	131-11-3	194	163	17.125	0.01	plasticizer	business/household
168	3-Hydroxycarbofuran 1	C12H15NO4	16655-82-6	237	137	17.174	0.01	insecticide	agriculture
169	2,6-Dinitrotoluene	C7H6N2O4	606-20-2	182	165	17.217	0.01	intermediate in organic synthesis	industry
170	Trichlorfon	C4H8Cl3O4P	52-68-6	256	109	17.231	0.01	insecticide	agriculture
171	Acenaphthylene	C12H8	208-96-8	152	152	17.237	0.01	PAH	industry
172	4-n-Pentylphenol	C15H24O	104-40-5	220	107	17.247	0.01	co-stabilizer	industry
173	Propham	C10H13NO2	122-42-9	179	179	17.287	0.01	herbicide	agriculture
174	1,2-Dimethylnaphthalene	C12H12	573-98-8	156	141	17.297	0.03	PAH	industry
175	2-Isopropylnaphthalene	C13H14	2027-17-0	170	155	17.31	0.03	PAH	industry
176	Pebulate	C10H21NOS	1114-71-2	203	128	17.328	0.01	herbicide	agriculture
177	2,6-Di-tert-butyl-4-benzoquinone	C14H20O2	719-22-2	220	177	17.365	0.03	antioxidant	business/household
178	Phthalimide	C8H5NO2	85-41-6	147	147	17.441	0.01	leaching from tire	business/household
179	1,8-Dimethylnaphthalene	C12H12	569-41-5	156	156	17.642	0.03	PAH	industry
180	3-Nitroaniline	C6H6N2O2	99-09-2	138	138	17.646	0.01	intermediate in organic synthesis	industry
181	2-tert-Butyl-4-methoxyphenol	C12H10O	90-43-7	170	170	17.694	0.01	intermediate in organic synthesis	industry
182	Acenaphthene	C12H10	83-32-9	154	153	17.771	0.01	PAH	industry
183	Methacrifos	C7H13O5PS	30864-28-9	240	208	17.897	0.01	insecticide	agriculture
184	Dimethylterephthalate	C10H10O4	120-61-6	194	163	17.917	0.03	intermediate for resin	industry
185	Thiocyclam	C5H11NS3	31895-21-3	181	135	17.931	0.01		
186	n-C15H32	C15H32	629-62-9	212	85	17.945	0.02	petroleum/plant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
187	2,4-Dinitrophenol	C6H4N2O5	51-28-5	184	184	17.963	0.01	intermediate in organic synthesis	industry
188	2-Bromo-4,6-dichloroaniline	C6H4BrCl2N	697-86-9	239	241	17.984	0.03	reagent	business/household
189	PCB #1	2-	2051-60-7	188	188	17.984	0.01	PCB	industry
190	4-Methyl-2,6-di-t-butylphenol	C7H8O	108-39-4&106-44-5	108	107	18.008	0.02	disinfectant	business/household
191	Chloroneb	C8H8Cl2O2	2675-77-6	206	191	18.022	0.01		
192	Crimidine	C7H10ClN3	535-89-7	172	156	18.025	0.01		
193	Acetamide, N-(2-phenylethyl)-	C10H13NO	877-95-2	163	104	18.028	0.01	leaching from tire	business/household
194	4-Bromo-2,6-dichloroaniline	C6H4BrCl2N	697-86-9	239	241	18.077	0.03	reagent	business/household
195	1-Naphthol	C10H8O	90-15-3	144	144	18.104	0.01		
196	Pentachlorobenzene	C6HCl5	608-93-5	248	250	18.192	0.01	intermediate for pentachloronitrobenzene	industry
197	2-Amino-6-nitrotoluene	C7H8N2O2	603-83-8	152	152	18.194	0.01		industry
198	2-Phenylphenol	C7H8O	95-48-7	108	108	18.209	0.01	disinfectant	business/household
199	4-Nitrophenol	C6H5NO3	100-02-7	139	139	18.266	0.01	intermediate in organic synthesis/fungcide	industry
200	2-Naphthol	C7H8O2	90-05-1	124	109	18.266	0.03	leaching from tire	business/household
201	Methyl dodecanoate	C13H26O2	111-82-0	214	87	18.294	0.01	fatty acid methy ester	business/household
202	Dibenzofuran	C12H8O	132-64-9	168	168	18.304	0.01	heat-transfer oils/intermediate in organic synthesis	industry
203	4-Methyl-3-nitrophenol	C7H7NO3	2042-14-0	153	136	18.368	0.01		industry
204	2,4-Dinitrotoluene	C7H6N2O4	121-14-2	182	165	18.391	0.01	intermediate in organic synthesis	industry
205	Metribuzin DADK	C8H14N4OS	21087-64-9	214	154	18.456	0.01	herbicide	agriculture
206	Isoprocarb	C11H15NO2	2631-40-5	193	121	18.487	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
207	1-Naphthylamine	C10H9N	134-32-7	143	143	18.546	0.01	reagent	business/household
208	2,3,5,6-&2,3,4,5-Tetrachlorophenol	C6H2Cl4O	935-95-5&4901-51-3	230	232	18.579	0.02		industry
209	4-Amino-2-nitrotoluene	C7H8N2O2	119-32-4	152	152	18.584	0.01		industry
210	Molinate	C9H17NOS	2212-67-1	187	126	18.597	0.01	herbicide	agriculture
211	Amitraz (deg)				162	18.633	0.01	other pesticide	agriculture
212	2,3,4,6-Tetrachlorophenol	C6H2Cl4O	58-90-2	230	232	18.716	0.01	fungicide	agriculture
213	2,6-Di-t-butyl-4-ethylphenol	C8H10O	105-67-9	122	107	18.777	0.01	intermediate in organic synthesis	industry
214	2-Naphthylamine	C10H9N	91-59-8	143	143	18.796	0.01	reagent	business/household
215	Aspirin	C9H8O4	50-78-2	180	120	18.809	0.01	PPCPs	business/household
216	4-n-Hexylphenol	C10H14O	1638-22-8	150	107	18.846	0.01	intermediate for liquid crystal	industry
217	1,4-&1,6-Dichloronaphthalene	C10H6Cl2	1825-31-6&2050-72-8	196	196	18.856	0.01	PCN	industry
218	XMC	C10H13NO2	2655-14-3	179	122	18.872	0.01	insecticide	agriculture
219	1,5-Dichloronaphthalene	C10H6Cl2	1825-30-5	196	196	18.911	0.01	PCN	industry
220	2,6-&1,7-Dichloronaphthalene	C10H6Cl2	2065-70-5&2050-73-9	196	196	19.021	0.01	PCN	industry
221	Diethyltoluamide	C12H17NO	134-62-3	191	119	19.082	0.01	PPCPs	business/household
222	PCB #3	4-	2051-62-9	188	188	19.253	0.01	PCB	industry
223	Omethoate	C5H12NO4PS	1113-02-6	213	156	19.255	0.01	insecticide	agriculture
224	Tecnazene	C6HCl4NO2	117-18-	259	261	19.277	0.01		
225	Diethyl phthalate	C14H14O4	84-66-2	246	149	19.285	0.01	plasticizer	business/household
226	2,4,6-Tri-tert-butylphenol	C18H30O	732-26-	262	247	19.322	0.01		

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
227	Fluorene	C13H10	86-73-7	166	166	19.326	0.01	PAH	industry
228	Crotamiton	C13H17NO	483-63-6	203	69	19.367	0.01	PPCPs	business/household
229	Xylylcarb	C10H13NO2 2425-10-		179	122	19.407	0.01		
230	5-Nitro-o-toluidine	C7H8N2O2	99-55-8	152	152	19.431	0.01	intermediate for dyes	industry
231	4-Chloro-2-nitroaniline	C6H5ClN2O2	89-63-4	172	172	19.438	0.03	intermediate in organic synthesis	industry
232	4-Nitroaniline	C6H6N2O2	100-01-6	138	138	19.449	0.01	intermediate in organic synthesis	industry
233	n-C16H34	C16H34	544-76-3	226	85	19.469	0.01	petroleum	business/household
234	2,6-Dibromo-4-chloroaniline	C6H4Br2ClN	874-17-9	283	285	19.47	0.03	reagent	business/household
235	Ethenzamide	C9H11NO2	738-73-8	165	120	19.472	0.01	PPCPs	business/household
236	4-Chlorophenylphenyl ether	C12H9ClO	7005-72-3	204	204	19.478	0.01	dielectric fluid	business/household
237	4-tert-Octylphenol	C12H10O	92-69-3	170	170	19.492	0.01	intermediate in organic synthesis	industry
238	Fenobucarb	C12H17NO2	3766-81-2	207	150	19.537	0.01	insecticide	agriculture
239	Propachlor	C11H14ClNO	1918-16-7	211	120	19.546	0.01	herbicide	agriculture
240	2-Methyl-4,6-dinitrophenol	C7H6N2O5	534-52-1	198	198	19.555	0.01	pesticide/intermediate for dyes	industry
241	Tris(2-chloroethyl)phosphite	C6H12Cl3O3P	140-08-9	268	233	19.565	0.01	fire retardant	business/household
242	Propoxur	C11H15NO3	114-26-1	209	110	19.565	0.01	insecticide	agriculture
243	2-Nitronaphthalene	C10H7NO2	581-89-5	173	173	19.593	0.01	PAH	industry
244	2-(Methylthio)-benzothiazol	C8H7NS2	615-22-5	181	181	19.597	0.03	leaching from tire	business/household
245	Ibuprofen	C13H18O2	15687-27-1	206	161	19.685	0.01	PPCPs	business/household
246	Chlorethoxyfos	C6H11Cl4O3PS	54593-83-8	334	153	19.685	0.02	insecticide	agriculture
247	3,4,5-Trichlorophenol	C6H3Cl3O	609-19-8	196	196	19.695	0.01	intermediates in the synthesis of dyes, pigments, and phenolic resins	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
248	Demeton-S-methyl	C6H15O3PS2	919-86-8	230	142	19.76	0.01	insecticide	agriculture
249	PCB #4&10	22'-&26-	13029-08-8&33146-45-1	222	222	19.771	0.02	PCB	industry
250	Methyl tridecanoate	C14H28O2	1731-88-0	228	87	19.803	0.01	fatty acid methy ester	business/household
251	Diphenylamine	C12H11N	122-39-4	169	169	19.82	0.01	intermediate for dyes & rubber	industry
252	Ethoprophos	C8H19O2PS2	13194-48-4	242	158	19.972	0.01	insecticide	agriculture
253	Cycloate	C11H21NOS	1134-23-2	215	154	20.01	0.01	herbicide	agriculture
254	2,4,6-Tribromophenol	C6H3Br3O	118-79-6	328	332	20.044	0.03	intermediate for resin	industry
255	Tributyl phosphate	C12H27O4P	126-73-8	266	99	20.067	0.01	fire retardant	business/household
256	Phenmedipham deg.				167	20.094	0.25		
