



ELSEVIER

Journal of Chromatography A, 754 (1996) 103–123

JOURNAL OF
CHROMATOGRAPHY A

Review

Occurrence and determination of pesticides in natural and treated waters

M. Biziuk^{a,*}, A. Przyjazny^b, J. Czerwinski^a, M. Wierowski^a

^aFaculty of Chemistry, Technical University of Gdansk, 11112 G. Narutowicza Street, 80-952 Gdansk, Poland

^bScience and Mathematics Department, GMI Engineering and Management Institute, 1700 West Third Ave., Flint, MI 48504, USA

Abstract

Pesticides as environmental pollutants are described in detail along with their sources and paths of entry into various elements of the environment. Comprehensive literature data on the concentration of these pollutants in natural and treated waters and wastewaters are discussed. A wide selection of isolation and preconcentration techniques for these pollutants in water is presented and discussed. An emphasis is put on solid-phase extraction. In the case of the authors' work, a more detailed description is given.

Keywords: Reviews; Environmental analysis; Water analysis; Pesticides; Polychlorinated biphenyls

Contents

1. Introduction	104
2. Sources of environmental pollution by pesticides	104
3. Occurrence of pesticides in surface waters	105
4. Methods of isolation and determination of pesticides in water	106
4.1. General problems of the determination of pesticides in water	106
4.2. Application of liquid–liquid extraction methods for the isolation of pesticides from the aqueous phase	107
4.3. Methods of isolation of organic compounds from the aqueous phase based on sorption on solid sorbents (solid-phase extraction)	108
4.3.1. XAD porous polymeric sorbents	108
4.3.2. Chemically modified silica gels	110
4.3.3. Tenax sorbents	113
4.4. Supercritical fluid extraction	113
4.4.1. Solid-phase extraction–supercritical fluid extraction	114
4.4.2. Direct extraction of organic compounds from water samples using SFE	114
5. Conclusions	114
Acknowledgments	115
References	115

*Corresponding author.

1. Introduction

Water, the substance so vital to humans and their well-being, the source and basis of life on earth, has recently become a serious hazard to our health and life, as well as to the life and health of the flora and fauna. Furthermore, desertification of large areas of the world is now observed and water, particularly clean water, is rapidly becoming a much desirable and increasingly expensive commodity. Intensive industrialization of the world results in an increased input of toxic inorganic and organic compounds to waters, drastically reducing the quality of surface waters which are a source of drinking water for a large part of the world population. Groundwater supply, which is basically non-renewable, is diminishing and, in addition, anthropogenic groundwater pollution has become a fact of life. A majority of anthropogenic water pollutants are toxic, dangerous not only to humans but also to animals and plants.

2. Sources of environmental pollution by pesticides

Anthropogenic organic compounds present the main hazard to life and health of humans and the flora and fauna. The number of known organic compounds is now estimated to be about 16 million [1], 2 million of which are the compounds produced by synthesis alone. Every year, approximately 250 000 new compounds are synthesized, about 1000 of which are manufactured on an industrial scale [2]. Presently, about 70 000 organic compounds are commercially available with an annual global production of 100–200 million tons. Approximately one-third of all organic compounds produced ends up in the environment, including water. Over 700 chemical compounds, including more than 600 organic compounds [3,4] many of which are biologically active, have been detected in some water samples.

Pesticides are particularly important pollutants among organic compounds as a result of their common use, persistence in the environment and toxicity. They are predominantly anthropogenic. Pesticides are a group of organic compounds hazardous to human health and life. They increase crop

yields by reducing the amount of a crop that is lost to pests and control diseases transmitted by insects. Pesticides are necessarily toxic, as they are used in agriculture, industry and households to kill weeds (herbicides), insects (insecticides), fungi (fungicides), rodents (rodenticides), mollusks (molluscicides), mites (acaricides), round worms (nematocides), aphids (aphicides) and eggs (ovicides). They are also used as fumigants, attractants or repellents [2,6–15].

Chemical structures of pesticides are very diversified, including for example organohalogen compounds, organophosphates, carbamates, triazines, phenol derivatives, etc. Presently, the number of compounds applied as pesticides is about several thousand and undergoes a continuous variation. The modern definition of a pesticide given by Van Thiele [16] includes in addition to chemical compounds also viruses and microorganisms.

Until recently, the kind and amount of pesticides applied have been practically uncontrolled. Initial considerable successes in fighting insects and weeds damaging crops and drastically reducing crop yields have resulted in an exponential increase in pesticide use and the development of new, increasingly active preparations. In 1948 P.H. Miller was awarded the Nobel prize in physiology and medicine for the recognition of properties of DDT.

The present global annual production of pesticides is estimated at several hundred thousand tons. The majority of pesticides are sown directly to soil or sprayed over crop fields, plantations or forests, and hence released directly to the environment. Pesticides enter water either directly in applications such as mosquito and water plant control, or indirectly from drainage of agricultural lands, permeation through soil, in wastewater from pesticide production, municipal waste (fungicides and bactericides), wastewater from washing devices spraying pesticides or wastewater from plants using pesticides (e.g., textile plants), etc.. Recently, some pesticides (particularly nonbiodegradable) have been abandoned and pesticide uses have been curtailed. Other pesticides, such as DDT, have been banned in most countries.

In addition to pesticides, large amounts of other toxic pollutants, including polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs),

crude oil derivatives, phenols, etc., enter the environment. PCBs have been used as coolant-insulation fluids in transformers and capacitors, hydraulic fluid components in vacuum pumps and gas turbines, plasticizers and as additives to some epoxy paints [5,7]. PCBs are mixtures of congeners with a variable chlorine content (40–60%).

3. Occurrence of pesticides in surface waters

As a consequence of large production and high stability of compounds being discussed in this review, they are present in all kinds of water. In nature, pesticides are transferred among different parts of the environment undergoing a number of transformations, such as hydrolysis, biodegradation, oxidation, photolysis, biotransformation, metabolic reactions in living organisms, etc. [9,17].

In the case of surface water and groundwater, the presence of pesticides is a very serious hazard [1,5–10,18–224]. The presence of PCBs, which are commonly found in water, is a similar problem [2, 6, 14, 82, 94, 99–101, 103, 108, 109, 114, 156, 157, 173, 176–179, 190, 211, 225]. The amount of pesticides in water depends largely on the intensity of food production in a given region which in turn determines the amount of pesticides applied, on the kind of crops, season, precipitation, and water flow rate. Atmospheric precipitation is an important route of transport of pesticides, resulting in contamination of water reservoirs remote from agricultural areas, e.g., mountain lakes [87] or even polar regions [94–96]. Consequently, a number of papers have been published confirming the presence of pesticides and PCBs not only in surface waters, but also in seawater [14, 107–109, 114, 136, 138, 143, 147, 149, 150, 226–228], potable water, particularly in water from wells existing in agricultural areas [48–80, 82–86, 192, 197, 229–233], rainwater [87–92, 186, 191, 208, 234–237], as well as in water and ice from polar regions [94–96, 190, 238, 239]. Substantial amounts of pesticides and PCBs have also been found in bottom sediments [97–109, 178, 184, 190, 206, 222, 223, 238–244], mammals, eggs, fish and mollusks [88, 106–127, 179, 206, 216, 224, 240, 241, 244–262], or even in human tissue and milk [15, 121, 122, 124, 128].

The major paths of entry of organic compounds

into human and animal organisms are through the gastrointestinal tract, the respiratory system, and the skin, particularly sweaty. Once taken in, pesticides slowly undergo metabolic transformations and are excreted with urine, feces and milk both in animals and humans. Pesticides are readily accumulated in organisms in lipid tissue, liver, kidneys, brain and heart [5–8, 17, 263, 264]. For humans, the major route of exposure to these pollutants is the gastrointestinal system. In addition to regular use of contaminated water, humans eat large amounts of food in which harmful compounds have been enriched, e.g., milk and dairy products, marine and freshwater fish, crustaceans, poultry and mammal meat and vegetables [5–9, 216, 247, 258, 263–266]. The food chain in water is an example of such accumulation. If we assign an arbitrary concentration of 1 to traces of an insecticide in water, then the concentration in plankton will be 10, in the crustaceans it will be 500, in small fish the concentration will be 2500, in carnivorous fish it will be 5000 and in fish-eating birds it will be 125 000 [6]. The investigation of DDT content in different parts of the water environment of Lake Michigan confirms such an accumulation. The DDT concentrations in wet bottom silt, in crustaceans, in various fish (eelpout, ide) and in fish-eating sea gulls are 0.014 g/l, 0.41 g/kg, 3 to 6 g/kg and over 2400 g/kg, respectively [6].

The data shown in many papers [15, 121, 122, 124, 128, 267] clearly demonstrate the correlation between the concentration of pesticides in lipid tissue and mother's milk and the intensity of application of particular pesticides in a given region. This effect is apparent in the case of DDT. High DDT contents in lipid tissue of people in Costa Rica and Zaire as well as in mother's milk in Hong Kong result from continuous application of this pesticide in these countries. In Europe, where DDT has been practically eliminated from use since 1970, a steady decline of the DDT concentration in all parts of the environment (including human organisms) is observed. Nevertheless, the DDT concentration in human milk frequently greatly exceeds the maximum allowed DDT concentration for commercial dairy products and in a 'different package' such milk would not be approved for consumption. In Germany, according to estimates from 1981, a breast-fed infant takes in twice more DDT, 8 times more HCB and 13 times

more PCBs than the acceptable daily intake (ADI) [6]. In milk of some mothers these standards can be exceeded by a factor of 80, 90 and 60 for DDT, HCB and PCBs, respectively [6].

It is practically impossible to avoid the exposure to toxic organic compounds even though it has been known for a long time that the majority of pesticides as well as many other organic compounds constitute a hazard to human life due to their carcinogenic, mutagenic and teratogenic nature. This fact, along with some pesticide properties such as their persistence and bioaccumulation, demonstrates that pesticides are one of the most toxic groups of chemicals to which humans are exposed. All pesticides are toxic to some degree, e.g., organochlorine pesticides are predominantly carcinogenic, and cause skin abnormalities, cardiovascular disease, atherosclerosis, hypertension and diabetes; acute pesticide poisoning can result in paralysis of the central nervous system and death by paralysis of the respiratory system and pulmonary edema; subacute pesticide poisoning results in ataxia, hearing loss and muscular atrophy; chronic pesticide poisoning can cause headache, neurological disorders and fatigue. Lindane causes hypoplastic anemia and damage to bone marrow which decreases the production of erythrocytes. DDT and DDE are embryotoxic and teratogenic; they are also very toxic to aquatic organisms, increasing fish mortality and hindering their reproductive ability and growth. DDT causes a decrease in shell thickness of bird eggs and, consequently, in population of birds; it also accumulates in the bodies of marine mammals lowering their reproduction [4]. Triazine herbicides (simazine, atrazine) are very toxic to phytoplankton resulting in lowered photosynthesis which in turn causes a decrease in the population of fish and crustaceans eating the phytoplankton.

PCBs were involved in an accident in Japan wherein PCB-contaminated edible oil was distributed in 1968. Over 1000 persons suffered from nausea, headache and diarrhea (Yusho disease). Among 13 babies born by women suffering from the disease two were stillborn and 11 suffered from growth retardation. This demonstrates strong embryotoxic and teratogenic character of PCBs [5].