257	5-Bromoindole	C8H6BrN	10075-50-0	195	195	20.153	0.01		industry
258	Ethalfluralin	C13H14F3N3O4	55283-68-6	333	316	20.16	0.01	herbicide	agriculture
259	Naled	C4H7Br2Cl2O4P	300-76-5	378	109	20.223	0.01	insecticide	agriculture
260	1-Nitronaphthalene	C10H7NO2	86-57-7	173	173	20.233	0.01	PAH	industry
261	Benzaldehyde, 4-hydroxy-3,5-dimethoxy-	C9H10O4	134-96-3	182	182	20.241	0.01	leaching from tire	business/household
262	Dibenzylether	C14H14O	103-50-4	198	91	20.259	0.03	solvent	industry
263	Dichlofluanid metabolite	C13H24N4O3S	41483-43-6	316	273	20.267	0.01	fungicide	agriculture
264	Chlorpropham	C10H12ClNO2	101-21-3	213	213	20.296	0.01	herbicide	agriculture
265	2,6-Dichlorobenzamid	C7H5Cl2NO	2008-58-4	190	173	20.338	0.01		
266	Flusilazole metabolite	C10H9Cl4NO2S	2425-06-1	347	79	20.356	0.01	fungicide	agriculture
267	Dicrotophos	C8H9O3PS	3811-49-2	237	127	20.357	0.01		
268	4-n-Heptylphenol	C15H24O	128-37-0	220	220	20.375	0.03	antioxidant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
269	Trifluralin	C13H16F3N3O4	1582-09-8	335	306	20.406	0.01	herbicide	agriculture
270	Dioxabenzofos(Salithion)	C8H9O3PS	3811-49-2	216	216	20.451	0.01		
271	Benfluralin	C13H16F3N3O4	1861-40-1	335	292	20.474	0.03	herbicide	agriculture
272	Bendiocarb	C11H13NO4	22781-23-3	223	151	20.493	0.01	insecticide	agriculture
273	2(3H)-Benzothiazolone	C7H5NOS	934-34-9	151	151	20.523	0.01	leaching from tire	business/household
274	Sulfotep	C8H20O5P2S2	3689-24-5	322	322	20.53	0.01		
275	Metribuzin DK	C8H14N4OS	21087-64-9	214	168	20.603	0.01	herbicide	agriculture
276	Monocrotophos	C7H14NO5P	6923-22-4	223	127	20.607	0.01	insecticide	agriculture
277	1,3,5-Trinitrobenzene	C6H3N3O6	99-35-4	213	213	20.702	0.01	vulcanization/reagent	business/household
278	Cadusafos	C10H23O2PS2	95465-99-9	270	159	20.709	0.01	insecticide	agriculture
279	Pencycron	C12H13NO2S	5234-68-4	235	235	20.717	0.01	fungicide	agriculture
280	2,4,6-Trinitrotoluene	C7H5N3O6	118-96-7	227	210	20.789	0.01	explosive	industry
281	Phorate	C7H17O2PS3	298-02-2	260	260	20.81	0.01	insecticide	agriculture
282	Phenacetin	C10H13NO2	62-44-2	179	179	20.855	0.01	PPCP	business/household
283	a-HCH	C6H6Cl6	319-84-6	288	219	20.886	0.01	insecticide	agriculture
284	n-C17H36	C17H36	629-78-7	240	85	20.913	0.01	petroleum/plant	business/household
285	4-Bromophenylphenyl ether	C12H9BrO	101-55-3	248	248	20.917	0.01	reagent	business/household
286	Hexachlorobenzene	C10H6N2OS2	2439-01-2	234	206	20.951	0.01	fungicide	agriculture
287	PCB #8	24'-	34883-43-7	222	222	20.977	0.01	PCB	industry
288	2,4,6-Tribromoaniline	C6H4Br3N	147-82-0	327	331	21.007	0.03	reagent	business/household
289	4-Phenylphenol	C14H22O	1806-26-4	206	107	21.108	0.01	nonionic detergent metabolite	business/household
290	Thiometon	C6H15O2PS3	640-15-3	246	88	21.115	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
291	Desmedipham	C16H16N2O4	13684-56-5	300	181	21.173	0.5		
292	2,6-Dichloro-4-nitroaniline	C6H4Cl2N2O2	99-30-9	206	206	21.206	0.03	intermediate in organic synthesis	industry
293	Dicloran	C8H8Cl2O2	2675-77-6	206	191	21.227	0.01	fungicide	agriculture
294	Methyl myristate	C15H30O2	124-10-7	242	87	21.235	0.01	fatty acid methy ester	business/household
295	Dimethoate	C5H12NO3PS2	60-51-5	229	125	21.244	0.01	insecticide	agriculture
296	2,6-Diisopropylnaphthalene	C16H20	24157-81-1	212	197	21.326	0.03	PAH	industry
297	1,3,7-&1,4,6-Trichloronaphthalene	C10H5Cl3	55720-37-1 &2737-54-9	230	230	21.343	0.01	PCN	industry
298	Ethoxyquin	C8Cl4N2	1897-45-6	264	266	21.363	0.01	fungicide	agriculture
299	Furilazole	C11H13Cl2NO3	121776-33-8	277	220	21.408	0.01	herbicide	agriculture
300	Carbofuran	C12H15NO3	1563-66-2	221	164	21.441	0.01	insecticide	agriculture
301	Simazine (CAT)	C7H12ClN5	122-34-9	201	201	21.455	0.01	herbicide	agriculture
302	PCB #19	22'6-	38444-73-4	256	256	21.46	0.01	PCB	industry
303	1,2,4,5-Tetrabromobenzene	C6H2Br4	636-28-2	390	394	21.488	0.01		industry
304	b-HCH	C6H6Cl6	319-85-7	288	219	21.548	0.01	insecticide	agriculture
305	Phenazine	C12H8N2	92-82-0	180	180	21.558	0.01		industry
306	Pentachlorophenol	C6HCl5O	87-86-5	264	266	21.563	0.01	herbicide	agriculture
307	Nonylphenol	C14H22O	140-66-9	206	135	21.57	0.01	nonionic detergent metabolite	business/household
308	Swep	C8H7Cl2NO2	1918-18-9	219	187	21.575	0.01	herbicide	agriculture
309	Dimethipin	C6H10O4S2	55290-64-7	210	54	21.579	0.01	herbicide	agriculture
310	Atrazine	C8H14cLN5	1912-24-9	215	200	21.581	0.01	herbicide	agriculture
311	Pentachloronitrobenzene (Quintozene)	C6Cl5NO2	82-68-8	293	237	21.609	0.01		

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
312	Cyromazine	C6H10N6	66215-27-8	166	151	21.655	0.01	insecticide	agriculture
313	Clomazone	C12H14ClNO2	81777-89-1	239	204	21.669	0.01	herbicide	agriculture
314	4-Aminobiphenyl	C12H11N	92-67-1	169	169	21.672	0.01	reagent	business/household
315	Propazine	C9H16ClN5	139-40-2	229	214	21.69	0.01	herbicide	agriculture
316	Tris(2-chloroethyl) phosphate	C6H12Cl3O4P	115-96-8	284	249	21.696	0.01	fire retardant	business/household
317	3,5-di-tert-Butyl-4-hydroxybenzaldehyde	C15H22O2	1620-98-0	234	219	21.73	0.01	antioxidant/leaching from tire	business/household
318	Tolylfluanid metabolite	C15H18ClN3O	113096-99-4	291	222	21.764	0.01	fungicide	agriculture
319	Diazinon oxon	C12H21N2O4P	962-58-3	288	273	21.779	0.01	insecticide	agriculture
320	g-HCH	C6H6Cl6	58-89-9	288	219	21.782	0.01	insecticide	agriculture
321	Dibenzothiophene	C12H8S	132-65-0	184	184	21.788	0.03	petroleum	business/household
322	4-n-Octylphenol	C12H18O	2446-69-7	178	107	21.823	0.01		business/household
323	Dipropyl phthalate	C14H18O4	131-16-8	250	149	21.833	0.01	plasticizer	business/household
324	Cyanophos, CYAP	C9H10NO3PS	2636-26-2	243	243	21.936	0.01	insecticide	agriculture
325	Propetamphos	C10H20NO4PS	31218-83-4	281	138	21.94	0.01	insecticide	agriculture
326	Terbufos	C9H21O2PS3	13071-79-9	288	231	21.945	0.01	insecticide	agriculture
327	1,2,3-Trichloronaphthalene	C10H5Cl3	50402-52-3	230	230	21.981	0.01	PCN	industry
328	Fonofos	C10H15OPS2	994-22-9	246	246	22.027	0.01		
329	Phenoxathiin	C12H8OS	262-20-4	200	200	22.031	0.01		industry
330	Propyzamide	C12H11Cl2NO	23950-58-5	255	173	22.031	0.01	herbicide	agriculture
331	2,2'-Dibromobiphenyl (BB-4)	C12H8Br2	59080-37-4	310	312	22.061	0.01	fire retardant	business/household
332	Pyroquilon	C14H15N3	121552-61-2	225	224	22.068	0.01	fungicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
333	PCB #18	22'5-	37680-65-2	256	256	22.085	0.01	PCB	industry
334	Diazinon	C12H21N2O3PS	333-41-5	304	137	22.137	0.01	insecticide	agriculture
335	Phenanthrene	C14H10	85-01-8	178	178	22.166	0.01	PAH	industry
336	Chlorothalonil (TPN)	C9H11Cl2FN2O2S2	1085-98-9	332	224	22.179	0.01	fungicide	agriculture
337	Pyrimethanil	C12H13N3	53112-28-0	199	198	22.212	0.01		
338	PCB #15	44'-	2050-68-2	222	222	22.242	0.01	PCB	industry
339	Dichlone	C10H4Cl2O2	117-80-6	226	226	22.283	0.01	fungicide	agriculture
340	n-C18H38	C18H38	593-45-3	254	85	22.284	0.01	petroleum	business/household
341	Dinoseb	C10H12N2O5	88-85-7	240	211	22.295	0.01	insecticide	agriculture
342	Flufenoxuron dec2	C21H11ClF6N2O3	101463-69-8	488	331	22.309	0.01	insecticide	agriculture
343	Anthracene	C14H10	120-12-7	178	178	22.316	0.01	PAH	industry
344	1,4,5-Trichloronaphthalene	C10H5Cl3	2437-55-0	230	230	22.37	0.01	PCN	industry
345	Terbacil	C9H13ClN2O2	5902-51-2	216	161	22.385	0.01	herbicide	agriculture
346	Disulfoton	C8H19O2PS3	298-04-4	274	88	22.398	0.01	insecticide	agriculture
347	Prohydrojasmon	C15H26O3	158474-72-7	254	153	22.416	0.01	other pesticide	agriculture
348	Isazofos	C9H17ClN3O3PS	42509-80-8	313	161	22.431	0.01	insecticide	agriculture
349	Tefluthrin	C17H14ClF7O2	79538-32-2	418	177	22.517	0.01	insecticide	agriculture
350	d-HCH	C6H6Cl6	319-86-8	288	219	22.528	0.01	insecticide	agriculture
351	Etrimfos	C10H17N2O4PS	38260-54-7	292	292	22.547	0.01	insecticide	agriculture
352	Tri-allate	C10H16Cl3NOS	2303-17-5	303	268	22.592	0.01	herbicide	agriculture