Environmental hazards of pesticides to humans as

well as to the flora and fauna force us to control closely the content of these pollutants in different parts of the environment, and particularly in water, soil and food.

4. Methods of isolation and determination of pesticides in water

4.1. General problems of the determination of pesticides in water

In the first period of application of pesticides analytical methods of their determination were limited to the analysis of final products or to the monitoring of technological processes. Practically, until 1946 only the residue of arsenic compounds and several inorganic insecticides impenetrable to plant tissue have been determined. Wide introduction of synthetic organic pesticides posed to chemists many new analytical tasks associated with the determination of these pesticides and the products of their decomposition which are frequently even more toxic than the original compounds. However, the analysis of residue requires methods capable of determining the analytes at the ppm, ppb, ppt or even lower levels.

In the case of determination of pesticides in water, as is the case with many other environmental pollutants, a sample pretreatment step includes the analyte enrichment as well as the removal (complete or partial) of matrix components. Classification of methods of isolation and preconcentration of organic compounds from water was presented in a previous paper [268]. The most common methods of preconcentration of pesticides from environmental samples are [15,267–295]:

1. liquid–liquid extraction (LLE);
2. solid-phase extraction (SPE);

Presently, three novel methods of isolation and enrichment of the analytes are being vigorously developed:

1. supercritical fluid extraction (SFE) of samples preconcentrated on solid sorbents [296–310];

2. solid-phase microextraction (SPME) [268,311–316] and
3. extraction using hollow fiber membranes [215,268]

A bioimmunoassay test is another new technique used for the determination of pesticides [181,183,191,215,231,317–319].

Headspace GC techniques are not used in the analysis of pesticides, since these compounds are usually nonvolatile. Direct aqueous injection with GC–nitrogen–phosphorus detection (NPD) method have been applied for determination of triazine pesticides [320].

4.2. Application of liquid–liquid extraction methods for the isolation of pesticides from the aqueous phase

Liquid–liquid extraction has found very wide applicability for the isolation from the aqueous phase of semivolatile and nonvolatile compounds. Examples of such applications include the determination of triazine, phenylurea and phenylcarbamate herbicides following their extraction with dichloromethane [24, 29–32, 42, 46–58, 67, 73, 97, 129, 131, 202–204, 217, 221–224, 226, 233, 243, 249, 255, 321–325], trichloromethane [71,326] or a mixture of benzene and ethyl acetate [326,327], or the determination of halogenated pesticides and PCBs following their extraction with hexane [57,60,94,107,138,141,144, 146, 149, 150, 152, 163, 170, 189, 219, 328, 329], dichloromethane [30, 80, 149, 154, 160, 163, 188, 204, 235,330–332], freon TF [145], acetonitrile [333], diethyl ether [62,75], naphtha ether [173,328], ethyl acetate [130,334], pentane [330,335], tetrachloromethane [336], trichloromethane [149,337] or a mixture of solvents [209,328].

The extract obtained during a liquid–liquid extraction usually has to undergo a further clean-up. Typically, water is removed by an addition of anhydrous sodium sulfate. Solvent evaporation is performed in a rotary evaporator, a Kuderna–Danish evaporator or in a needle evaporator in a stream of inert gas.

To clean up the extract and isolate the analyte

fraction, columns with the following packings are used:

1. silica gel is most widely employed for the extract clean-up and the removal of strongly basic compounds;
2. alumina. Most often used, neutral alumina (pH 6.9–7.1) is employed to isolate hydrocarbons, esters, aldehydes, ketones, lactones, quinones, alcohols and weak organic acids and bases. Basic alumina (pH 10–10.5) is used to isolate compounds unstable in an acidic medium, and acidic alumina (pH 3.5–4.5) is utilized to isolate organic acids and inorganic compounds;
3. Florisil (magnesium silicate) is widely used to clean up natural samples to determine trace amounts of pesticides, etc. [222,223,237,241,327].

Following sample clean-up and chromatographic separation, nitrogen- and phosphorus-containing organic compounds are usually determined using NPD, and organohalogen compounds are determined using the electron-capture detection (ECD). GC–MS is also used extensively for the identification and determination of the analytes. HPLC with UV, diode-array (DAD), MS and fluorimetric detection has recently found a wide applications in the determination of a broad spectrum of compounds.

The cleaned extracts have also been used to determine total parameters, for example EOX, following their combustion and coulometric determination of the resulting halide ions [338–340].

The liquid–liquid extraction methods, although simple and not requiring sophisticated apparatus, have a number of drawbacks. They require the use of large volumes of expensive, and possibly toxic, solvents, which are then evaporated thus creating the problem of storage of hazardous waste. Working with these solvents may require the use of personal protection devices, such as respirators.

The solvents used for extraction should be of very high purity, free from traces of the analytes and other potential interferents. Otherwise, a large solvent background is obtained, frequently precluding the determination. The formation of emulsions is yet another problem with liquid–liquid extraction. Considering all these disadvantages, solid phase extrac-

tion seems to be better suited for the isolation of organic compounds from water.

4.3. Methods of isolation of organic compounds from the aqueous phase based on sorption on solid sorbents (solid-phase extraction)

Sorption on solid sorbents is presently the most common technique of isolation of pesticides from water [76,268–274,289–294,341–346]. Advantages of this isolation technique have been described in detail in a previous paper [268]. In analytical practice, a variety of adsorbents have been used for the isolation of organic compounds from water, and new, improved sorbents are being developed, better suited for specific sample matrices, methods of final determination or selective isolation of a particular group of compounds. The oldest known adsorbent, that is activated charcoal, has found a wide applicability for the determination of total parameters of an AOX type. Such an application of activated charcoal was described in detail in our recent paper [268]. However, this sorbent has not been widely used for the determination of individual compounds due to such drawbacks as incompleteness of sorption and desorption, the possibility of reactions of the sorbent with the analytes and a relatively high background. Despite these problems, graphitized carbon blacks (GCB) with the highest surface area has been successfully used for the preconcentration of organochlorine pesticides, triazine and phenoxyacid herbicides from water samples [76,153,292,347]. Selective determinations of triazine herbicides have been achieved by a two-trap tandem system with GCB and strong cation exchanger [347].

In the following text, we will discuss the sorbents most commonly applied to the isolation of pesticides from the aqueous phase.

4.3.1. XAD porous polymeric sorbents

Amberlite XAD macroporous resins have found a wide application for the isolation of organic compounds from water [269,270,344]. Pretreatment of the commercial sorbent involves sieving to obtain the desired particle size fraction and decantation in methanol to remove very fine particles. Further pretreatment of the sorbent is carried out in a Soxhlet extractor where it is successively extracted for 8 h

with methanol, acetonitrile and diethyl ether, followed by thermal desorption for 8 h in a stream of inert gas. Thus prepared sorbent is then stored in a glass container under methanol [269].

XAD-2 and XAD-4 (styrene–divinylbenzene copolymers) have been used most frequently because of their hydrophobicity and lack of adsorption of inorganic ions. XAD-2 has been utilized for preconcentration of volatile and nonvolatile organohalogen compounds [88,98,165,241,328,348–352], herbicides [69,165,348], PCBs [88,98,226,227,241,351], phenols [349,353], chlorobenzenes [98,354], MX [355,356], polycyclic aromatic hydrocarbons (PAHs) [98,348,352,353,357,358], organosulfur compounds [359] and other groups of compounds [98,350,353,360,361].

Solvent extraction is the most common method of desorption of the trapped analytes from the sorbent bed. The solvents used for desorption included diethyl ether [88,350,351,354,360,362–364], ethyl acetate [356,361], methanol [352,359,365], acetone [165,366], acetonitrile [226,227,367], cyclohexane [336,352], *n*-hexane [364], dichloromethane [98,328,353], ethanol [337] or a mixture of two solvents [98,344,355,357,364].

XAD-4 has been used for preconcentration of pesticides [368–370], and other compounds [349,371]. The solvents used for recovery of the trapped analytes included pentane [372], dichloromethane [349,370], diethyl ether [278,350], ethanol [364], acetone [357,369] and hexane [370].

Other XAD resins have found much more limited application [269,271,272]. XAD-7 and XAD-8 (methyl methacrylate polymers) were utilized for the isolation and enrichment of pesticides [88,349,350], PCBs [88,373] and humic acids [373,374].

Sorbents analogous to XAD resins (called temporarily 542 and 655) have been developed in Poland [349]. They are also styrene–divinylbenzene copolymers and they reveal sorption properties similar to XAD-2 and XAD-4 as measured by the total recovery of analytes from the aqueous phase following their isolation on the sorbent and desorption with dichloromethane in a Soxhlet extractor for 12 h. Investigations were carried out for ten model compounds, including *n*-hexadecane, *p*-dichlorobenzene, DDT and phenol.

Solvent extraction of a sorbent bed is usually carried out by passing a solvent through a column

packed with the sorbent, but it can also be performed in a Soxhlet extractor [227,336,349,357]. The extracts are often pretreated before the final chromatographic analysis by removing water using anhydrous sodium sulfate [226,227,327,349,361,369] or freezing it out [354,360], by evaporating the solvent [227,349,360,361,363], by replacing the solvent with another solvent [375,376], or by fractionating the analytes in the extract using columns packed with Sephadex [344], Zorbax–CN [363] and Spherisorb–CN [363].

Final determinations of the analytes are typically performed by gas chromatography with flame-ionization or mass spectrometric detection [69,98,354,355,362], but selective detectors, such as ECD [88,238,333,344,368] and electrolytic conductivity detector [333,361] for organohalogen compounds, NPD [333,344,361] for organonitrogen and organophosphorus compounds atomic emission detection (AED) [333,377] and flame photometric detection (FPD) [369] for organosulfur compounds are also employed. HPLC with the UV and DAD detection has recently found large applications in this area [290,358,363,367].

An example of application of XAD-2 resin to the determination of selected nitrogen- and phosphorus-containing pesticides is described below.

Determination of selected organonitrogen and organophosphorus pesticides after isolation on XAD-2 and extraction with dichloromethane [15,378–380]

A styrene–divinylbenzene copolymer, XAD-2, has been used for the isolation and preconcentration of triazine herbicides as well as other organonitrogen and organophosphorus pesticides [15]. Triazine herbicides, especially simazine and atrazine, represent one of the major groups of anthropogenic water pollutants due to their high solubility in water, relatively long half-lives and wide application [11–15, 28, 37, 47–51, 76, 87, 180–188, 232, 236, 237, 324]. Because the maximum allowable concentrations of pesticides in drinking water are very low (for example, in the EEC they are 0.1 $\mu\text{g}/\text{l}$) [37], there is a need for selective and sensitive methods of their determination. As a result of its simplicity and speed, minimal adsorption of water and the lack of solvent background, solid-phase extraction (SPE) [33–35, 37, 47–49, 60, 76, 87, 136, 269–272, 367] seems

to be preferable for this purpose over liquid–liquid extraction [29–32]. The choice of sorbent depends on the kind of analytes and is usually a compromise between the sorption and desorption conditions. The choice of XAD-2 for the isolation of herbicides resulted from its sorption characteristics, the ease of desorption and common use for the preconcentration of pesticides. The trapped pesticides are solvent desorbed with dichloromethane and determined by gas chromatography with thermionic detection. Two procedures for the determination of selected pesticides in water have been developed, one using small columns packed with XAD-2, the other one using large columns packed with the same sorbent. The procedure using small amounts of XAD-2 is suitable for the determination of pesticides in relatively polluted water (rivers), whereas the procedure employing larger amounts of XAD-2 is best suited for the determination of pesticides in relatively clean water (seawater, estuary water).