353	Methyl pentadecanoate	C16H32O2	7132-64-1	256	87	22.597	0.01	fatty acid methy ester	business/household
354	MCPA-thioethyl (Phenothiol)	C11H13ClO2S	25319-90-8	244	244	22.62	0.01		

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
355	Flufenoxuron dec3	C21H11ClF6N2O3	101463-69-8	488	305	22.716	0.01	insecticide	agriculture
356	Tebupirimfos	C13H23N2O3PS	96182-53-5	318	318	22.743	0.01		
357	Pirimicarb	C11H18N4O2	23103-98-2	238	166	22.748	0.01	insecticide	agriculture
358	Iprobenfos (IBP)	C13H21O3PS	26087-47-8	288	204	22.784	0.01		
359	Oxabetrinil	C12H12N2O3	74782-23-3	232	73	22.791	0.01		
360	Tridemorph	C15H18Cl2N2O	139920-32-4	312	277	22.81	0.01	fungicide	agriculture
361	Diphenyldisulfide	C12H10S2	882-33-7	218	218	22.826	0.03		industry
362	Caffeine	C8H10N4O2	58-08-2	194	194	22.849	0.01	PPCPs	business/household
363	Benoxacor	C11H11Cl2NO2	98730-04-2	259	120	22.857	0.01	herbicide	agriculture
364	Carbazole	C12H9N	86-74-8	167	167	22.895	0.03	intermediate in organic synthesis	industry
365	PCB #54	22'66'-	15968-05-5	290	292	22.905	0.01	PCB	industry
366	Metribuzin DA	C8H13N3OS	35045-02-4	199	199	22.919	0.01	herbicide	agriculture
367	Ethiofencarb	C11H15NO2S	29973-13-5	225	107	22.939	0.01	insecticide	agriculture
368	MCPB-ethyl	C13H17ClO3	10443-70-6	256	115	22.982	0.01		
369	Diisobutyl phthalate	C16H22O4	84-69-5	278	149	23.06	0.03	plasticizer	business/household
370	Phosphamidon	C10H19ClNO5P	13171-21-6	299	127	23.093	0.01	insecticide	agriculture
371	Phenoxazine	C12H9NO	135-67-1	183	183	23.125	0.01		industry
372	Benfuresate	C12H16O4S	68505-69-1	256	163	23.132	0.01	herbicide	agriculture
373	1-Phenylnaphthalene	C16H12	605-02-7	204	204	23.138	0.03	PAH	industry
374	Fenitrothion oxon	C9H12NO6P	2255-17-6	261	244	23.155	0.01	insecticide	agriculture
375	Dichlofenthion, ECP	C10H13Cl2O3PS	97-17-6	314	279	23.168	0.01	insecticide	agriculture
376	Dimethenamid	C12H18ClNO2S	87674-68-8	275	154	23.169	0.01	herbicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
377	Propanil	C9H9Cl2NO	709-98-8	217	161	23.191	0.01	herbicide	agriculture
378	4-n-Nonylphenol	C13H20O	1987-50-4	192	107	23.209	0.01		business/household
379	Terbcarb (MBPMC)	C17H27NO2	1918-11-2	277	205	23.231	0.01	herbicide	agriculture
380	Acetochlor	C14H20ClNO2	34256-82-1	269	223	23.272	0.01	herbicide	agriculture
381	Chlorpyrifos-methyl	C7H7Cl3NO3PS	5598-13-0	321	286	23.309	0.01	insecticide	agriculture
382	Bromobutide	C15H22BrNO	74712-19-9	311	119	23.315	0.01	herbicide	agriculture
383	Metribuzin	C8H14N4OS	21087-64-9	214	198	23.318	0.01	herbicide	agriculture
384	PCB #28	244'-	7012-37-5	256	256	23.334	0.01	PCB	industry
385	Oxpoconazole-formyl	C15H18Cl2N2O	139920-32-4	312	277	23.386	0.01	fungicide	agriculture
386	Vinclozolin	C11H8Cl2N2O	62865-36-5	254	254	23.396	0.01	fungicide	agriculture
387	3-Hydroxycarbofuran 2				137	23.434	0.01	insecticide	agriculture
388	o-Terphenyl	C18H14	84-15-1	230	230	23.459	0.01	storage and transfer agents /intermediate for resin	industry
389	Methyl parathion	C8H10NO5PS	298-00-0	263	263	23.467	0.01	insecticide	agriculture
390	Tolclofos-methyl	C6H4Cl2N2O2	99-30-9	206	206	23.492	0.01	fungicide	agriculture
391	Simeconazole	C14H21NO4	87130-20-9	267	225	23.501	0.01	fungicide	agriculture
392	Alachlor	C14H20ClNO2	15972-60-8	269	188	23.502	0.01	herbicide	agriculture
393	Spiroxamine 1	C19H17Cl2N3O3	119446-68-3	405	265	23.505	0.01	fungicide	agriculture
394	PCB #33	2'34-	38444-86-9	256	256	23.536	0.01	PCB	industry
395	1,2,5,7-&1,2,4,6-&1,2,4,7 -Tetrachloronaphthalene	C10H4Cl4	67922-23-0&51570 -45-7&67922-21-8	264	266	23.537	0.01	PCN	industry
396	2,4-Diamino-6-nitrotoluene	C7H9N3O2	6629-29-4	167	167	23.55	0.1	explosive	industry
397	n-C19H40	C19H40	629-92-5	268	85	23.591	0.01	petroleum/plant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
398	Simetryn	C8H15N5S	1014-70-6	213	213	23.601	0.01	herbicide	agriculture
399	Carbaryl	C12H11NO2	63-25-2	201	144	23.613	0.01	insecticide	agriculture
400	Methyl palmitoleate	C17H32O2	1120-25-8	268	236	23.625	0.01	fatty acid methy ester	business/household
401	Heptachlor	C10H5Cl7	76-44-8	370	272	23.66	0.01	insecticide	agriculture
402	Mefenoxam	C19H17Cl2N3O3	119446-68-3	405	265	23.683	0.01	fungicide	agriculture
403	Metalaxyl	C21H22ClNO4	110488-70-5	387	301	23.69	0.01	fungicide	agriculture
404	Ametryn	C9H17N5S	834-12-8	227	227	23.706	0.01	herbicide	agriculture
405	Fenchlorphos	C8H8Cl3O3PS	299-84-3	320	285	23.736	0.01	insecticide	agriculture
406	PCB #22	234'-	38444-85-8	256	256	23.741	0.01	PCB	industry
407	2-Methylphenanthrene	C15H12	2531-84-2	192	192	23.752	0.01	PAH	industry
408	Cinmethylin	C18H26O2	87818-31-3	274	105	23.777	0.01	herbicide	agriculture
409	Prometryn	C10H19N5S	7287-19-6	241	241	23.787	0.01	herbicide	agriculture
410	Diethyl-p-nitrophenyl phosphate	C10H14NO6P	311-45-5	275	109	23.796	0.03	insecticide, metabolite of parathion	agriculture
411	4-Amino-2,6-dinitrotoluene	C7H7N3O4	19406-51-0	197	180	23.823	0.01	explosive	industry
412	1,4,6,7-Tetrachloronaphthalene	C10H4Cl4	55720-43-9	264	266	23.843	0.01	PCN	industry
413	Dithiopyr	C15H16F5NO2S2	97886-45-8	401	286	23.845	0.01	herbicide	agriculture
414	Bensulide	C14H24NO4PS3	741-58-2	397	77	23.853	0.01	herbicide	agriculture
415	Methyl palmitate	C17H34O2	112-39-0	270	270	23.895	0.02	fatty acid methy ester	business/household
416	Demeton-S-methylsulphon	C6H15O5PS2	17040-19-6	262	169	23.916	0.01	insecticide	agriculture
417	4,5-Methylene-phenanthrene	C15H10	203-64-5	190	189	23.936	0.03	PAH	industry
418	Phenol, 4-(phenylamino)-	C12H11NO	122-37-2	185	185	23.971	0.01	leaching from tire	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
419	1-Methylphenanthrene	C15H12	832-69-9	192	192	24.054	0.01	PAH	industry
420	Pirimiphos-methyl	C11H20N3O3PS	29232-93-7	305	290	24.055	0.01	insecticide	agriculture
421	Terbutryn	C10H19N5S	886-50-0	241	226	24.101	0.01	herbicide	agriculture
422	2,4-Dibromodiphenyl ether (BDE-7)	C12H8Br2O		326	328	24.106	0.01	fire retardant	business/household
423	Fenitrothion (MEP)	C9H12NO5PS	122-14-5	261	277	24.107	0.01	insecticide	agriculture
424	1,2,3,5-Tetrachloronaphthalene	C10H4Cl4	53555-63-8	264	266	24.113	0.01	PCN	industry
425	2,4-Dinitroaniline	C6H5N3O4	97-02-9	183	183	24.127	0.01	intermediate for dyes	industry
426	Methiocarb	C11H15NO2S	2032-65-7	225	168	24.139	0.01	insecticide	agriculture
427	Spiroxamine 2	C21H22ClNO4	110488-70-5	387	301	24.162	0.01	fungicide	agriculture
428	Probenazole	C10H9NO3S	27605-76-1	223	130	24.173	0.01	other pesticide	agriculture
429	Ethofumesate	C13H18O5S	26225-79-6	286	286	24.174	0.01	herbicide	agriculture
430	Bromacil	C9H13BrN2O2	314-40-9	260	205	24.186	0.01	herbicide	agriculture
431	PCB #52	22'55'-	35693-99-3	290	292	24.19	0.01	PCB	industry
432	Dimetylvinphos 1	C10H10Cl3O4P	2274-67-1	330	295	24.219	0.01	insecticide	agriculture
433	Di-n-butyl phthalate	C16H22O4	84-74-2	278	149	24.272	0.01	plasticizer	business/household
434	PCB #49	22'45'-	41464-40-8	290	292	24.282	0.01	PCB	industry
435	Dichlofluanid	C9H11Cl2FN2O2S2	1085-98-9	332	224	24.283	0.01		
436	Propyphenazone	C14H18N2O	479-92-5	230	215	24.306	0.01	PPCPs	business/household
437	Esprocarb	C15H23NOS	85785-20-2	265	222	24.328	0.01	herbicide	agriculture
438	Quinoclamine	C10H6ClNO2	2797-51-5	207	172	24.358	0.01	herbicide	agriculture
439	Malathion	C10H19O6PS2	121-75-5	330	173	24.359	0.01	insecticide	agriculture
440	Benzamide, N-phenyl-	C13H11NO	93-98-1	197	197	24.367	0.01	leaching from tire	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
441	2-Mercaptobenzothiazole	C7H5NS2	149-30-4	167	167	24.41	0.01	vulcanization-accelerator/ leaching from tire	business/household
442	Metolachlor	C15H22ClNO2	51218-45-2	283	162	24.449	0.01	herbicide	agriculture
443	2-Amino-4,6-dinitrotoluene	C7H7N3O4	35572-78-2	197	180	24.467	0.01	explosive	industry
444	Chlorpyrifos	C9H11Cl3NO3PS	2921-88-2	349	314	24.499	0.01	insecticide	agriculture
445	PCB #104	22'466'-	56558-16-8	324	326	24.524	0.01	PCB	industry
446	N-Nitroquinoline-N-oxide	C9H6N2O3	56-57-5	190	190	24.541	0.01	reagent	business/household
447	Thiobencarb	C12H16ClNOS	28249-77-6	257	100	24.543	0.01	herbicide	agriculture
448	Dimetylvinphos 2	C10H10Cl3O4P	2274-67-1	330	295	24.55	0.01	insecticide	agriculture
449	Aldrin	C12H8Cl6	309-00-2	362	263	24.557	0.01	insecticide	agriculture
450	2,6-Diamino-4-nitrotoluene	C7H9N3O2	59229-75-3	167	167	24.569	0.1	explosive	industry
451	Diethofencarb	C12H11N	122-39-4	169	169	24.577	0.01	fungicide/leaching from tire	business/household
452	2,3,6,7-&1,2,4,8-Tetrachloronaphthalene	C10H4Cl4		264	266	24.598	0.01	PCN	industry
453	Anthraquinone	C14H8O2	84-65-1	208	208	24.601	0.03	fragrance/solvent	business/household
454	Chlorthal-dimethyl	C10H6Cl4O4	1861-32-1	330	301	24.612	0.01	herbicide	agriculture
455	Fenthion	C10H15O3PS2	55-38-9	278	278	24.613	0.01	insecticide	agriculture
456	2-Phenylnaphthalene	C16H12	612-94-2	204	204	24.621	0.03	PAH	industry
457	Cyanazine	C9H13ClN6	21725-46-2	240	225	24.628	0.01	herbicide	agriculture
458	PCB #44	22'35'-	41464-39-5	290	292	24.662	0.01	PCB	industry
459	Parathion	C10H14NO5PS	56-38-2	291	291	24.699	0.01	insecticide	agriculture
460	Fenpropimorph	C20H33NO	67306-03-0	303	128	24.712	0.01		
461	Isofenphos oxon	C15H24NO5P	31120-85-1	329	229	24.747	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
462	Tetraconazole	C14H15O2PS2	17109-49-8	310	173	24.781	0.01	fungicide	agriculture
463	Triadimefon	C14H19NO	91-53-2	217	202	24.782	0.01	fungicide	agriculture
464	Isocarbophos	C11H16NO4PS	24353-61-5	289	136	24.796	0.01		
465	Carbetamide	C12H16N2O3	16118-49-3	236	119	24.816	0.05		
466	1,2,5,8-&1,2,6,8-Tetrachloronaphthalene	C10H4Cl4	&67922-24-1	264	266	24.817	0.01	PCN	industry
467	PCB #37	344'-	38444-90-5	256	258	24.82	0.01	PCB	industry
468	Fenoprofen	C15H14O3	31879-05-7	242	197	24.838	0.01	PPCPs	business/household
469	n-C20H42	C20H42	112-95-8	282	85	24.839	0.01	petroleum	business/household
470	cis-10-Heptadecenoic acid methyl ester	C18H32O2	75190-82-8	282	250	24.879	0.01	fatty acid methy ester	business/household
471	Dicofol-deg	C14H9Cl5O	115-32-2	368	139	24.88	0.01	other pesticide	agriculture
472	Nitrothal-isopropyl	C5H5Cl3N2OS	2593-15-9	246	211	24.901	0.03	fungicide	agriculture
473	Fthalide	C8H2Cl4O2	27355-22-2	270	243	24.905	0.01		
474	Triphenylmethane	C19H16	519-73-3	244	244	24.909	0.01	intermediate for dyes	industry
475	Bentazone	C10H12N2O3S	25057-89-0	240	198	24.975	0.01	herbicide	agriculture
476	Methapyrilene	C14H19N3S	91-80-5	297	97	24.982	0.01	PPCP	business/household
477	Bromophos	C8H8BrCl2O3PS	2104-96-3	364	331	25.039	0.01	insecticide	agriculture
478	3,6-Dimethylphenanthrene	C16H14	1576-67-6	206	206	25.058	0.03	PAH	industry
479	Diphenamid	C16H17NO	957-51-7	239	167	25.064	0.01	herbicide	agriculture
480	Fosthiazate 1	C9H18NO3PS2	98886-44-3	283	195	25.082	0.01	other pesticide	agriculture
481	Methyl heptadecanoate	C18H36O2	1731-92-6	284	284	25.135	0.01	fatty acid methy ester	business/household
482	Fosthiazate 2	C9H18NO3PS2	98886-44-3	283	195	25.143	0.01	other pesticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
483	Isopropalin	C15H23N3O4	33820-53-0	309	280	25.177	0.01	herbicide	agriculture
484	Thiamethoxam deg.				212	25.196	0.1		
485	Fluazinam	C17H17N3OS	161326-34-7	311	238	25.246	0.01	fungicide	agriculture
486	1,4,5,8-Tetrachloronaphthalene	C10H4Cl4	3432-57-3	264	266	25.256	0.01	PCN	industry
487	Pendimethalin	C13H19N3O4	40487-42-1	281	252	25.313	0.01	herbicide	agriculture
488	Chlorfenvinphos E	C12H14Cl3O4P	18708-86-6	358	267	25.322	0.01	insecticide	agriculture
489	Cyprodinil	C17H12Cl2N2O	60168-88-9	330	219	25.342	0.01	fungicide	agriculture
490	Fipronil	C12H4Cl2F6N4OS	120068-37-3	436	367	25.436	0.01	insecticide	agriculture
491	Penconazole	C19H17ClN4	114369-43-6	336	198	25.471	0.01	fungicide	agriculture
492	Dimethametryn	C11H21N5S	22936-75-0	255	212	25.49	0.01	herbicide	agriculture
493	Heptachlor epoxide (B)	C10H5Cl7O	1024-57-3	386	353	25.524	0.01	insecticide	agriculture
494	Tolylfluanid	C10H13Cl2FN2O2S2	731-27-1	346	145	25.531	0.01	fungicide	agriculture
495	Oxychlordane	C10H4Cl8O	27304-13-8	420	387	25.537	0.02	insecticide	agriculture
496	Isofenphos	C15H24NO4PS	25311-71-1	345	213	25.553	0.01	insecticide	agriculture
497	Pyrifenox Z	C14H12Cl2N2O	88283-41-4	294	256	25.555	0.01	fungicide	agriculture
498	Phenothiazine	C12H9NS	92-84-2	199	199	25.561	0.01	intermediate in organic synthesis	industry
499	PCB #74	244'5-	32690-93-0	290	292	25.562	0.01	PCB	industry
500	Ethychlozate	C11H11ClN2O2	27512-72-7	238	165	25.571	0.1		
501	Methyl dymron	C17H20N2O	42609-73-4	268	107	25.576	0.01	herbicide	agriculture
502	1,3-Dicyclohexylurea	C13H24N2O	2387-23-7	224	224	25.577	0.01	leaching from tire	business/household
503	Chlorfenvinphos Z	C12H14Cl3O4P	18708-87-7	358	267	25.578	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
504	Allethrin 1	C19H26O3	584-79-2	302	123	25.583	0.01	insecticide	agriculture
505	Tetryl	C7H5N5O8	479-45-8	287	194	25.588	0.01	explosive	industry
506	Mecarbam	C10H20NO5PS2	2595-54-2	329	131	25.636	0.01		
507	PCB #70	23'4'5-	32598-11-1	290	292	25.672	0.01	PCB	industry
508	PCB #95	22'35'6-	38379-99-6	324	326	25.705	0.01	PCB	industry
509	Phenthoate	C12H17O4PS2	2597-03-7	320	274	25.713	0.01	insecticide	agriculture
510	Diclocymet 1	C15H18Cl2N2O2	115852-48-7	328	293	25.715	0.01	fungicide	agriculture
511	Allethrin 2 & Bioallethrin 1	C19H26O3	584-79-2	302	123	25.715	0.01	insecticide	agriculture
512	Thiabendazole	C20H33NO	67306-03-0	303	128	25.717	0.01	fungicide	agriculture
513	Fluoranthene	C16H10	206-44-0	202	202	25.721	0.01	PAH	industry
514	Quinalphos	C12H15N2O3PS	13593-03-8	298	146	25.728	0.01	insecticide	agriculture
515	gamma-Linolenic acid methyl ester	C19H32O2	301-00-8	292	292	25.737	0.01	fatty acid methy ester	business/household
516	Captan	C9H8Cl3NO2S	133-06-2	299	79	25.759	0.01	herbicide	agriculture
517	1,2,3,5,7-Pentachloronaphthalene	C10H3Cl5	53555-65-0	298	300	25.77	0.01	PCN	industry
518	Procymidone	C15H18N4	89269-64-7	254	239	25.786	0.01	fungicide	agriculture
519	Triadimenol 1	C13H4Cl2F6N4O4	79622-59-6	464	372	25.811	0.01	fungicide	agriculture
520	Triflumizole	C12H6F2N2O2	131341-86-1	248	248	25.843	0.01	fungicide	agriculture
521	Dimepiperate	C15H21NOS	61432-55-1	263	119	25.845	0.01	herbicide	agriculture
522	Folpet	C16H8Cl2FN5O	136426-54-5	375	340	25.877	0.01	fungicide	agriculture
523	4-Chloro-o-terphenyl	C18H13Cl		264	229	25.897	0.01		industry
524	Linolelaidic acid methyl ester	C19H34O2	2566-97-4	294	294	25.938	0.01	fatty acid methy ester	business/household
525	Ferimzone	C16H15F2N3Si	85509-19-9	315	233	25.958	0.01	fungicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
526	Methoprene	C19H34O3	40596-69-8	310	73	25.967	0.02	insecticide	agriculture
527	Naproxen	C14H14O3	22204-53-1	230	185	25.973	0.01	PPCPs	business/household
528	Triadimenol 2	C14H18ClN3O2	82200-72-4	295	168	25.997	0.01		
529	Linolenic acid methyl ester	C19H32O2	301-00-8	292	292	26.006	0.01	fatty acid methy ester	business/household
530	PCB #155	22'44'66'-	33979-03-2	358	360	26.013	0.01	PCB	industry
531	Oleic acid methyl ester	C19H36O2	112-62-9	296	264	26.019	0.01	fatty acid methy ester	business/household
532	n-C21H44	C21H44	629-94-7	296	85	26.03	0.01	petroleum/plant	business/household
533	Linoleic acid methyl ester	C19H34O2	112-63-0	294	294	26.033	0.01	fatty acid methy ester	business/household
534	Methidathion	C6H11N2O4PS3	950-37-8	302	145	26.069	0.01	insecticide	agriculture
535	Elaidic acid methyl ester	C19H36O2	1937-62-8	296	264	26.093	0.01	fatty acid methy ester	business/household
536	trans-Chlordane	C10H6Cl8	5103-74-2	406	373	26.11	0.01	insecticide	agriculture
537	Diclocymet 2	C13H7Cl2F3N2O4S	106917-52-6	414	179	26.11	0.02	fungicide	agriculture
538	Triclosan	C12H7Cl3O2	3380-34-5	288	290	26.118	0.01	PPCPs	business/household
539	Hexythiazox	C17H21ClN2O2S	78587-05-0	352	227	26.118	0.01	other pesticide	agriculture
540	Chinomethionat	C17H16F3NO2	66332-96-5	323	173	26.133	0.01	fungicide	agriculture