After preliminary investigations, the following analytical procedure was selected for small columns containing 1.8 g of XAD-2:

1. natural samples were filtered through Whatman filters using a Millipore filtration kit;
2. known amounts (about 500 ng per sample) of Cyprazine or Prometryn were added to each sample as an internal standard;
3. 2 l of the analyzed solution were passed through the XAD-2 column at 20 ml/min;
4. after preconcentration of pesticides the entire apparatus including the sorbent bed were washed with 100 ml of doubly distilled water;
5. the sorbent bed was then dried for about 15 min using an aspirator;
6. the trapped analyte was extracted by backflushing the bed with 20 ml of dichloromethane at 1.5 ml/min;
7. residual water was removed from the extract by adding ca. 8 g of anhydrous sodium sulfate;
8. the extract was transferred to a test tube using a Pasteur pipet;
9. dichloromethane was gently evaporated using a needle evaporator and replaced by isooctane;
10. for natural samples, solvent exchange was followed by filtration using a syringe filtering device with 12-mm Whatman filters;

11. the final extract was evaporated to ca. 0.4 ml;
12. the extract was then stored in a freezer;
13. following extraction, the sorbent bed was additionally cleaned by passing 50 ml of methanol and 100 ml of doubly distilled water;
14. final determinations were carried out by gas chromatography with the Varian NPD detector.

For large columns containing 53 g of XAD-2, the following analytical procedure was used:

1. filtration and internal standard addition as for 'small columns';
2. preconcentration of the analytes from 10–20 l of samples passed at 100 ml/min;
3. following preconcentration, the system was washed with 500 ml of doubly distilled water;
4. the sorbent bed was then dried for about 30 min using an aspirator;
5. the trapped analytes were extracted by backflushing the bed with 100 ml of dichloromethane at 2 ml/min. Initially, residual water is removed from the column. The water can be collected using a Pasteur pipet which facilitates drying of the extract;
6. the extract was dried using ca. 20 g of anhydrous sodium sulfate;
7. further procedure was the same as for 'small columns'.

The detection limits for the analyzed pesticides were very low (10 ng/l for small columns to 1–2 ng/l for large columns). The precision of both procedures was good, as demonstrated by the relative standard deviation which ranged from 2.9 to 9.8%. The accuracy was also good: the average recovery was 91–106% for small columns and 82–101% for large columns. Model solutions of 5 common pesticides (simazine, atrazine, ethyl parathion, malathion, and methyl parathion) along with 2 degradation products of atrazine [desisopropylatrazine (DIA) and desethylatrazine (DEA)] were investigated. The concentrations of the pesticides in the model solutions varied from 50 to 250 ng/l for small columns and from 10 to 50 ng/l for large columns. The procedures developed were successfully applied to the determination of these pesticides in surface water, potable water and seawater. The results of these

determinations are listed in Table 1. Simazine, atrazine, DEA and DIA were found in all of the examined samples. Their respective concentrations in seawater, potable water and river water were 7.6, ca. 16 and ca. 110 ng/l for simazine; 16, 40 and 140 ng/l for atrazine; 5, 45 and 60 ng/l for DEA and 10, 40 and 400 ng/l for DIA. The identity of the analytes (except for DIA) was confirmed by GC-MS. High recovery of an internal standard (cyprazine) added to filtered samples (86 to 105%) should also be emphasized.

4.3.2. Chemically modified silica gels

Chemically modified silica gels are relatively new sorbents used for the isolation of organic compounds from the aqueous phase. Such sorbents are prepared by chemically bonding organic groups to the surface of silica gel. Depending on the organic group bonded to the surface, the modified sorbent can then be used for the isolation of selected classes of organic analytes which is an obvious advantage of this approach. Most often, commercially available pre-packed sorption tubes are utilized.

The use of chemically modified silica gels allows the separation of organic compounds of interest from all other compounds which are not to be determined. This can be achieved in three ways:

1. by selective extraction: only the compounds to be determined are trapped on the sorbent bed during preconcentration, other compounds are not retained;
2. by selective desorption of impurities: during solvent extraction of the sorbent bed only impurities are removed, the analytes are retained on the bed and are subsequently eluted with a different solvent;
3. by selective desorption of analytes: only the analytes are solvent extracted from the sorbent bed, the impurities remain retained on the sorbent.

The selection of solvents for each step of the procedure depends primarily on the kind of analytes and potential interferents. The procedures using sorption tubes packed with modified silica gels usually involve the following steps:

Table 1
Results of determination of selected pesticides in natural samples after sorption on XAD-2 and extraction with dichloromethane

Sample	Date	V (l)	Compound									
			DIA (ng/l)	DEA (ng/l)	Simazine (ng/l)	Atrazine (ng/l)	Methyl parathion (ng/l)	Malathion (ng/l)	Ethyl parathion (ng/l)	Cyprazine R (%)		
<i>Large absorbers</i> (53 g XAD-2); $v_s = 100$ ml/min, $V_e = 100$ ml, $v_i = 2$ ml/min												
Absorber blank	92.09.29	0	0	0	0	0	0	0	0	0	0	—
Bidistilled water (IFREMER, Nantes, France)	92.09.30	10	0	0	0	0	0	0	0	0	0	—
Tap water (IFREMER)	92.09.24	20	35.9	44.4	17.1	44.3	0	0	0	0	0	102.7
Loire by Montjean, France	92.10.05	4.44	162.9	70.8	79.9	128.5	0	0	0	0	0	96.1
Tap water (IFREMER)	92.10.05	5	46.6	47.6	16.1	38.5	0	0	0	0	0	98.4
Sea water (St. Nazaire Deep)	92.10.06	20	10.6	5.2	7.6	15.8	0	0	0	0	0	101.0
Loire (Pellerin), France (1 m)	92.10.07	4.85	127.4	66.5	79.9	125.8	0	0	0	0	0	87.3
<i>Small absorbers</i> (1.8 g XAD-2); $v_s = 20$ ml/min; $V_e = 20$ ml; $v_i = 1.5$ ml/min												
Rhone (Arles), France	92.0.257	2	340	87	41	151	0	0	0	0	0	0
Tap water, IFREMER, Nantes, France	92.09.14	2	0	40.5	24	0	0	0	0	0	0	0
Loire (Pellerin), France (1 m)	98.2	2	98.2	48.8	93.5	0	0	0	0	0	0	0
Seine (La Bouille), France	92.11.20	2	0	241.4	54.2	0	0	0	11.3	0	0	0

This work was made in IFREMER, Nantes (France) and was supported by IFREMER and the Ministry of Research and Technology (France).

1. the selection of kind and amount of a sorbent depending on the concentration level of the analytes and interferences, the matrix composition, and the sample volume available. Most commonly, sorption tubes packed with 1 ml of the sorbent are used but the tubes containing 3 or 6 ml of the sorbent can also be utilized;
2. conditioning of sorption tubes in order to activate the sorbent bed. The method of conditioning depends on the kind of sorbent used. After this step, the sorbent bed should not be allowed to dry;
3. preconcentration of the analyte(s) on the sorbent bed. The sample volume may vary from milliliters to liters. In order to improve the selectivity of enrichment of the analytes, the pH of the sample can be adjusted, inorganic salts or organic solvents can be added to the sample, and the analytes can be converted into derivatives that are better adsorbed on the sorbent or are easier to determine. An internal standard is usually added to the sample prior to the preconcentration step;
4. washing the sorbent bed to remove impurities weakly retained on the bed;
5. extraction of the trapped analytes with a suitable solvent (0.2 to 2 ml);
6. regeneration of the sorbent bed aimed at a removal of the remaining impurities.

The obtained extract can be treated further by fractionation, solvent evaporation or solvent exchange depending on the method of final determination. Final determinations are carried out by gas chromatography, high-performance liquid chromatography (HPLC) or supercritical fluid chromatography.

The most commonly used sorbents from this group are silica gels with bonded octadecylsilyl (ODS) or octylsilyl groups. Examples of application of these sorbents include:

1. determination of organophosphorus and organonitrogen pesticides using GC-NPD [34,35, 49,163,182,208,220,287,294,325,381–386];
2. determination of organochlorine pesticides and PCBs using GC-ECD [136,142,163,210,287, 383,384,387–392];
3. determination of a variety of pesticides and other

compounds using GC-MS [26,33,37,38,44,47, 55, 79–81, 87, 174, 208, 230, 245, 295, 325, 385, 388–390,393–399];

4. determination of a broad spectrum of compounds using on-line or off-line solid-phase extraction technique and HPLC with UV, DAD, MS or fluorimetric detection [40,48,51,66,76,184,230, 287,290,291,295,345,367,386,398,400–418].

Another approach to using chemically modified silica gel is the extraction disk technique in which sorption tubes are replaced by disks with diameters of 25, 47 or 90 mm and 0.5 mm thick made of PTFE and sorbent particles. The disks with the ODS phase are used most often, but the disks with the octylsilyl phase or styrene-divinylbenzene copolymer are also common [289,415]. A sorbent makes about 90% of the mass of a disk and in the case of disks having 47 mm in diameter the sorbent mass is about 500 mg. The diameter of sorbent particles is ca. 8 μm , and the pore size is about 0.6 μm .

Fig. 1 shows an assembly for extraction disks manufactured by J.T. Baker company [415]. The analytical procedure involving extraction disks is

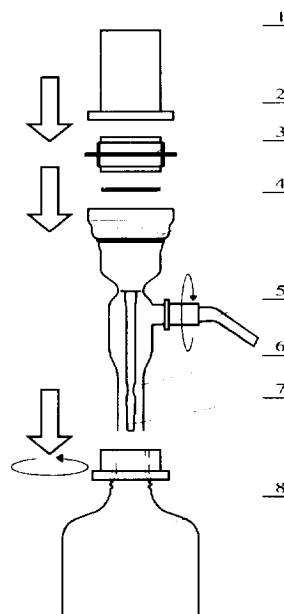


Fig. 1. Vacuum assembly for extraction disks: (1) graduated funnel for the analyzed sample; (2) PTFE connectors; (3) Empore SPE disk; (4) disk base with glass frits; (5) outlet to a vacuum pump; (6) glass connector; (7) Luer tip and (8) suction flask.

completely analogous to that utilizing sorption tubes and includes the same steps: assembly of the apparatus, sorbent conditioning with a suitable organic solvent, passing a sample (up to 2.5 l) at about 3 ml/min using an aspirator, drying the disk, collecting the extract, followed by further treatment of the extract in the same manner as for sorption tubes.

A number of procedures using extraction disks have been developed, including those for the determination of dioxins, PAHs, PCBs, phthalates, pesticides, phenols, and explosives in water and wastewater [41,161,245,288,323,415,416,419–426]. The advantages of using extraction disks include an increased efficiency of sorption/desorption compared to sorption tubes due to: (1) a decrease in particle diameter of sorbents, (2) improved packing of the sorbent particles which reduces the mean free path of the analyte molecules to the sorbent particle, and (3) a decrease in linear velocity of the analyzed sample with the simultaneous reduction in its flow-rate as a result of considerable increase in the flow cross-section. The extraction disk technique is relatively recent; it was first applied in 1990. However, its advantages, and mainly the simplicity and low cost, have caused a growing interest among the analysts and increased applications, despite high blank values and batch-to-batch variations of the sorbents.

4.3.3. Tenax sorbents

So far, Tenax sorbents have been used mostly for the isolation of organic compounds from gaseous samples. In the case of analysis of water pollutants, Tenax has been employed primarily in the purge and trap and CLSA. The use of Tenax for direct pre-concentration of organic compounds from the aqueous phase was much less common [427–429]. This seems justified if the trapped analytes are to be solvent extracted [83], but thermal desorption from Tenax appears to be advantageous [427–429]. Tenax sorbents have high thermal stability (up to 400°C) which is superior to that of XAD resins. Consequently, thermal desorption from Tenax can be carried out at a high temperature (240–250°C) and the liberated analytes are directed onto a GC column operated at ambient temperature [30°C cold injection system (CIS)] [427] or below ambient temperature [–30°C whole column cryotrapping (WCC)] [423]. Even though small volumes of water samples (about

50 ml) are used, the detection limits for individual compounds are still in the order of ng/l, because the entire amount of the analyte is subjected to GC analysis. Procedures using direct sorption on Tenax of analytes from water samples have been applied to the determination of pesticides and PAHs [427], methyl esters of carboxylic acids [428], hydrocarbons and volatile organohalogen compounds [429].

4.4. Supercritical fluid extraction

Supercritical fluid extraction (SFE) is a new technique of sample pretreatment which has a potentially wide applicability, particularly for complex environmental samples due to its speed, selectivity and ease of use [296–310]. Unique advantages of supercritical fluids are related to their properties. Some parameters of supercritical fluids are similar to those of gases whereas others resemble those of liquids.

Although a number of compounds have been proposed as SFE solvents, only carbon dioxide has been widely used because of its low critical temperature (31.1°C), relatively low critical pressure (73.8 bar), low reactivity and because it is readily available in high-purity form and is nontoxic, inexpensive, and nonflammable. Even though carbon dioxide is classified as a nonpolar solvent, as a result of its large quadrupole moment it exhibits some affinity also toward polar organic compounds. The elution strength of carbon dioxide increases with an increase in its temperature and pressure. At a relatively low pressure (75 bar; 80°C; $d=0.15$ g/ml), its elution strength is similar to that of pentane. At higher pressures (380 bar; 40°C; $d=0.95$ g/ml) the elution strength of CO₂ resembles that of dichloromethane, toluene or benzene [296,300,303].

Nitrous oxide, ethane, butane, sulfur hexafluoride, and chlorodifluoromethane have also been used as supercritical fluids; however, due to their toxicity, flammability or environmental impact their application has been limited [296,300,303]. The main application of supercritical fluid extraction is the extraction of analytes from solid samples.

In order to improve the extraction efficiency for a number of polar compounds, several modifiers can be added to carbon dioxide; methanol, ethanol, 1-propanol, 2-propanol, tetrahydrofuran, 1,4-dioxane,

acetonitrile, water and carbon disulfide are used most often for this purpose.

The main advantages of supercritical fluid extraction are:

1. elimination of expensive high-purity solvents, some of which are toxic (e.g., dichloromethane, tetrachloroethylene) thus posing laboratory hazard and the problem of disposal of waste;
2. substantial reduction of time of the extraction due to the rapid mass transfer between the two phases;
3. ease of on-line or off-line coupling to other instruments used for the final determination of analytes (HPLC, GC, supercritical fluid chromatography, spectrophotometry);
4. selectivity of extraction of a group of analytes due to ease of modification of properties of the extracting agent (density, viscosity, elution strength) which can be effectively adjusted by varying the pressure and temperature and by the addition of modifiers;
5. supercritical fluid extracts are usually solvent free and no evaporation or concentration steps are necessary prior to final determination of the analytes;
6. the extracts are cleaner than those obtained using solvent extraction, because CO₂ is available in a high-purity grade that is also inexpensive and readily available;

SFE can be performed under mild conditions which minimizes thermal degradation.

4.4.1. Solid-phase extraction–supercritical fluid extraction

A combination of SPE and SFE techniques is the most common procedure for isolation and preconcentration of analytes from water samples using supercritical fluids. In the first step, organic compounds are extracted from the aqueous phase to a solid phase. Tenax, XAD resins, modified silica gels and polyurethane foam are typically used as sorbents [423,425–429]. Next, the sorbent bed is dried (usually in a stream of nitrogen at 60°C) and transferred to an extraction cell where a selective extraction of particular analytes by a supercritical fluid takes place.

4.4.2. Direct extraction of organic compounds from water samples using SFE

Direct extraction of organic compounds from water samples suffers a number of drawbacks, the most severe of which is a relatively high solubility of water in supercritical CO₂ (ca. 0.3%). The problem of presence of water, which can block the restrictor or enter the chromatographic system, is partly solved by using dynamic supercritical fluid extraction of solid sorbents with the trapped analytes; however, the present state of the art of SFE is still not acceptable for routine analyses.

The first system for supercritical fluid extraction of organic compounds from water was based on the closed-loop stripping analysis (CLSA). In this system, a supercritical fluid circulates from the outlet of an extraction cell back to the water sample. When the system sample-supercritical fluid reaches an equilibrium, a portion of the supercritical fluid is directed to a sampling valve. The sample is then analyzed by supercritical fluid chromatography. Such a system was investigated for the determination of diisopropyl methylphosphonate in water. The time of equilibration was 1.5 h, but after an addition of 0.1 mg of NaCl to the water sample it was reduced to below 5 min. The same method has also been used for the isolation and determination of phenol in water [430].

Another system [431] is based on a sandwich phase separator in which supercritical CO₂ and the aqueous phase are separated by means of hydrophobic membranes resistant to high pressure made from PVDF [(-CH₂-CF₂-)_n] and Delrin [(-CH₂-O-)_n]. This system was used for the isolation of phenol and 4-chlorophenol from water.

In summary, it should be emphasized that supercritical fluid extraction is a vigorously developed technique for the isolation and enrichment of analytes. Its importance in the analysis of environmental sample cannot be overstated.

5. Conclusions

Pesticides constitute one of the most hazardous groups of environmental pollutants. They are toxic to humans as well as to the flora and fauna. As a result of their wide application and persistence, they can be found in all parts of the environment, including

muscle and lipid tissue of fish, mammals and humans. The analysis of pesticides is a formidable task considering very low concentrations of the analytes, their diversity and different physical and chemical properties, and much higher concentrations of interfering compounds. Nevertheless, because of the environmental impact of pesticides, a lot of effort has been devoted to the development of methods of determination of these analytes. The methods of determination of pesticides are now well developed, as evidenced by their inclusion into standard procedures used, for example, by the United States Environmental Protection Agency (e.g. Methods 505, 507, 515, 531, 549.1, 553, 608, 619, 632, and 8325) or OSHA (Method 62). Sample preparation techniques in these methods frequently involve solid-phase extraction using extraction tubes or extraction disks, or solid-phase microextraction employing PDMS- or polyacrylate-coated fibers. Final determination is typically carried out by gas chromatography with FID, NPD, FPD, ECD or MS detection or by high-performance liquid chromatography with UV detection.

The present paper discusses only the most common techniques used for the isolation and determination of pesticides in water, namely solid phase extraction utilizing XAD resins, Tenax and modified silica gels, liquid–liquid extraction and supercritical fluid extraction. Solid phase microextraction and membrane techniques have been discussed in detail in a previous paper dealing with volatile organic compounds [268] and are not included in this review.

Acknowledgments

This work was partially supported by grant No. 2 P303 091 07 from Committee on Scientific Research (Poland).

References

- [1] A. Bruchet, M.F. Legrand, C. Anselme and J. Mallevalle, *Water Supply*, 7 (1989) 77.
- [2] J. Dojlido, *Chemia wody*, Arkady, Warszawa, 1987.
- [3] W.E. Coleman, R.G. Melton, F.C. Kopfler, K.A. Barone, T.A. Aurand and M.G. Jellison, *Environ. Sci. Technol.*, 14 (1980) 576.
- [4] H.A.M. de Kruijf and J.H. Kool (Editors), *Organic Micropollutants in Drinking Water and Health*, Elsevier, Amsterdam, 1985.
- [5] J.W. Moore and S. Ramamoorthy, *Organic Chemicals in Natural Waters. Applied Monitoring and Impact Assessment*, Springer Verlag, New York, 1984.
- [6] Z. Przeździecki, *Biologiczne skutki chemizacji środowiska*, PWN, Warszawa, 1984.
- [7] W. Eichler, *Trucizny w naszym pożywieniu*, Państw. Zakł. Wydawnictw Lekarskich, Warszawa, 1989.
- [8] M. Nikonorow (Editor), *Pestycydy w świetle toksykologii środowiska*, Państw. Wydaw. Rolnicze i Leśne, Warszawa, 1979.
- [9] L. Różański, *Przemiany pestycydów w organizmach żywych i środowisku*, Państw. Wydaw. Rolnicze i Leśne, Warszawa, 1992.
- [10] N.L. Nemerow, *Stream, Lake, Estuary and Ocean Pollution*, Van Nostrand Reinhold Co., New York, 1985.
- [11] J. Fournier, *Chimie des Pesticides, Cultures et Techniques*, Nantes, 1988.
- [12] A.S.Y. Chau, B.K. Afgan and J.W. Robinson, *Analysis of Pesticides in Water, Vol. III Nitrogen-Containing Pesticides*, CRC Press, Boca Raton, FL, 1982.
- [13] P.C. Kearney and D.D. Kaufman (Editors), *Herbicides. Chemistry, Degradation and Mode of Action*, Marcel Dekker, New York, 1975.
- [14] D.V. Chapman, *Pesticides in the Aquatic Environment. A global assessment of use and effects*, Monitoring and Assessment Research Center, Report No. 39, King's College London, University of London, London, 1987.
- [15] M. Biziuk, *Zeszyty Naukowe Politechniki Gdańskiej*, No. 513, *Chemia XXXI*, Politechnika Gdańska, Gdańsk, 1994.
- [16] N. Van Thiel, *Pestic. Sci.*, 6 (1975) 189.
- [17] V. Petit, R. Cabridenc, R.P.J. Swannell and R.S. Sokhi, *Environ. Int.*, 21 (1995) 167.
- [18] H. Hellmann, *Analysis of Surface Waters*, Ellis Horwood, New York, 1987.
- [19] M.E. Comba and K.L.E. Kaiser, *J. Great Lakes Res.*, 11 (1985) 404.
- [20] B. Yaron, *Agriculture, Ecosystems and Environment*, 26 (1989) 275.
- [21] J. Beltran, F. Hernandez, I. Morell, P. Navarrete and E. Aroca, *Sci. Total Environ.*, 132 (1993) 243.
- [22] C. Lee and S.G. Wakeham, *Organic Matter in Seawater: Biogeochemical Processes in Chemical Oceanography*, Vol. 9, chapt. 49, Acad. Press, 1988, p. 1.
- [23] D.A. Belluck, S.L. Benjamin and T. Dawson, L. Somasundaram and J.R. Coats (eds.), *Groundwater Contamination by Atrazine and Its Metabolites in Pesticide Transformation Products, Fate and Significance in the Environment*, Amer. Chem. Soc., Washington, 1991, chapt. 18, p. 224.
- [24] S.K. Widmer, J.M. Olson and W.C. Koskinen, *J. Environ. Sci. Health*, B28 (1993) 19.