541	1,2,4,6,8-Pentachloronaphthalene	C10H3Cl5		298	300	26.154	0.01	PCN	industry
542	Propaphos	C13H21O4PS	7292-16-2	304	220	26.154	0.01		
543	o,p'-DDE	C14H8Cl4	3424-82-6	316	246	26.176	0.01	insecticide	agriculture
544	Pyrifenox E	C16H13F2N3O	76674-21-0	301	219	26.184	0.01	fungicide	agriculture
545	Tetrachlorvinphos	C10H9Cl4O4P	22248-79-9	364	329	26.216	0.01	insecticide	agriculture
546	Trichlamid	C9H4Cl3NO2S	133-07-3	295	260	26.247	0.02	fungicide	agriculture
547	Paclobutrazol	C15H20ClN3O	76738-62-0	293	236	26.26	0.01	other pesticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
548	PCB #101	22'455'-	37680-73-2	324	326	26.282	0.01	PCB	industry
549	Butachlor	C17H26ClNO2	23184-66-9	311	176	26.3	0.01	herbicide	agriculture
550	Benzidine	C12H12N2	92-87-5	184	184	26.31	0.01	intermediate for dyes	industry
551	Stearic acid methyl ester	C19H38O2	112-61-8	298	298	26.322	0.01	fatty acid methy ester	business/household
552	Fenothiocarb	C13H19NO2S	62850-32-2	253	160	26.35	0.01	other pesticide	agriculture
553	Pyrene	C16H10	129-00-0	202	202	26.362	0.01	PAH	industry
554	PCB #99	22'44'5-	38380-01-7	324	326	26.388	0.01	PCB	industry
555	Endosulfan I	C9H6Cl6O3S	959-98-8	404	241	26.414	0.01	insecticide	agriculture
556	cis-Chlordane	C10H6Cl8	5103-71-9	406	373	26.421	0.01	insecticide	agriculture
557	Mepanipyrim	C8H2Cl4O2	27355-22-2	270	243	26.468	0.01	fungicide	agriculture
558	Ditalimfos	C17H20ClN3O2	123572-88-3	333	157	26.47	0.01	fungicide	agriculture
559	9-Nitrophenanthrene	C14H9NO2	954-46-1	223	165	26.48	0.01	PAH	industry
560	Butamifos	C13H21N2O4PS	36335-67-8	332	286	26.488	0.01	herbicide	agriculture
561	trans-Nonachlor	C10H5Cl9	5103-73-1	440	409	26.49	0.1	insecticide	agriculture
562	2,5-Dichloro-o-terphenyl	C18H12Cl2		298	228	26.504	0.01		industry
563	PCB #119	23'44'6-	56558-17-9	324	326	26.518	0.01	PCB	industry
564	Dipentyl phthalate	C18H26O4	131-18-0	306	149	26.537	0.01	plasticizer	business/household
565	9-Nitroanthracene	C14H9NO2	602-60-8	223	223	26.538	0.01	PAH	industry
566	Flutriafol	C16H13F2N3O	76674-21-0	301	219	26.548	0.01		
567	Fenamiphos	C13H22NO3PS	22224-92-6	303	303	26.589	0.01	other pesticide	agriculture
568	Napropamide	C6Cl6	118-74-1	282	284	26.612	0.01	fungicide	agriculture
569	N-Phenyl-1-naphthylamine	C16H13N	90-30-2	219	219	26.622	0.03	antioxidant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
570	Mefenamic Acid	C15H15NO2	61-68-7	241	223	26.625	0.01	PPCPs	business/household
571	TCMTB	C14H17Cl2N3O	79983-71-4	313	214	26.647	0.01	fungicide	agriculture
572	Flutolanil	C4H5NO2	10004-44-1	99	99	26.674	0.01	fungicide	agriculture
573	Chlorfenson	C12H8Cl2O3S	80-33-1	302	175	26.705	0.01		
574	Imazalil	C14H14Cl2N2O	35554-44-0	296	215	26.71	0.01	fungicide	agriculture
575	m-Terphenyl	C18H14	92-06-8	230	230	26.713	0.01	storage and transfer agents /intermediate for resin	industry
576	Fludioxonil	C17H13Cl3N4S	86598-92-7	410	125	26.713	0.01	fungicide	agriculture
577	Metominostrobin E	C13H21O3PS	26087-47-8	288	204	26.726	0.01	fungicide	agriculture
578	1,2,4,5,6-Pentachloronaphthalene	C10H3Cl5		298	300	26.729	0.01	PCN	industry
579	Hexaconazole	C13H13Cl2N3O3	36734-19-7	329	314	26.731	0.01	fungicide	agriculture
580	Prothiofos	C11H15Cl2O2PS2	34643-46-4	344	309	26.759	0.01	insecticide	agriculture
581	2-Hydroxy-4-methoxy-4'-methyl-benzophenone	C15H14O4		258	241	26.782	0.01		industry
582	Pretilachlor	C17H26ClNO2	51218-49-6	311	162	26.789	0.01	herbicide	agriculture
583	Isoprothiolane	C12H18O4S2	50512-35-1	290	118	26.794	0.01		
584	Tricyclazole	C9H7N3S	41814-78-2	189	189	26.798	0.01	fungicide	agriculture
585	Isoxathion oxon	C13H16NO5P	32306-29-9	297	161	26.808	0.01	insecticide	agriculture
586	Bisphenol A	C10H14O	99-71-8	150	121	26.83	0.01		business/household
587	1,2,4,7,8-Pentachloronaphthalene	C10H3Cl5		298	300	26.842	0.01	PCN	industry
588	Profenofos	C11H15BrClO3PS	41198-08-7	372	337	26.873	0.01	insecticide	agriculture
589	PCB #87	22'345'-	38380-02-8	324	326	26.904	0.01	PCB	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
590	1,2,3,5,8-&1,2,3,6,8-Pentachloronaphthalene	C10H3Cl5		298	300	26.922	0.01	PCN	industry
591	PCB #81	344'5-	70362-50-4	290	292	26.937	0.01	PCB	industry
592	p,p'-DDE	C14H8Cl4	72-55-9	316	246	26.96	0.01	insecticide	agriculture
593	Uniconazole P	C15H18ClN3O	83657-17-4	291	234	26.972	0.01	other pesticide	agriculture
594	Oxadiazon	C15H18Cl2N2O3	19666-30-9	344	258	26.975	0.01	herbicide	agriculture
595	Thifluzamide	C18H19NO4	143390-89-0	313	116	26.992	0.01	fungicide	agriculture
596	Flamprop-methyl	C17H15ClFNO3	52756-25-9	335	105	27.019	0.01	herbicide	agriculture
597	Tribufos	C12H27OSP3	78-48-8	314	169	27.035	0.01	other pesticide	agriculture
598	Myclobutanil	C15H21NO4	70630-17-0	279	206	27.053	0.01	fungicide	agriculture
599	Dieldrin	C12H8Cl6O	60-57-1	378	79	27.067	0.01	insecticide	agriculture
600	Oxyfluorfen	C15H11ClF3NO4	42874-03-3	361	252	27.098	0.01	herbicide	agriculture
601	Flusilazole	C14H13N3	110235-47-7	223	222	27.102	0.01	fungicide	agriculture
602	PCB #110	233'4'6-	38380-03-9	324	326	27.111	0.01	PCB	industry
603	o,p'-DDD	C14H10Cl4	53-19-0	318	235	27.113	0.01	insecticide	agriculture
604	Bupirimate	C17H19NO2	55814-41-0	269	119	27.129	0.01	fungicide	agriculture
605	p-Terphenyl	C18H14	92-94-4	230	230	27.147	0.01	storage and transfer agents/ intermediate for resin	industry
606	1,2,4,5,8-Pentachloronaphthalene	C10H3Cl5		298	300	27.149	0.01	PCN	industry
607	Buprofezin	C16H23N3OS	69327-76-0	305	105	27.152	0.01	insecticide	agriculture
608	Difenzoquat metilsulfate	C18H20N2O4S	43222-48-6	360	234	27.153	0.02	herbicide	agriculture
609	Amino-chlornitrofen	C12H8Cl3NO	26306-61-6	287	289	27.164	0.01	herbicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
610	Kresoxim methyl	C15H21NO4	57837-19-1	279	206	27.166	0.01	fungicide	agriculture
611	Imazamethabenz-methyl	C16H20N2O3	81405-85-8	288	256	27.171	0.01	herbicide	agriculture
612	n-C22H46	C22H46	629-97-0	310	85	27.172	0.01	petroleum	business/household
613	Carboxin	C16H16N2O3	133408-50-1	284	191	27.179	0.01	fungicide	agriculture
614	PCB #77	33'44'-	32598-13-3	290	292	27.18	0.01	PCB	industry
615	Diclobutrazol	C16H16N2O3	133408-50-1	284	191	27.202	0.01	fungicide	agriculture
616	Metominostrobin Z	C15H17ClN4	88671-89-0	288	179	27.203	0.01	fungicide	agriculture
617	Azaconazole	C17H21NO2	15299-99-7	271	128	27.209	0.01	fungicide	agriculture
618	Pyrethrin 1		8003-34-7		123	27.224	0.01	insecticide	agriculture
619	Triclopyr	C7H4Cl3NO3	55335-06-3	255	212	27.233	0.01	herbicide	agriculture
620	Chlorfenapyr	C15H11BrClF3N2O	122453-73-0	406	59	27.344	0.01	insecticide	agriculture
621	PCB #151	22'355'6-	52663-63-5	358	360	27.374	0.01	PCB	industry
622	Cyflufenamid	C14H17NO6	10552-74-6	295	236	27.384	0.01	fungicide	agriculture
623	4-Nitrophenanthrene	C14H9NO2	82064-15-1	223	223	27.405	0.01	PAH	industry
624	Isoxathion	C13H16NO4PS	18854-01-8	313	177	27.424	0.01	insecticide	agriculture
625	Cyproconazole	C14H18N2O4	77732-09-3	278	163	27.472	0.01	fungicide	agriculture
626	4-Dimethylaminoazobenzene	C14H15N3	60-11-7	225	225	27.508	0.01	reagent	business/household
627	Fenoxanil	C15H18Cl2N2O2	115852-48-7	328	293	27.52	0.01		
628	Nitrofen (NIP)	C12H7Cl2NO3	1836-75-5	283	283	27.532	0.03	herbicide	agriculture
629	Endrin	C12H8Cl6O	72-20-8	378	263	27.568	0.01	insecticide	agriculture
630	2,3-Benzofluorene	C17H12	243-17-4	216	216	27.652	0.03	PAH	industry
631	PCB #149	22'34'5'6-	38380-04-0	358	360	27.653	0.01	PCB	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
632	N-Phenyl-2-naphthylamine	C16H13N	135-88-6	219	219	27.659	0.03	antioxidant	business/household
633	PCB #123	2'344'5-	65510-44-3	324	326	27.667	0.01	PCB	industry
634	Pyriminobac-methyl Z	C17H19N3O6	136191-64-5	361	302	27.726	0.01	herbicide	agriculture
635	Chlorpropylate	C17H16Cl2O3	5836-10-2	338	251	27.748	0.01		
636	Chlorobenzilate	C16H14Cl2O3	510-15-6	324	251	27.751	0.01	other pesticide	agriculture