- [25] E.M. Thurman, D.A. Goolsby, M.T. Mayer, M.S. Mills and M.L. Pomes, *Environ. Sci. Technol.*, 26 (1992) 2440.
- [26] D.W. Kolpin and S.J. Kalkhoff, *Environ. Sci. Technol.*, 27 (1993) 134.
- [27] P. Matthiessen, C. Allchin, R.J. Williams, S.C. Bird, D. Brooke and P.J. Glendinning, *J. IWEM*, 6 (1992) 496.
- [28] W. Leuchs, E. Plöger, H. Friege, U. Vogt and P. Obermann, *Vom Wasser*, 76 (1991) 39.
- [29] W.E. Pereira, C.E. Rostad and T.J. Leiker, *Anal. Chim. Acta*, 228 (1990) 69.
- [30] V. Leoni, C. Cremisini, A. Casuccio and A. Gullotti, *Pestic. Sci.*, 31 (1991) 209.
- [31] H.-B. Lee and Y.D. Stokker, *J. Assoc. Off. Anal. Chem.*, 69 (1986) 568.
- [32] G. Durand, V. Bouvot and D. Barceló, *J. Chromatogr.*, 607 (1992) 319.
- [33] E. Davoli, E. Benfenati, R. Bagnati and R. Fanelli, *Chemosphere*, 16 (1987) 1425.
- [34] J.C. Molto, Y. Pico, G. Font and J. Manes, *J. Chromatogr.*, 555 (1991) 137.
- [35] M. Ahel, K.M. Evans, T.W. Fileman and R.F.C. Mantoura, *Anal. Chim. Acta*, 268 (1992) 195.
- [36] G.R. Hallberg, *Agric. Ecosyst. Environ.*, 26 (1989) 229.
- [37] E. Benfenati, P. Tremolada, L. Chiappetta, R. Frassanito, G. Bassi, N. Di Toro, R. Fanelli and G. Stella, *Chemosphere*, 21 (1990) 1411.
- [38] Z. Cai, V.M. Ramanujam, D.E. Giblin and M.L. Gross, *Anal. Chem.*, 65 (1993) 21.
- [39] P. Krämer and R. Schmid, *Vom Wasser*, 75 (1990) 99.
- [40] P.R. Loconto, *J. Liq. Chromatogr.*, 14 (1991) 1297.
- [41] D. Barceló, G. Durand, V. Bouvot and M. Nielen, *Environ. Sci. Technol.*, 27 (1993) 271.
- [42] J. Tronczynski, C. Munsch, G. Durand and D. Barceló, *Sci. Total Environ.*, 132 (1993) 327.
- [43] S.S.D. Foster, P.J. Chilton and M.E. Stuart, *J. IWEM*, 5 (1991) 186.
- [44] D. Duncan, D.T. Pederson, T.R. Shepherd and J.D. Carr, *Ground Water Monitor. Rev.*, 11 (1991) 144.
- [45] U. De la Vigne and D.E. Jänchen, *J. Chromatogr.*, 553 (1991) 489.
- [46] E.E. Pick, L.P. van Dyk and E. Botha, *Chemosphere*, 25 (1992) 335.
- [47] L.Q. Huang, *J. Assoc. Off. Anal. Chem.*, 72 (1989) 349.
- [48] R. Reupert and E. Plöger, *Vom Wasser*, 72 (1989) 211.
- [49] R.G. Nash, *J. Assoc. Off. Anal. Chem.*, 73 (1990) 438.
- [50] P. Levallois and D. Gauvin, *Sci. Technol. de l'eau*, Février 1991, 65.
- [51] R. Reupert, E. Plöger and G. Brausen, HPLC Determination of 29 Controlled Herbicides in Water Supplies, Application Note, Publication Number 12-5952-2229, Hewlett-Packard, 1990.
- [52] E.M. Keil, G. Kurz and M. Steinwand, *LaborPraxis*, März 1991, 146.
- [53] D. Fröhlich and W. Meier, *J. High Resolut. Chromatogr.*, 12 (1989) 340.
- [54] G. Von Petrick, D.E. Schulz-Bull and J.C. Duinker, *Z. Wasser-Abwasser-Forsch.*, 25 (1992) 115.
- [55] R. Bagnati, E. Benefanti, E. Davoli and R. Fanelli, *Chemosphere*, 17 (1988) 59.
- [56] T.M. Younos and D.L. Weigmann, *J. WPCF*, 60 (1988) 1199.
- [57] J. Gawdzik, *Chem. Anal.*, 33 (1988) 445.
- [58] H.B. Pionke, D.E. Glotfelty, A.D. Lucas and J.B. Urban, *J. Environ. Quality*, 17 (1988) 76.
- [59] R.F. Spalding, M.E. Burbach and M.E. Exner, *Ground Water Monit. Rev.*, 9 (1989) 126.
- [60] C. Ang, K. Meleady and L. Wallace, *Bull. Environ. Contam. Toxicol.*, 42 (1989) 595.
- [61] M. Leistra and J.J.T.I. Boesten, *Agric. Ecosyst. Environ.*, 26 (1989) 369.
- [62] H.B. Pionke and D.E. Glotfelty, *Water Res.*, 23 (1989) 1031.
- [63] A. Żelechowska and co-workers, Ocena zagrożenia wód powierzchniowych wykorzystywanych jako wody do picia aktualnie stosowanymi pestycydami, personal communication, IMGW, Gdańsk, 1989.
- [64] A. Żelechowska, Ustalenie podstaw do przedsięwzięć w zakresie ochrony wód przed zanieczyszczeniami pestycydami, grant CPBR 11.10.35.1.01., report, IMGW, Gdańsk, 1990.
- [65] W.F. Ritter, *J. Environ. Sci. Health, Part B.*, 25 (1990) 1.
- [66] C.H. Marvin, I.D. Brindle, C.D. Hall and M. Chiba, *J. Chromatogr.*, 503 (1990) 167.
- [67] R. Frank, H.E. Braun, B.S. Clegg, B.D. Ripley and R. Johnson, *Bull. Environ. Contam. Toxicol.*, 44 (1990) 410.
- [68] M.J. Walker and K.S. Porter, *Ground Water Monit. Rev.*, 10 (1990) 116.
- [69] M.E. Exner, *Ground Water Monit. Rev.*, 10 (1990) 147.
- [70] E. Funari, A. Bastone, P. Bottoni, S. Carbone, L. Donati, I. Marcello, I. Camoni and G. Giuliano, Il rischio della contaminazione della risorse idropotabili da erbicidi. La situazione in Italia, Istituto Superiore di Sanita, Roma, 1991.
- [71] C. Molino, M. Franchi, F. Giampaoli and G. Disperati, *Acqua Aria*, 7 (1991) 663.
- [72] T.G. Huat, G.S. Hock and K. Vijayaletchumy, *Environ. Monit. Assess.*, 19 (1991) 469.
- [73] M.F. Legrand, E. Costentin and A. Bruchet, *Environ. Toxicol.*, 12, 985 (1991).
- [74] M. Babut, F. Marchand, M. Schiavon, X. Thevenot and T. Francais, *J. Franc. d'Hydrol.*, 22 (1991) 235.
- [75] C. Schlett, *Fresenius J. Anal. Chem.*, 339 (1991) 344.
- [76] A. Di Corcia and M. Marchetti, *Anal. Chem.*, 63 (1991) 580.
- [77] F.K. Von Ohnesorge, *Gesunde Pflanzen*, 43 (1991) 26.
- [78] C. Trova, G. Cossa and G. Gandolfo, *Acqua-Aria*, 7 (1992) 649.
- [79] P. Zouzaneas, F. Sövegjarto, H. Stadlbauer and E.R. Schmid, *Ernährung/Nutrition*, 17 (1993) 77.
- [80] F. Sövegjarto, P. Zouzaneas, C. Neumüller and E.R. Schmid, *Ernährung/Nutrition*, 17 (1993) 9.
- [81] J. Pietsch, W. Schmidt, F. Sacher, S. Fichtner and H.-J. Brauch, *Fresenius J. Anal. Chem.*, 353 (1995) 75.
- [82] M. Chevreuil and L. Granier, *Chemosphere*, 23 (1991) 1637.
- [83] J. Lintemann, C. Mengel and A. Kettrup, *Fresenius J. Anal. Chem.*, 346 (1993) 752.
- [84] S. Chiron and D. Barceló, *J. Chromatogr.*, 645 (1993) 125.

- [85] H. Badach, K. Pomorska and T. Nazimek, *Ochrona Roślin*, 37 (1993) 15.
- [86] H. Miyata, O. Aozasa, S. Ohta, T. Chang and Y. Yasuda, *Chemosphere*, 26 (1993) 1527.
- [87] H.-R. Buser, *Environ. Sci. Technol.*, 24 (1990) 1049.
- [88] W.M.J. Strachan and H. Huneault, *Environ. Sci. Technol.*, 18 (1984) 127.
- [89] R. Arimoto, *J. Great Lakes Res.*, 15 (1989) 339.
- [90] F. Trautner, K. Huber and R. Niessner, *J. Aerosol Sci.*, 23 (1993) S999.
- [91] S. Li and J. Migita, *Water Sci. Tech.*, 25 (1992) 69.
- [92] J. Daub, T. Striebel, A. Robien and R. Herrmann, *Vom Wasser*, 80 (1993) 155.
- [93] R. Wittlinger and K. Ballschmiter, *Fresenius J. Anal. Chem.*, 336 (1990) 193.
- [94] P. Desideri, L. Lepri and L. Checchini, *Ann. Chim.*, 79 (1989) 589.
- [95] L. Morselli, S. Zappoli and A. Donati, *Ann. Chim.*, 79 (1989) 677.
- [96] G.W. Patton, D.A. Hinckley, M.D. Walla, T.F. Bidleman and B.T. Hargrave, *Tellus*, 41B (1989) 243.
- [97] R.F. Platford, R.J. Maguire, R.J. Tkacz, M.E. Comba and K.L.E. Kaiser, *J. Great Lakes Res.*, 11 (1985) 379.
- [98] C. Lanlois and H. Sloterdijk, *Rev. Sci. de l'Eau*, 2 (1989) 659.
- [99] F.G. Taylor, *Polychlorinated Biphenyls in the Aquatic Environment of Oak Ridge National Laboratory, Report of 1989*, Oak Ridge Nat. Lab., Oak Ridge, 1990.
- [100] M.E. Comba and K.L.E. Kaiser, *Sci. Total Environ.*, 97/98 (1990) 191.
- [101] J.E. Sanders, *Northeastern Environ. Sci.*, 8 (1989) 1.
- [102] K.-E. Quentin, *Acta Hydrochim. Hydrobiol.*, 17 (1989) 359.
- [103] K.L.E. Kaiser, B.G. Oliver, M.N. Charlton, K.D. Nicol and M.E. Comba, *Sci. Total Environ.*, 97/98 (1990) 495.
- [104] J. Falandysz, *Bromat. Chem. Toksykol.* XXV, 4 (1992) 387.
- [105] U. Järneberg and co-workers, *Environ. Sci. Technol.*, 27 (1993) 1364.
- [106] J.C. Colombo, M.F. Khalil, M. Arnac and A.C. Horth, *Environ. Sci. Technol.*, 24 (1990) 498.
- [107] W. Ślaczka, E. Andrulowicz, A. Trzosińska, *Harmful Substances, 3.2., Chlorinated Hydrocarbons (DDT and PCB Compounds), First Periodic Assessment of the State of the Marine Environment of the Baltic Sea Area, 1980–1985; Background Documents*, *Baltic Sea Environ. Proc.*, 17B, chapt. 3, 1987, p. 131.
- [108] M. Marchand, *Rev. Sci. de l'Eau*, 2 (1989) 373.
- [109] M. Marchand, A. Abarnou and C. Marcaillou-Lebaut C, *Les Polychlorobiphényles (PCB) en Milieu Marin. Biogéochimie et Ecotoxicologie, Rapports scientifiques et techniques de l'IFREMER No. 18, 1990*, IFREMER, Nantes, 1990.
- [110] J.P. Boon and J.C. Duinker, *Environ. Monit. Assess.*, 7 (1986) 189.
- [111] D.W. Kononen, *Chemosphere*, 18 (1989) 2065.
- [112] M.S. Evans, G.E. Noguchi and C.P. Rice, *Arch. Environ. Contam.*, 20 (1991) 87.
- [113] R. Tatsukawa, *Water Sci. Technol.*, 25 (1992) 1.
- [114] A. Mudroch, F.I. Onuska and L. Kalas, *Chemosphere*, 18 (1989) 2141.
- [115] E.A. Gutiérrez, G.F. Munoz, L.O. Garcia and J.A.V. Celaya, *Ciencias Marinas*, 18 (1992) 77.
- [116] C.F. Mason and W.M. O'Sullivan, *Bull. Environ. Contam. Toxicol.*, 48 (1992) 387.
- [117] J. Falandysz and K. Ośmiałowski, *Studia i Materiały Oceanologiczne*, 62 (1992) 27.
- [118] J. Falandysz and K. Kannan, *Z. Lebensm. Unters. Forsch.*, 195 (1992) 17.
- [119] K. Kannan, J. Falandysz, N. Yamashita, S. Tanabe and R. Tatsukawa, *Marine Pollut. Bull.*, 24 (1992) 358.
- [120] J. Falandysz, N. Yamashita, S. Tanabe and R. Tatsukawa, *Z. Lebensm. Unters. Forsch.*, 194 (1992) 120.
- [121] S. Tanabe, J. Falandysz, T. Higaki, K. Kannan and R. Tatsukawa, *Environ. Pollut.*, 79 (1993) 45.
- [122] S. Tanabe, N. Kannan, M. Fukushima, T. Okamoto, T. Wakimoto and R. Tatsukawa, *Marine Pollut. Bull.*, 20 (1989) 344.
- [123] M. Oehme, S. Mano and W. Thomas, *Fresenius Z. Anal. Chem.*, 321 (1985) 655.
- [124] S. Sabbah, M.R. Driss and M.L. Bouguerra, *Analysis*, 17 (1989) 259.
- [125] D.M. Lockerbie and T.A. Clair, *Bull. Environ. Contam. Toxicol.*, 41 (1988) 625.
- [126] C.F. Mason, *Chemosphere*, 26 (1993) 2289.
- [127] A.T.C. Bosveld, J. Gradener, M. van Kampen, A.J. Murk, E.G.H. Evers and M. Van den Berg, *Chemosphere*, 27 (1993) 419.
- [128] E.D. Goldberg, *Sci. Total Environ.*, 100 (1991) 17.
- [129] R.J. Maguire and R.J. Tkacz, *Arch. Environ. Contam. Toxicol.*, 25 (1993) 220.
- [130] D. Couillard, *Environ. Pollut.*, B3 (1982) 239.
- [131] B.G. Oliver and K.L.E. Kaiser, *Water Pollut. Res. J. Canada*, 21 (1986) 344.
- [132] K.L.E. Kaiser, M.E. Comba, H. Hunter, R.J. Maguire, R.J. Tkacz and R.F. Platford, *J. Great Lakes Res.*, 11 (1985) 386.
- [133] E. Plöger, *Gewässerschutz-Wasser-Abwasser*, 72 (1984) 15.
- [134] H. Bohlen, K. Hicke, A.O. Stöbel, M. Zierott, W. Thiemann, *Vom Wasser*, 72 (1989) 185.
- [135] T. Galamon and Z. Paszkiewicz, *Roczn. PZH*, 40 (1989) 297.
- [136] D.A. Hinckley and T.F. Bidleman, *Environ. Sci. Technol.*, 23 (1989) 995.
- [137] R.J. Maguire and R.J. Tkacz, *Chemosphere*, 19 (1989) 1277.
- [138] M.Th.J. Hillebrand, J.M. Everaarts, H. Razak, D. Moelyadi Moelyo, L. Stolwijk and J.P. Boon, *Netherlands J. Sea Res.*, 23 (1989) 369.
- [139] R. Reupert and E. Plöger, *Gewässerschutz-Wasser-Abwasser*, 106 (1989) 219.
- [140] M.A. Alawi, M.K. Fayyad and I. Issa, *Dirasat*, B17 (1990) 83.

- [141] J.F.C. Montanez, R.W. Risebrough, B.W. De Lappe, M.G. Marino and J. Albaigés, *Marine Pollut. Bull.*, 21 (1990) 518.
- [142] J.C. Molto, C. Albelda, G. Font and J. Manes, *Int. J. Environ. Anal. Chem.*, 41 (1990) 21.
- [143] S.W. Fowler, *Marine Environ. Res.*, 29 (1990) 1.
- [144] M.B. Vojinović, S.T. Pavkov and D.D. Buzarov, *Water Sci. Tech.*, 22 (1990) 107.
- [145] G.H. Tan, S.H. Goh, K. Vijayaletchumy, C.P. Loong and K.L. Ang, *Bull. Sing. N.I. Chem.*, 18 (1990) 67.
- [146] C.M. Cooper, *Arch. Hydrobiol.*, 121 (1991) 103.
- [147] J. Faller, H. Hühnerfuss, W.A. König and P. Ludwig, *Marine Pollut. Bull.*, 22 (1991) 82.
- [148] R. Frank, L. Logan and B.S. Clegg, *Arch. Environ. Contam. Toxicol.*, 21 (1991) 585.
- [149] A. Provini, E. Di Maio and S. Galassi, *Toxicol. Environ. Chem.*, 31/32 (1991) 157.
- [150] A.M. Abd-Allah, *Toxicol. Environ. Chem.*, 36 (1992) 89.
- [151] L.M. Hernandez, M.A. Fernandez and J. Gonzalez, *Bull. Environ. Contam. Toxicol.*, 46 (1991) 9.
- [152] S. Fingler, V. Drevencar, B. Tkalčević and Z. Smit, *Bull. Environ. Contam. Toxicol.*, 49 (1992) 805.
- [153] A. Di Corcia and M. Marchetti, *Environ. Sci. Technol.*, 26 (1992) 66.
- [154] G.E. Miliadis, *Bull. Environ. Contam. Toxicol.*, 50 (1993) 247.
- [155] K. Senkpiel, O. Lenze, H. Ohgke and J. Beckert, *Forum Städte-Hygiene*, 42 (1991) 120.
- [156] M. Chevreuil and L. Granier, *Water, Air and Soil Pollution*, 59 (1991) 217.
- [157] S. Litten, B. Mead and J. Hassett, *Environ. Toxicol. Chem.*, 12 (1993) 639.
- [158] A. Żelechowska, Z. Makowski and J. Karnas, *Ochrona Środowiska*, 3(44) (1991) 7.
- [159] A. Żelechowska and Z. Makowski, *Gaz, Woda i Technika Sanitarna*, 2/3 (1990) 35.
- [160] G.D. Foster, P.M. Gates, W.T. Foreman, S.W. McKenzie and F.A. Rinella, *Environ. Sci. Technol.*, 27 (1993) 1911.
- [161] B.A. Tomkins, R. Merriweather and R.A. Jenkns, *J. AOAC Int.*, 75 (1992) 1091.
- [162] A. Tanamati, A.F. Rubira, M. Matsushita and N.E. de Souza, *Arq. Biol. Tecnol.*, 34 (1991) 303.
- [163] F. Hernandez, J. Beltran and J.V. Sancho, *Sci. Total Environ.*, 132 (1993) 297.
- [164] L.J. Janiot, A.M. Orlando and O.E. Roses, *Acta Farmac. Bonaerense*, 10 (1991) 15.
- [165] A. Brambilla, B. Rindone, S. Polesello, S. Galassi and R. Balestrini, *Sci. Total Environ.*, 132 (1993) 339.
- [166] W.E. Pereira and F.D. Hostettler, *Environ. Sci. Technol.*, 27 (1993) 1542.
- [167] T. Pham, K. Lum and C. Lemieux, *Chemosphere*, 26 (1993) 1595.
- [168] E. Niemirycz, E. Korzec and Z. Makowski, *Outflow of some organic substances transported by the Vistula River into the Baltic Sea*, Proc. 16th Conference of the Baltic Oceanographers, Kiel, September 1988, p. 760.
- [169] R.J. Maguire and R.J. Tkacz, *Water Pollut. Res. J. Canada*, 23 (1988) 292.
- [170] C. Rico, L. Hernandez, M. Fernandez, J. Gonzalez and M. Montero, *Water Res.*, 23 (1989) 57.
- [171] A. Żelechowska, Z. Makowski and J. Rybiński, *Discharge of some pesticides from the small agricultural draining area of the Gulf of Gdańsk*, Proc. 16th Conference of the Baltic Oceanographers, Kiel, September 1988, p. 1183.
- [172] P.J. Wangersky, in O. Hutzinger (ed.), *Organic Material in Sea Water*, w *The Handbook of Environmental Chemistry*, vol. 1/Part C, Springer, Berlin, 1984, p. 25.
- [173] M. Kessabi, A. Elhraiki and B. Nader, *Sci. Total Environ.*, 71 (1988) 209.
- [174] H. Koboyashi, K. Ohyama, N. Tomiyama, Y. Jimbo, O. Matano and S. Goto, *J. Chromatogr.*, 643 (1993) 197.
- [175] T. Tsuda, S. Aoki, M. Kojima and T. Fujita, *Toxicol. Environ. Chem.*, 41 (1994) 85.
- [176] J.C. Molto, Y. Pico, J. Manes and G. Font, *J. AOAC Int.*, 75 (1992) 714.
- [177] J.P. Thome, M. Louvet and J.L. Hugla, *Bulletin de la Societe Royale des Sciences de Liege*, 63 (1994) 167.
- [178] R.W. Risebrough, B.W. de Lappe and Ch. Youngmans-Haug, *Mar. Pollut. Bull.*, 21 (1990) 523.
- [179] R.J. Norstrom and D.C.G. Muir, *Sci. Total Environ.*, 154 (1994) 107.
- [180] A.S. Mayer and co-workers, *Fate and Effects of Pollutants*, June 1995, 629.
- [181] M. Sanchez-Camazano, M.J. Sanchez-Martin and T. Cris-tanto, *Toxicol. Environ. Chem.*, 47 (1995) 203.
- [182] J.M. Portal, M. Babut, M. Schiavon and B. Gerard, *J. Eur. d'Hydro.*, 25 (1994) 135.
- [183] J. Gascón, E. Martinez and D. Barceló, *Anal. Chim. Acta*, 311 (1995) 357.
- [184] C. Skark and P. Obermann, *Int. J. Environ. Anal. Chem.*, 58 (1995) 163.
- [185] C. Skark and N. Zullei-Seibert, *Int. J. Environ. Anal. Chem.*, 58 (1995) 387.
- [186] H.Y. Ng, J.D. Gaynor, C.S. Tan and C.F. Drury, *Water Res.*, 29 (1995) 2309.
- [187] S.P. Scholter and S.J. Eisenreich, *Environ. Sci. Technol.*, 26 (1994) 2228.
- [188] J.D. Fischer, B.E. Apedaile and L.K. Vanclief, *Water Qual. Res. J. Canada*, 30 (1995) 533.
- [189] L.J. Janiot, J.L. Sericano and O.R. Roses, *Water, Air Soil Pollut.*, 76 (1994) 323.
- [190] H. Iwata, S. Tanabe, M. Aramoto, N. Sakai and R. Tatsukawa, *Marine Pollut. Bull.*, 28 (1994) 746.
- [191] A. Dankwart, S. Wüst, W. Elling, E.M. Thurman and B. Hock, *Environ. Sci. Pollut. Res.*, 1 (1994) 196.
- [192] Ch. Guyot, *Chem. Plant Protection*, 9 (1994) 87.
- [193] B. Stachel, O. Elscholzh and H. Reincke, *Fresenius J. Anal. Chem.*, 353 (1995) 21.
- [194] P.J. Chilton, A.R. Lawrence and J.A. Barker, *Hydrological, Chemical and Biological Processes of Transformation and Transport of Contaminants in Aquatic Environment*, Proceedings of the Rostov-on-Don Symposium, May 1993, IAHS Publ. No. 219, 1994, p. 51.
- [195] D.E. Line and co-workers, *Fate and Effects of Pollutants*, June 1995, 685.