637	PCB #118	23'44'5-	31508-00-6	324	326	27.758	0.01	PCB	industry
638	Arachidonic acid methyl ester	C21H34O2	2566-89-4	318	150	27.788	0.01	fatty acid methy ester	business/household
639	Fensulfothion	C11H17O4PS2	115-90-2	308	293	27.789	0.01	other pesticide	agriculture
640	Endosulfan II	C9H6Cl6O3S	33213-65-9	404	195	27.828	0.01	insecticide	agriculture
641	Diniconazole	C15H17Cl2N3O2	83657-24-3	325	268	27.853	0.01		
642	cis-5,8,11,14,17-Eicosapentaenoic acid, methyl ester	C21H32O2	2734-47-6	316	119	27.854	0.01	fatty acid methy ester	business/household
643	Oxadixyl	C13H15Cl2N3	66246-88-6	283	248	27.91	0.01	fungicide	agriculture
644	3-Nitrophenanthrene	C14H9NO2	17024-19-0	223	223	27.949	0.01	PAH	industry
645	p,p'-DDD	C14H10Cl4	72-54-8	318	235	27.957	0.01	insecticide	agriculture
646	Ethion	C9H22O4P2S4	563-12-2	384	231	27.977	0.01	insecticide	agriculture
647	PCB #114	2344'5-	74472-37-0	324	326	27.991	0.01	PCB	industry
648	cis-8,11,14-Eicosatrienoic acid methyl ester	C21H36O2	21061-10-9	320	150	27.994	0.01	fatty acid methy ester	business/household
649	o,p'-DDT	C14H9Cl5	789-02-6	352	235	28.009	0.01	insecticide	agriculture
650	PCB #188	22'34'566'-	74487-85-7	392	394	28.032	0.01	PCB	industry
651	Pyrethrin 2		8003-34-7		123	28.087	0.01	insecticide	agriculture
652	Fluacrypyrim	C20H21F3N2O5	229977-93-9	426	204	28.147	0.01	other pesticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
653	Endrin aldehyde	C12H8Cl6O	7421-93-4	378	345	28.163	0.01	insecticide	agriculture
654	cis-11,14-Eicosadienoic acid methyl ester	C21H38O2	2463-02-7	322	322	28.207	0.01	fatty acid methy ester	business/household
655	PCB #153&168	22'44'55'-& 23'44'5'6-	35065-27-1& 41411-63-6	358	360	28.251	0.02	PCB	industry
656	n-C23H48	C23H48	638-67-5	324	85	28.265	0.01	petroleum/plant	business/household
657	cis-11-Eicosenoic acid methyl ester	C21H40O2	2390-09-2	324	292	28.269	0.01	fatty acid methy ester	business/household
658	cis-11,14,17-Eicosatrienoic cid methyl ester	C21H36O2	55682-88-7	320	95	28.277	0.01	fatty acid methy ester	business/household
659	Mepronil	C19H21ClN2O	66063-05-6	328	125	28.288	0.01	fungicide	agriculture
660	1,2,3,4,6,7-Hexachloronaphthalene	C10H2Cl6	103426-96-6	332	334	28.295	0.01	PCN	industry
661	Triazophos	C12H16N3O3PS	24017-47-8	313	161	28.306	0.01	insecticide	agriculture
662	PCB #105	233'44'-	32598-14-4	324	326	28.342	0.01	PCB	industry
663	Sulprofos	C12H19O3PS2	38527-90-1	306	322	28.345	0.1		
664	Pyrethrin 3		8003-34-7		123	28.402	0.01	insecticide	agriculture
665	Tris(1,3-dichloro-2-propyl) phosphate	C9H15Cl6O4P	13674-87-8	428	381	28.432	0.03	fire retardant	business/household
666	Azamethiphos	C9H10ClN2O5PS	35575-96-3	324	215	28.443	0.01	insecticide	agriculture
667	Famphur	C10H16NO5PS2	52-85-7	325	218	28.468	0.01	insecticide	agriculture
668	Isoxadifen-ethyl	C18H17NO3	163520-33-0	295	182	28.488	0.02	herbicide	agriculture
669	Carfentrazone-ethyl	C15H14Cl2F3N3O3	128639-02-1	411	312	28.504	0.01	herbicide	agriculture
670	Benalaxyl	C6Cl5NO2	82-68-8	293	237	28.523	0.01	fungicide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
671	Chlornitrofen (CNP)	C12H6Cl3NO3	1836-77-7	317	317	28.524	0.03	herbicide	agriculture
672	Arachidic acid methyl ester	C21H42O2	1120-28-1	326	326	28.547	0.01	fatty acid methy ester	business/household
673	Carbophenothion	C11H16ClO2PS3	786-19-6	342	121	28.593	0.01		
674	Norflurazon	C12H9ClF3N3O	27314-13-2	303	303	28.596	0.01	herbicide	agriculture
675	2,2',5,5'-Tetrabromobiphenyl (BB-52)	C12H6Br4		466	310	28.612	0.01	fire retardant	business/household
676	Cyanofenphos	C15H14NO2PS	13067-93-1	303	169	28.624	0.05		
677	Trifloxystrobin	C15H16Cl3N3O2	67747-09-5	375	180	28.633	0.01	fungicide	agriculture
678	Edifenphos	C13H11Cl2NO2	32809-16-8	283	283	28.656	0.01	fungicide	agriculture
679	Propiconazole 1	C9H20N2O2	25606-41-1	188	58	28.672	0.01	fungicide	agriculture
680	Quinoxyfen	C15H17Cl2N3O2	60207-90-1	341	259	28.699	0.01	fungicide	agriculture
681	Diofenolan 1	C18H20O4	63837-33-2	300	300	28.702	0.01	insecticide	agriculture
682	1,2,3,5,7,8-Hexachloronaphthalene	C10H2Cl6		332	334	28.722	0.01	PCN	industry
683	Endosulfan sulfate	C9H6Cl6O4S	1031-07-8	420	272	28.723	0.01	insecticide	agriculture
684	Butyl benzyl phtalate	C19H20O4	85-68-7	312	149	28.734	0.01	plasticizer	business/household
685	Pyriminobac-methyl E	C17H19N3O6	136191-64-5	361	302	28.738	0.01	herbicide	agriculture
686	Lenacil	C13H18N2O2	2164-08-1	234	153	28.752	0.01	herbicide	agriculture
687	Chloridazon	C10H8ClN3O	1698-60-8	221	221	28.785	0.1		
688	Pyraflufen ethyl	C15H13Cl2F3N2O4	129630-17-7	412	412	28.79	0.01	herbicide	agriculture
689	Propiconazole 2	C15H17Cl2N3O2	60207-90-1	341	259	28.812	0.01	fungicide	agriculture
690	Carbamazepine	C15H12N2O	298-46-4	236	193	28.835	0.01	PPCPs	business/household
691	p,p'-DDT	C14H9Cl5	50-29-3	352	235	28.844	0.01	insecticide	agriculture
692	Diofenolan 2	C18H20O4	63837-33-2	300	300	28.854	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
693	1,2,4,5,6,8-&1,2,4,5,7,8-Hexachloronaphthalene	C10H2Cl6		332	334	28.857	0.01	PCN	industry
694	PCB #138&158	22'344'5'-&233'44'6-	35065-28-2& 74472-42-7	358	360	28.886	0.02	PCB	industry
695	EPN oxon	C14H14NO5P	2012-00-2	307	141	28.946	0.01	insecticide	agriculture
696	Hexazinone	C12H20N4O2	51235-04-2	252	171	28.947	0.01	herbicide	agriculture
697	PCB #178	22'33'55'6-	52663-67-9	392	394	28.994	0.01	PCB	industry
698	Thenylchlor	C16H18ClNO2S	96491-05-3	323	127	29.057	0.01	herbicide	agriculture
699	PCB #126	33'44'5-	57465-28-8	324	326	29.113	0.01	PCB	industry
700	Tebuconazole	C16H22ClN3O	107534-96-3	307	250	29.167	0.01		
701	Di(2-ethylhexyl)adipate	C22H42O4	103-23-1	370	129	29.173	0.01	plasticizer	business/household
702	PCB #187	22'34'55'6-	52663-68-0	392	394	29.196	0.01	PCB	industry
703	Diclofop-methyl	C16H14Cl2O4	51338-27-3	340	340	29.202	0.01	herbicide	agriculture
704	2-Acetylaminofluorene	C15H13NO	53-96-3	223	181	29.22	0.01	reagent	business/household
705	Propargite 1	C19H26O4S	2312-35-8	350	135	29.221	0.01	other pesticide	agriculture
706	Diflufenican	C19H11F5N2O2	83164-33-4	394	266	29.232	0.01	herbicide	agriculture
707	Propargite 2	C19H26O4S	2312-35-8	350	135	29.251	0.01	other pesticide	agriculture
708	Diclomezine	C14H20N3O5PS	13457-18-6	373	221	29.295	0.01	fungicide	agriculture
709	n-C24H50	C24H50	646-31-1	338	85	29.316	0.01	petroleum	business/household
710	PCB #183	22'344'5'6-	52663-69-1	392	394	29.324	0.01	PCB	industry
711	Nitralin	C13H19N3O6S	4726-14-1	345	316	29.345	0.25		
712	Captafol	C14H12Cl2N2O	88283-41-4	294	262	29.367	0.01	fungicide	agriculture
713	Piperonyl butoxide	C19H30O5	51-03-6	338	176	29.371	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
714	Benzo(c)phenenthrene	C18H12	195-19-7	228	228	29.375	0.01	PAH	industry
715	Bioresmethrin	C22H26O3	28434-01-7	338	123	29.437	0.01	insecticide	agriculture
716	PCB #128	2,2',3,3',4,4'-	38380-07-3	358	360	29.468	0.01	PCB	industry
717	Zoxamide	C14H12Cl2N2O	88283-41-4	294	262	29.501	0.01	fungicide	agriculture
718	Mefenpyr-diethyl	C16H18Cl2N2O4	135590-91-9	372	253	29.527	0.01	herbicide	agriculture
719	PCB #167	23'44'55'-	52663-72-6	358	360	29.528	0.01	PCB	industry
720	4-Chloro-p-terphenyl	C18H13Cl		264	264	29.537	0.01		industry
721	1,2,3,4,5,8-Hexachloronaphthalene	C10H2Cl6		332	334	29.555	0.01	PCN	industry
722	Heneicosanoic acid methyl ester	C22H44O2	6064-90-0	340	340	29.592	0.01	fatty acid methy ester	business/household
723	Pyributicarb	C18H22N2O2S	88678-67-5	330	165	29.617	0.01	herbicide	agriculture
724	Acetamiprid	C10H11ClN4	135410-20-7	222	152	29.719	0.01	insecticide	agriculture
725	Pyridaphenthion	C14H17N2O4PS	119-12-0	340	340	29.744	0.01	insecticide	agriculture
726	Iprodione	C12H13N3	53112-28-0	199	198	29.755	0.01	fungicide	agriculture
727	cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester	C23H34O2	301-01-9	342	119	29.805	0.01	fatty acid methy ester	business/household
728	Tris(2-ethylhexyl) phosphate	C24H51O4P	78-42-2	434	99	29.81	0.03	fire retardant /plasticizer	business/household