- [196] S. Hall, J. Chamberlain and E. Godwin-Saad, *Fate and Effects of Pollutants*, June 1995, 713.
- [197] D.H. Hutson and T.R. Roberts (Editors), *Environmental Fate Pesticides*, John Wiley and Sons Ltd., 1990.
- [198] G. Bormann, J. Gandraz, H.-D. Knauth and R.-D. Wilken, *Vom Wasser*, 80 (1993) 15.
- [199] J.E.M. Beurskens, H.J. Winkles, J. de Wolf and C.G.C. Dekker, *Water Sci. Technol.*, 29 (1994) 77.
- [200] R. Hobby and R. Gimbel, *Water Sci. Technol.*, 29 (1994) 87.
- [201] J. Gascón, G. Durand and D. Barceló, *Environ. Sci. Technol.*, 29 (1995) 1551.
- [202] C. Lemieux, B. Quémerais and K.R. Lum, *Water Res.*, 29 (1995) 1491.
- [203] J.J. Miller, N. Foroud, B.D. Hill and C.W. Lindwall, *Can. J. Soil Sci.*, 75 (1995) 145.
- [204] C. Gómez-Gómez and co-workers, *Bull. Environ. Contam. Toxicol.*, 55 (1995) 431.
- [205] P.B. McMahon, D.W. Litke, J.E. Paschal and K.F. Dennehy, *Water Res. Bull.*, June 1994, 521.
- [206] Ch. A. Bishop and co-workers, *Environ. Toxicol. Chem.*, 14 (1995) 491.
- [207] M.A. Miller and J.F. Amrhein, *Bull. Environ. Contam. Toxicol.*, 55 (1995) 96.
- [208] K. Bester, H. Hühnerfuss, B. Neudorf and W. Theimann, *Chemosphere*, 30 (1995) 1639.
- [209] N.P. Agnihotri, V.T. Gajbhiye, M. Kumar and S.P. Mohapatra, *Environ. Monit. and Assessment*, 30 (1994) 105.
- [210] P.A. Kosian, R.A. Hoke, G.T. Ankley and F.M. Vandermeiden, *Environ. Toxicol. Chem.*, 14 (1995) 445.
- [211] H.G.J. Mol, H.-G.M. Janssen, C.A. Cramers, J.J. Vreuls and U.A.Th. Brinkman, *J. Chromatogr. A*, 703 (1995) 277.
- [212] A.J. Beck and co-workers, *J. Contam. Hydrol.*, 19 (1995) 237.
- [213] A.J. Beck and co-workers, *J. Agric. Food Chem.*, 43 (1995) 1368.
- [214] L.W. Canter and K.M. Maness, *Int. J. Environ. Studies*, 47 (1995) 1.
- [215] J.D. Petty, J.N. Huckins, D.B. Martin and T.G. Adornato, *Chemosphere*, 30 (1995) 1891.
- [216] V.S. Rao, *Geobios*, 21 (1994) 68.
- [217] J.M. Luco, E.G. Aguilar, G. Silva, O.M. Baudano and D.M. Gonzalez, *Acta Farm. Bonaerense*, 11 (1992) 121.
- [218] B. Kuhlmann, B. Kaczmarczyk and U. Schöttler, *Int. J. Environ. Anal. Chem.*, 58 (1995) 199.
- [219] M. Kamal and I. Scheunert, *Fresenius Environ. Bull.*, 2 (1993) 137.
- [220] J.D. Gaynor, C.S. Tan, C.F. Drury, I.J. Van Wesenbeeck and T.W. Welacky, *Water Qual. Res. J. Canada*, 30 (1995) 513.
- [221] J. Gandras, G. Bormann and R.-D. Wilken, *Fresenius J. Anal. Chem.*, 353 (1995) 70.
- [222] K. Fytianos, *Fresenius Environ. Bull.*, 4 (1995) 401.
- [223] T.A. Albanis, T.G. Danis and D.G. Hela, *Sci. Total Environ.*, 171 (1995) 85.
- [224] D. Barceló, C. Porte, J. Cid and J. Albigés, *Int. J. Environ. Anal. Chem.*, 38 (1990) 199.
- [225] M. Marchand, *Rev. Sci. de l'Eau*, 2 (1989) 229.
- [226] D.E. Schulz-Bull, G. Petrick, N. Kannan and J.C. Duinker, *Marine Chem.*, 48 (1995) 245.
- [227] D.E. Schulz-Bull, G. Petrick and J.C. Duinker, *Marine Chem.*, 36 (1991) 365.
- [228] P.J. Vourinen, T. Rantio, A. Witick and M. Vuorinen, *Aqua Fennica*, 24,1 (1994) 29.
- [229] M. Minunni and M. Mascini, *Anal. Lett.*, 26 (1993) 1441.
- [230] R.J. Meesters, F. Forge and F. Schröder, *Vom Wasser*, 84 (1995) 287.
- [231] R.I. McConnell, J.V. Lamont and S.P. Fitzgerald, *Food Agric. Immunol.*, 6 (1994) 401.
- [232] J. Carré, S. Bouteiller, J. Dufils and M. Freslon, *J. Europ. d'Hydrol.*, 25 (1994) 169.
- [233] D.R. Briggs and D.E. Moerman, *Water Qual. Res. J. Can.*, 30 (1995) 429.
- [234] S. Schleyer, J. Hammer and J. Fillibeck, in K. Kovar and J. Soveri (Editors), *Groundwater Quality Management*, IAHS Press, Inst. of Hydrology, Wallingford, Oxfordshire, 1994, p. 73.
- [235] R.A. Baumann, V.M. 't Hart-de Kleijn and R. Hoogerbrugge, *Int. J. Environ. Anal. Chem.*, 58 (1995) 1.
- [236] V. Gouy and R. Bélamie, *Water Sci. Tech.*, 28 (1993) 679.
- [237] D.T. Waite and co-workers, *Environ. Toxicol. Chem.*, 14 (1995) 1171.
- [238] D.C.G. Muir and co-workers, *Sci. Total Environ.*, 160/161 (1995) 447.
- [239] R.W. Riserbrough, B.W. De Lappe and Ch. Younghans-Haug, *Marine Pollut. Bull.*, 21 (1990) 523.
- [240] M.M. Schantz, R.M. Parris, J. Kurz, K. Ballschmiter and S.A. Wise, *Fresenius J. Anal. Chem.*, 346 (1993) 766.
- [241] D.A. Brigt, W.T. Dushenko, S.L. Grundy and K.J. Reimer, *Sci. Total Environ.*, 160/161 (1995) 265.
- [242] O. Nagafuchi, T. Inoue and S. Ebise, *Water Sci. Tech.*, 30 (1994) 137.
- [243] K.L.E. Kaiser, K.R. Lum, M.E. Comba and V.S. Palabrica, *Sci. Total Environ.*, 97/98 (1990) 23.
- [244] W.E. Pereira, F. Hostettler, J.R. Cashman and R.S. Nishioka, *Marine Pollut. Bull.*, 28 (1994) 434.
- [245] H. Urdaneta, B. Medina and Z. Acosta, *Bull. Environ. Contam. Toxicol.*, 54 (1995) 703.
- [246] M.A. Miller, C.P. Madenjian and R.G. Masnado, *J. Great Lakes Res.*, 18 (1992) 742.
- [247] M. Chevreuil, L. Granier and A.-M. Carru, *Water, Air Soil Pollut.*, 81 (1995) 107.
- [248] M.A. Miller, *Arch. Environ. Contam. Toxicol.*, 27 (1994) 367.
- [249] M.A. Miller N.A. Kassulke and M.D. Walkowski, *Arch. Environ. Contam. Toxicol.*, 25 (1993) 212.
- [250] M.A. Miller, *Can. J. Fish Aquat. Sci.*, 50 (1993) 1405.
- [251] Ch.A. Bishop, D.R.S. Lean, R.J. Brooks, J.H. Carey and P. Ng, *Environ. Toxicol. Chem.*, 14 (1995) 421.
- [252] W. Vetter and co-workers, *Chemosphere*, 30 (1995) 1685.
- [253] Ch.P. Madenjian, S.R. Carpenter, G.W. Eck and M.A. Miller, *Can. J. Fish Aquat. Sci.*, 50 (1993) 97.
- [254] A. Borell, D. Bloch and G. Desportes, *Environ. Pollut.*, 88 (1995) 283.