729	Endrin ketone	C12H8Cl6O	53494-70-5	378	317	29.824	0.01	insecticide	agriculture
730	PCB #177	22'33'4'56-	52663-70-4	392	324	29.848	0.01	PCB	industry
731	Tetramethrin-1		8003-34-7		164	29.851	0.05		
732	Bromuconazole-1	C11H11NO	57369-32-1	173	130	29.861	0.01	fungicide	agriculture
733	PCB #202	22'33'55'66'-	2136-99-4	426	430	29.9	0.01	PCB	industry
734	Phosmet	C11H12NO4PS2	732-11-6	317	160	29.908	0.01	insecticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
735	PCB #171	22'33'44'6-	52663-71-5	392	394	29.956	0.01	PCB	industry
736	EPN	C14H14NO4PS	2104-64-5	323	157	29.981	0.01	insecticide	agriculture
737	Bifenthrin	C23H22ClF3O2	82657-04-3	422	181	29.982	0.01	insecticide	agriculture
738	Benzo(a)anthracene	C18H12	56-55-3	228	228	29.997	0.01	PAH	industry
739	Bromopropylate	C17H16Br2O3	18181-80-1	426	341	30.011	0.01	other pesticide	agriculture
740	PCB #156	233'44'5-	38380-08-4	358	360	30.027	0.01	PCB	industry
741	Picolinafen	C19H12F4N2O2	137641-05-5	376	376	30.037	0.01	herbicide	agriculture
742	Piperophos	C14H28NO3PS2	24151-93-7	353	320	30.05	0.01	herbicide	agriculture
743	Tetramethrin-2		8003-34-7		164	30.051	0.01		
744	Sulfentrazone	C11H10Cl2F2N4O3S	122836-35-5	386	307	30.056	0.01	herbicide	agriculture
745	PCB #201	22'33'45'66'-	40186-71-8	426	430	30.084	0.01	PCB	industry
746	Fenoxycarb	C17H19NO4	72490-01-8	301	116	30.085	0.01	insecticide	agriculture
747	3,3'-Dichlorobenzidine	C12H10Cl2N2	91-94-1	252	252	30.089	0.03	intermediate for dyes	industry
748	Bifenazate	C17H20N2O3	149877-41-8	300	258	30.091	0.1		
749	Chrysene & Triphenylene	C18H12	218-01-9& 217-59-4	228	228	30.094	0.01	PAH	industry
750	Methoxychlor	C16H15Cl3O2	72-43-5	344	227	30.124	0.01	insecticide	agriculture
751	Flusulfamide	C15H8Cl2FNO	124495-18-7	307	237	30.126	0.01	fungicide	agriculture
752	PCB #157	233'44'5'-	69782-90-7	358	360	30.132	0.01	PCB	industry
753	4,4'-Methylene-bis(2-chloroaniline)	C13H12Cl2N2	101-14-4	266	266	30.15	0.03	intermediate for resin	industry
754	Etoxazole	C21H23F2NO2	153233-91-1	359	204	30.163	0.01	other pesticide	agriculture
755	Fenpropathrin	C22H23NO3	39515-41-8	349	181	30.209	0.01	other pesticide	agriculture

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
756	Fenamidone	C17H17N3OS	161326-34-7	311	238	30.217	0.01		
757	Dicofol	C14H9Cl5O	115-32-2	368	139	30.246	0.01	other pesticide	agriculture
758	6-Benzylaminopurine	C12H11N5	1214-39-7	225	225	30.286	0.01	other pesticide	agriculture
759	cis-13,16-Docosadienoic acid methyl ester	C23H42O2	61012-47-3	350	350	30.302	0.01	fatty acid methy ester	business/household
760	2,4-&2,5-Dichloro-p-terphenyl	C18H12Cl2		298	298	30.315	0.02		industry
761	n-C25H52	C25H52	629-99-2	352	85	30.325	0.01	petroleum/plant	business/household
762	Tebufenpyrad	C18H24ClN3O	119168-77-3	333	171	30.333	0.01	other pesticide	agriculture
763	Indanofan	C20H17ClO3	133220-30-1	340	174	30.334	0.01		
764	Erucic acid methyl ester	C23H44O2	1120-34-9	352	320	30.348	0.01	fatty acid methy ester	business/household
765	Anilofos	C13H19ClNO3PS2	64249-01-0	367	226	30.35	0.01	herbicide	agriculture
766	Bifenox	C14H9Cl2NO5	42576-02-3	340	341	30.366	0.01	herbicide	agriculture
767	Bromuconazole-2	C18H35NO2	118134-30-8	297	100	30.381	0.01	fungicide	agriculture
768	PCB #180	22'344'55'-	35065-29-3	392	324	30.381	0.01	PCB	industry
769	Etoxazole metabolite				246	30.403	0.05		
770	Benzanthrone	C17H10O	82-05-3	230	230	30.433	0.03	intermediate foe dyes	industry
771	Dicyclohexyl phthalate	C20H26O4	84-61-7	330	149	30.473	0.01	plasticizer	business/household
772	Clomeprop	C16H15Cl2NO2	84496-56-0	324	288	30.48	0.01		
773	Furametpyr	C18H35NO2	118134-30-8	297	100	30.484	0.01	fungicide	agriculture
774	PCB #191	233'44'5'6-	74472-50-7	392	394	30.486	0.01	PCB	industry
775	Phenothrin 1	C23H26O3	26002-80-2	350	183	30.544	0.01	insecticide	agriculture
776	2,2',4,4'-Tetrabromodiphenyl ether (BDE-47)	C12H6Br4O		482	326	30.549	0.01	fire retardant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
777	Iprodione metabolite	C9H6N2S3	21564-17-0	238	180	30.581	0.02	fungicide	agriculture
778	Behenic acid methyl ester	C23H46O2	929-77-1	354	354	30.598	0.01	fatty acid methy ester	business/household
779	Tetradifon	C12H6Cl4O2S	116-29-0	354	356	30.616	0.01	other pesticide	agriculture
780	Bis(2-ethylhexyl)phthalate	C24H38O4	117-81-7	390	149	30.626	0.01	plasticizer	business/household
781	Phenothrin 2	C23H26O3	26002-80-2	350	183	30.686	0.01	insecticide	agriculture
782	Pentoxazone	C17H17ClFNO4	110956-75-7	353	285	30.733	0.01	herbicide	agriculture
783	Phosalone	C12H15ClNO4PS2	2310-17-0	367	182	30.763	0.01	insecticide	agriculture
784	Pyrethrin 4		8003-34-7		107	30.776	0.01	insecticide	agriculture
785	Leptophos	C13H10BrCl2O2PS	21609-90-5	412	377	30.784	0.01		
786	Azinphos-methyl	C10H12N3O3PS2	86-50-0	317	160	30.861	0.01	insecticide	agriculture
787	PCB #169	33'44'55'-	32774-16-6	358	360	30.875	0.01	PCB	industry
788	3-Nitrofluoranthene	C16H9NO2	892-21-7	247	247	30.94	0.01	PAH	industry
789	Cyhalothrin 1	C23H19ClF3NO3	68085-85-8	449	181	30.987	0.01	insecticide	agriculture
790	Pyriproxyfen	C20H19NO3	95737-68-1	321	136	30.994	0.01	insecticide	agriculture
791	1,2,3,4,5,6,7-Heptachloronaphthalene	C10HCl7		366	368	31.005	0.01	PCN	industry
792	PCB #170	22'33'44'5-	35065-30-6	392	324	31.008	0.01	PCB	industry
793	2,4,6-Trichloro-p-terphenyl	C18H11Cl3		332	332	31.034	0.01		industry
794	Cyhalofop Butyl	C20H20FNO4	122008-85-9	357	256	31.048	0.01	herbicide	agriculture
795	Mefenacet	C16H14N2O2S	73250-68-7	298	192	31.059	0.01	herbicide	agriculture
796	Furametpyr metabolite	C16H22ClN3O	107534-96-3	307	250	31.072	0.01	fungicide	agriculture
797	PCB #199	22'33'455'6'-	52663-75-9	426	358	31.162	0.01	PCB	industry
798	1,2,3,4,5,6,8-Heptachloronaphthalene	C10HCl7	58863-15-3	366	368	31.163	0.01	PCN	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
799	Amitraz	C19H23N3	33089-61-1	293	293	31.167	0.01	other pesticide	agriculture
800	Cyhalothrin 2	C23H19ClF3NO3	68085-85-8	449	181	31.208	0.01	insecticide	agriculture
801	n-C26H54	C26H54	630-01-3	366	85	31.297	0.01	petroleum	business/household
802	Pyrazophos	C6HCl4NO2	117-18-0	259	261	31.416	0.01	fungicide	agriculture
803	Fenarimol	C13H11Cl2F4N3O	112281-77-3	371	336	31.427	0.01	fungicide	agriculture
804	Acrinathrin	C26H21F6NO5	101007-06-1	541	181	31.431	0.01	other pesticide	agriculture
805	1-Nitropyrene	C16H9NO2	5522-43-0	247	247	31.498	0.01	PAH	industry
806	Tricosanoic acid methyl ester	C24H48O2	2433-97-8	368	368	31.566	0.01	fatty acid methy ester	business/household
807	Azinphos-ethyl	C12H16N3O3PS2	2642-71-9	345	132	31.598	0.05		
808	PCB #189	233'44'55'-	36935-31-9	392	394	31.665	0.01	PCB	industry
809	Dialifos	C14H17cLNO4PS2	10311-84-9	394	208	31.676	0.25		
810	Squalane	C30H62	111-01-3	422	85	31.768	0.03	PPCPs/organism	business/household
811	Pyraclofos	C14H18ClN2O3PS	77458-01-6	360	360	31.769	0.01	insecticide	agriculture
812	Oryzalin	C12H18N4O6S	19044-88-3	346	317	31.831	0.01	herbicide	agriculture
813	Fenoxaprop-ethyl	C18H16ClNO5	66441-23-4	361	288	31.872	0.01	herbicide	agriculture
814	PCB #208	22'33'455'66'-	52663-77-1	460	392	31.889	0.01	PCB	industry
815	Tricresyl phosphate	C21H21O4P	1330-78-5	368	368	31.994	0.03	fire retardant/plasticizer	business/household
816	Tris(4-chlorophenyl)methane	C19H13Cl3	27575-78-6	348	311	32.007	0.01		industry
817	Spirodiclofen	C21H24Cl2O4	148477-71-8	410	71	32.016	0.01	other pesticide	agriculture
818	Bitertanol	C10H7N3S	148-79-8	201	201	32.112	0.01	fungicide	agriculture
819	Permethrin 1	C21H20Cl2O3	52645-53-1	390	183	32.209	0.01	insecticide	agriculture
820	n-C27H56	C27H56	593-49-7	380	85	32.235	0.01	petroleum/plant	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
821	Nervonic acid methyl ester	C25H48O2	2733-88-2	380	348	32.279	0.01	fatty acid methy ester	business/household
822	Coumaphos	C14H16ClO5PS	56-72-4	362	362	32.335	0.01	insecticide	agriculture
823	Fluquinconazole	C13H6Br2F6N2O2S	130000-40-7	526	194	32.347	0.01	fungicide	agriculture
824	Permethrin 2	C21H20Cl2O3	52645-53-1	390	183	32.37	0.01	insecticide	agriculture
825	PCB #194	22'33'44'55'-	35694-08-7	426	358	32.396	0.01	PCB	industry
826	Pyridaben	C19H25ClN2OS	96489-71-3	364	147	32.403	0.01	insecticide	agriculture
827	Prochloraz	C9H11Cl2O3PS	57018-04-9	301	265	32.406	0.01	fungicide	agriculture
828	Oxpoconazole-fumalate	C10H13Cl2FN2O2S2	731-27-1	346	238	32.455	0.01	fungicide	agriculture
829	PCB #205	233'44'55'6-	74472-53-0	426	358	32.476	0.01	PCB	industry
830	Di-n-octyl phthalate	C24H38O4	117-84-0	390	149	32.49	0.01	plasticizer	business/household
831	Lignoceric acid, methyl ester	C25H50O2	2442-49-1	382	382	32.5	0.01	fatty acid methy ester	business/household
832	Butafenacil	C20H18ClF3N2O6	134605-64-4	475	331	32.573	0.05		
833	Etobenzanid	C16H15Cl2NO3	79540-50-4	339	179	32.737	0.01	herbicide	agriculture
834	Cafenstrole	C16H22N4O3S	125306-83-4	350	100	32.742	0.01	herbicide	agriculture
835	Fenbuconazole	C19H17ClN4	114369-43-6	336	198	32.833	0.01		
836	Cyfluthrin 1	C22H18Cl2FNO3	68359-37-5	433	163	32.877	0.01	insecticide	agriculture
837	Cyfluthrin 2	C22H18Cl2FNO3	68359-37-5	433	163	33.001	0.01	insecticide	agriculture
838	Benzo(j&b)fluoranthene	C20H12	205-82-3&205-99-2	252	252	33.005	0.02	PAH	industry
839	7,12-Dimethylbenz(a)anthracene	C20H16	57-97-6	256	256	33.017	0.01	PAH	industry
840	Benzo(k)fluoranthene	C20H12	207-08-9	252	252	33.064	0.01	PAH	industry
841	Cyfluthrin 3	C22H18Cl2FNO3	68359-37-5	433	163	33.071	0.01	insecticide	agriculture
842	Bis(2-ethylhexyl) sebacate	C26H50O4	122-62-3	426	185	33.093	0.01	plasticizer	business/household

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
843	Octachloronaphthalene	C10Cl8	2234-13-1	400	404	33.127	0.01	PCN	industry
844	Cyfluthrin 4	C22H18Cl2FNO3	68359-37-5	433	163	33.127	0.01	insecticide	agriculture
845	2,3,5,6-Tetrachloro-p-terphenyl	C18H10Cl4		366	368	33.137	0.01		industry
846	n-C28H58	C28H58	630-02-4	394	85	33.138	0.01	petroleum	business/household
847	PCB #206	22'33'44'55'6-	40186-72-9	460	464		0.01	PCB	industry
848	2,4,4',6-Tetrachloro-p-terphenyl	C18H10Cl4		366	368	33.245	0.01		industry
849	7-Nitrobenz(a)anthracene	C18H11NO2	20268-51-3	273	273	33.275	0.01	PAH	industry
850	Cypermethrin 1	C22H19Cl2NO3	52315-07-8	415	163	33.283	0.01	insecticide	agriculture
851	Tris(4-chlorophenyl)methanol	C19H13Cl3O	3010-80-8	364	251	33.296	0.01		industry
852	Halfenprox	C24H23BrF2O3	111872-58-3	476	263	33.377	0.01	other pesticide	agriculture
853	Cypermethrin 2	C22H19Cl2NO3	52315-07-8	415	163	33.414	0.01	insecticide	agriculture
854	Cypermethrin 3	C22H19Cl2NO3	52315-07-8	415	163	33.479	0.01	insecticide	agriculture
855	Flucythrinate 1	C26H23F2NO4	70124-77-5	451	199	33.492	0.01	insecticide	agriculture
856	Quizalofop-ethyl	C19H17ClN2O4	76578-14-8	372	299	33.502	0.01	herbicide	agriculture
857	Cypermethrin 4	C22H19Cl2NO3	52315-07-8	415	163	33.532	0.01	insecticide	agriculture
858	Fenbuconazole lactone B	C14H16ClN3O2	43121-43-3	293	208	33.552	0.01	fungicide	agriculture
859	Cholestane	C27H48	481-21-0	372	217	33.645	0.1	non-natural steroid	Sterols
860	Benzo(e)pylene	C20H12	192-97-2	252	252	33.664	0.03	PAH	industry
861	Etofenprox	C25H28O3	80844-07-1	376	163	33.68	0.01	insecticide	agriculture
862	Flucythrinate 2	C26H23F2NO4	70124-77-5	451	199	33.736	0.01	insecticide	agriculture
863	PCB #209	22'33'44'55'66'-	2051-24-3	494	498	33.791	0.01	PCB	industry
864	Benzo(a)pyrene	C20H12	50-32-8	252	252	33.797	0.01	PAH	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
865	Silafluofen	C25H29FO2Si	105024-66-6	408	179	33.869	0.01	insecticide	agriculture
866	Fluridone	C19H14F3NO	59756-60-4	329	328	33.901	0.01	herbicide	agriculture
867	Fenbuconazole lactone A	C14H18ClN3O2	89482-17-7	295	168	33.94	0.01	fungicide	agriculture
868	Perylene	C20H12	198-55-0	252	252	33.999	0.03	PAH	industry
869	n-C29H60	C29H60	630-03-5	408	85	34.013	0.01	petroleum/plant	business/household
870	6-Nitrochrysene	C18H11NO2	7496-02-8	273	273	34.07	0.01	PAH	industry
871	Pyrimidifen	C20H28ClN3O2	105779-78-0	377	184	34.171	0.01	other pesticide	agriculture
872	Pyridate	C19H23ClN2O2S	55512-33-9	378	207	34.285	0.02	herbicide	agriculture
873	Flumioxazin	C19H15FN2O4	103361-09-7	354	354	34.334	0.01	herbicide	agriculture
874	Esfenvalerate 1	C25H22ClNO3	66230-04-4	419	225	34.385	0.01	insecticide	agriculture
875	Fenvalerate 1	C25H22ClNO3	51630-58-1	419	167	34.389	0.01	insecticide	agriculture
876	Pyraclostrobin	C14H18ClN3O2	82200-72-4	295	168	34.491	0.01	fungicide	agriculture
877	Fluvalinate 1	C26H22ClF3N2O3	69409-94-5	502	250	34.531	0.01	insecticide	agriculture
878	2,2',4,4',5,5'-Hexabromobiphenyl (BB-153)	C12H4Br6	59080-40-9	622	308	34.554	0.01	fire retardant	business/household
879	Fluvalinate 2	C26H22ClF3N2O3	69409-94-5	502	250	34.613	0.01	insecticide	agriculture
880	Esfenvalerate 2	C25H22ClNO3	66230-04-4	419	225	34.637	0.01	insecticide	agriculture
881	Fenvalerate 2	C25H22ClNO3	51630-58-1	419	167	34.64	0.01	insecticide	agriculture
882	3-Methylcholanthrene	C21H16	56-49-5	268	268	34.714	0.01	PAH	industry
883	n-C30H62	C30H62	638-68-6	422	85	34.857	0.02	petroleum	business/household
884	Difenoconazole 1	C13H16Cl3NO3	70193-21-4	339	148	34.932	0.01	fungicide	agriculture
885	Difenoconazole 2	C9H7N3S	41814-78-2	189	189	35.007	0.01	fungicide	agriculture
886	Pyrazoxyfen	C20H16Cl2N2O3	71561-11-0	402	105	35.024	0.01	herbicide	agriculture

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No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
887	Indoxacarb	C22H17ClF3N3O7	144171-61-9	528	150	35.111	0.25		
888	2,3,4,5,6-Pentachloro-p-terphenyl	C18H9Cl5		400	402	35.132	0.01		industry
889	Diclosulam	C13H10Cl2FN5O3S	145701-21-9	405	342	35.294	0.01	herbicide	agriculture
890	2,2',4,4',5,5'-Hexabromodiphenyl ether (BDE-153)	C12H4Br6O		638	484	35.296	0.01	fire retardant	business/household
891	Tralomethrin-deg	C22H19Br4NO3	66841-25-6	661	253	35.298	0.01	insecticide	agriculture
892	Deltamethrin	C22H19Br2NO3	52918-63-5	503	181	35.299	0.01	insecticide	agriculture
893	Flumiclorac-pentyl	C21H23ClFNO5	87546-18-7	423	423	35.465	0.01	herbicide	agriculture
894	Azoxystrobin	C19H39NO	81412-43-3	297	153	35.482	0.01	fungicide	agriculture
895	Coprostanol	C27H48O	360-68-9	388	233	35.58	0.01	facal steroid	Sterols
896	Dimethomorph E	C20H19F3N2O4	141517-21-7	408	116	35.633	0.01	fungicide	agriculture
897	Tecloftalam	C14H5Cl6NO3	76280-91-6	473	394	35.66	0.02	other pesticide	agriculture
898	n-C31H64	C31H64	630-04-6	437	85	35.676	0.02	petroleum/plant	business/household
899	Famoxadone	C15H15ClF3N3O	99387-89-0	345	278	35.715	0.01	other pesticide	agriculture
900	Tolfenpyrad	C21H22ClN3O2	129558-76-5	383	383	35.83	0.01	insecticide	agriculture
901	Cholesterol	C27H46O	57-88-5	386	386	35.879	0.1	plant/animal steroid	Sterols
902	1,2,5,6,9,10-Hexabromocyclododecane	C12H18Br6	3194-55-6	636	239	35.974	0.01	fire retardant	business/household
903	Cholestanol	C27H48O	80-97-7	388	233	35.979	0.1	animal steroid	Sterols
904	Dimethomorph Z	C12H9Cl2NO3	50471-44-8	285	285	36.012	0.01	other pesticide	agriculture
905	Imibenconazole	C14H16Cl3NO2	156052-68-5	335	187	36.374	0.01	other pesticide	agriculture
906	Indeno(1,2,3-cd)pyrene	C22H12	193-39-5	276	276	36.435	0.01	PAH	industry
907	n-C32H66	C32H66	4981-99-1	451	85	36.521	0.02	petroleum	business/household
908	Dibenzo(a,h)anthracene	C22H14	53-70-3	278	278	36.532	0.01	PAH	industry

Appendix 1 Cont'd

No	Name	Formula	CAS RN	MW	m/z	RT, min	IDL	Use/Origin	Class
909	Fluthiacet-methyl	C15H15ClFN3O3S2	117337-19-6	403	403	36.831	0.01	herbicide	agriculture
910	Benzo(ghi)perylene	C22H12	191-24-2	276	276	37.056	0.01	PAH	industry
911	Stigmasterol	C29H48O	83-48-7	412	412	37.077	0.1	plant steroid	Sterols
912	n-C33H68	C33H68	630-05-7	465	85	37.478	0.02	petroleum/plant	business/household
913	Temephos	C16H20O6P2S3	3383-96-8	466	466	37.518	0.01	insecticide	agriculture
914	beta-Sitosterol	C29H50O	83-46-5	414	414	37.704	0.1	plant steroid	Sterols