- [255] R. Wilson, S. Allen-Gil, D. Griffin and D. Landers, *Sci. Total Environ.*, 160/161 (1995) 511.
- [256] D. Pastor, L. Jover, X. Ruiz and J. Albaigés, *Sci. Total Environ.*, 162 (1995) 215.
- [257] S. Mössner and co-workers, *Fresenius J. Anal. Chem.*, 349 (1994) 708.
- [258] A.C. de Kock, P.B. Best, V. Cockcroft and C. Bosma, *Sci. Total Environ.*, 154 (1994) 153.
- [259] B. Streit, *Water Sci. Tech.*, 29 (1994) 145.
- [260] M. Oehme, M. Schlabach, K. Hummert, B. Lukas and E.S. Nordoy, *Sci. Total Environ.*, 162 (1995) 75.
- [261] M. Matsushita and N.E. de Souza, *Arq. Biol. Technol.*, 637 (1994) 637.
- [262] N. Sotiriou, K. Ouzounis and E.S. Lahaniatis, *Fresenius Environ. Bull.*, 1 (1992) 203.
- [263] R.R. Trussel and M.D. Umphres, *Water Technol. Qual. JAWWA*, November 1988, 604.
- [264] O. Hutzinger (Editor), *The Handbook of Environmental Chemistry*, Vol. 1, Part A, *The natural Environment and Biogeochemical Cycles*, O. Hutzinger (Editor), Springer Verlag, Berlin, 1980.
- [265] B. Strait and K. Kuhn, *Water Sci. Tech.*, 29 (1994) 233.
- [266] D.V. Weseloh, P. Hamr, Ch.A. Bishop and R.J. Norstrom, *J. Great Lakes Res.*, 21 (1995) 121.
- [267] G. Baldauf, *Vom Wasser*, 67 (1986) 11.
- [268] M. Biziuk and A. Przyjazny, *J. Chromatogr. A*, 733 (1996) 417.
- [269] M. Dressler, *J. Chromatogr.*, 165 (1979) 167.
- [270] S.K. Poole, T.A. Dean, J.W. Oudsema and C.F. Poole, *Anal. Chim. Acta*, 236 (1990) 3.
- [271] J.A. Leenheer, *Water Analysis*, Vol. III, chapt. 3, *Concentration, partitioning and isolation techniques*, Academic Press, Oxford, 1984, p. 83.
- [272] J. Namieśnik, T. Górecki, M. Biziuk and L. Torres, *Anal. Chim. Acta*, 237 (1990) 1.
- [273] L. Stieglitz, *Kemia-Kemi*, 4 (1977) 127.
- [274] R.E. Majors, *LC-GC*, 11 (1993) 336.
- [275] J.F. Barker and R. Dickhout, *Ground Water Monit. Rev.*, 8 (1988) 112.
- [276] S. Lesage and R.E. Jackson, *Groundwater Contamination and Analysis at Hazardous Waste Sites*, Marcel Dekker, New York, 1992, p. 3.
- [277] R. Leboda, A. Łodyga, D. Sieńko and P. Grochowicz, *Wiadom. Chem.*, 45 (1991) 217.
- [278] C.C. Smith, *Environ. Sci. Res.*, 15 (1979) 227.
- [279] D. Rossi, R. Baudo and H. Muntau, *Acqua-Aria*, April 1992, 309.
- [280] H.J. Brauch and S. Schullerer, *Fresenius J. Anal. Chem.*, 339 (1991) 420.
- [281] M.K. Stenstrom, S. Fam and G.S. Silverman, *Environ. Technol. Lett.*, 7 (1986) 625.
- [282] R.E. Majors, *LC-GC*, 4 (1991) 10.
- [283] H.J. Brass, *Research and Technology*, *JAWWA*, February 1982, 107.
- [284] A. Bruchet, *Analysis*, 19 (1991) 123.
- [285] H. Hühnerfuss and H. Kallenborn, *J. Chromatogr.*, 580 (1992) 191.
- [286] W. Gisbach and E. Kleemann, *Acta Hydrochim. Hydrobiol.*, 21 (1993) 89.
- [287] R. Eiser, K. Levsen and G. Wünsch, *Int. J. Environ. Anal. Chem.*, 58 (1995) 103.
- [288] L. Starostin and Z. Witkiewicz, *Chem. Anal.*, 39 (1994) 263.
- [289] E. Burestedt, J. Emnéus, L. Gorton, G. Marko-Varga, E. Dominguez, F. Ortega, A. Narváez, H. Irth, M. Lutz, D. Puig and D. Barceló, *Chromatographia*, 41 (1995) 207.
- [290] M.-C. Hennion and V. Coquart, *J. Chromatogr.*, 642 (1993) 211.
- [291] J. Slobodnik, E.R. Brouwer, R.B. Geerdink, W.H. Mulder, H. Lingeman and U.Th. Brinkman, *Anal. Chim. Acta*, 268 (1992) 55.
- [292] A. Di Corcia, S. Marchese and R. Samperi, *J. Chromatogr.*, 642 (1993) 163.
- [293] M. Moors, D.L. Massart and R.D. McDowall, *Pure Appl. Chem.*, 66 (1994) 277.
- [294] U.A.Th. Brinkman, Th. Hankemeier and J.J. Vreuls, *Chem. Anal.*, 40 (1995) 495.
- [295] D. Barceló, *J. Chromatogr.*, 643 (1993) 117.
- [296] J.S. Ho, P.H. Tang and W.L. Budde, *J. Chromatogr. Sci.*, 33 (1995) 1.
- [297] T. Greibrokk, *J. Chromatogr. A*, 703 (1995) 523.
- [298] R.W. Vannoort, J.-P. Chervet, H. Lingeman, G.J. Dejong and U.A.Th. Brinkman, *J. Chromatogr.*, 505 (1990) 45.
- [299] Z. Witkiewicz, *Wiadomości Chemiczne*, 47 (1993) 193.
- [300] J.M. Bayona and Y. Cai, *Trends Anal. Chem.*, 13 (1994) 327.
- [301] M. Lee and K.E. Markides (Editors), *Analytical Supercritical Fluid Chromatography and Extraction*, Chromatography Conferences Inc., Provo, Utah, USA, Chapter 5, (Analytical Supercritical Fluid Extraction) str 313–362.
- [302] Y. Janda, K.D. Bartle and A.A. Clifford, *J. Chromatogr.*, 642 (1993) 283.
- [303] W. Pipkin, *LC-GC Int.*, 5 (1992) 8.
- [304] C.H. Kirschner and L.T. Taylor, *J. High Resolut. Chromatogr.*, 16 (1993) 73.
- [305] D.C. Messer and L.T. Taylor, *J. Chromatogr. Sci.*, 33 (1995) 290.
- [306] U. Ullsten and K.E. Markides, *J. Microcol. Sep.*, 6 (1994) 385.
- [307] F. David, M. Verschuere and P. Sandra, *Fresenius J. Anal. Chem.*, 344 (1992) 479.
- [308] Y. Cai and J.M. Bayona, *J. Chromatogr. Sci.*, 33 (1995) 89.
- [309] P. Sandra, A. Kot, A. Miedvedovici and F. David, *J. Chromatogr. A*, 703 (1995) 467.
- [310] A.L. Howard and L.T. Taylor, *J. Chromatogr. Sci.*, 30 (1992) 374.
- [311] R. Eisert and K. Levsen, *GIT Fachz. Lab.*, 1 (1995) 25.
- [312] P. Popp, G. Kalbitz and G. Oppermann, *J. Chromatogr. A*, 687 (1994) 133.
- [313] P. Popp, A. Kanert and K. Kalbitz, *GIT Fachz. Lab.*, 39 (1995) 325.
- [314] R. Eisert and K. Levsen, *Fresenius J. Anal. Chem.*, 351 (1995) 555.
- [315] R. Eisert, K. Levsen and G. Wünsch, *J. Chromatogr. A*, 683 (1994) 175.

- [316] R. Eisert and K. Levsen, *J. Am. Soc. Mass Spectrom.*, 6 (1995) 1119.
- [317] V. Vandepitte and co-workers, *Water, Air Soil Pollut.*, 78 (1994) 335.
- [318] M.F. Katmeh, G. Frost, W. Aherne and D. Stevenson, *Analyst*, 119 (1994) 431.
- [319] R. Kindervater, W. Künnecke and R.D. Schmid, *Anal. Chim. Acta*, 234 (1990) 113.
- [320] K. Grob and Z. Li, *J. Chromatogr.*, 473 (1989) 423.
- [321] V. Lopez-Avila, R. Wesselman and K. Edgell, *J. Assoc. Off. Anal. Chem.*, 73 (1990) 276.
- [322] K. Kadokami and co-workers, *Anal. Sci.*, 11, June 1995, 375.
- [323] L.M. Davi, M. Baldi, L. Penazzi and M. Liboni, *Pestic. Sci.*, 35 (1992) 63.
- [324] B.B. Mogensen and N.H. Spliid, *Chemosphere*, 31 (1995) 3977.
- [325] C. Barbieri, G. Beneventi, G. Del Carlo, S. Forti and V. Romano, *Boll. Chim. Igien.*, 45 (1994) 235.
- [326] L. Ogierman, *Chem. Anal.*, 37 (1992) 303.
- [327] J. Tekel, K. Schultzová and J. Kovačicová, *J. High Resolut. Chromatogr.*, 16 (1993) 126.
- [328] R. Smith and A.J. Hassett, NIWR Interlaboratory comparison study No. 81/C: Determination of chlorinated hydrocarbon pesticides in water. Evaluation of results, CSIR Research Report No. 572, National Institute for Water Research, Council for Sci. and Ind. Res., Pretoria, 1982.
- [329] K. Pomorska, *Chem. Anal.*, 38 (1993) 297.
- [330] M. Marchand, J.C. Caprais, P. Pignet and V. Porot, *Water Res.*, 23 (1989) 461.
- [331] G.D. Foster, W.T. Foreman and P.M. Gates, *J. Agric. Food Chem.*, 39 (1991) 1618.
- [332] E.A. Hogendoorn, Strategies in Method Development for the Determination of Polar Pesticides with Coupled-Column Liquid Chromatography, *Academisch Proefschrift, Elinkwijk BV, Utrecht* 1993.
- [333] S.M. Lee and P.L. Wylie, *J. Agric. Food Chem.*, 39 (1991) 2192.
- [334] S. Smith Jr., Effects of tillage on pesticide concentrations in shallow ground water and surface runoff from fragipan soils in North Mississippi, USA, in Y. Eckstein and A. Zaporozec (Editors), *Environmental Impact of Agricultural Practices and Agrichemicals*, Water Environment Federation, Alexandria, 1994.
- [335] I. Harrison, R.U. Leader, J.J.W. Higgo and J.C. Tjell, *J. Chromatogr. A*, 688 (1994) 181.
- [336] J. Ducreux, R. Boulet, J. Roussel and N. Petroff, *Int. J. Environ. Anal. Chem.*, 12 (1982) 195.
- [337] C. Fraschini, D. Cottica and P. Scrocco, *Boll. Chim. Igien.*, 41 (1990) 97.
- [338] R.A. Van Steenderen, *Water SA*, 7 (1981) 27.
- [339] R.A. Van Steenderen, *Lab. Pract.*, 29 (1980) 788.
- [340] M. Von Hahn, H.H. Rüttinger and H. Matschiner, *Z. Wasser-Abwasser-Forsch.*, 24 (1991) 191.
- [341] I. Liška, J. Krupčík and P.A. Leclercq, *J. High Res. Chromatogr.*, 12 (1989) 577.
- [342] M.W. Powell, *J. Chromatogr. A*, 697 (1995) 101.
- [343] J.K. Stamer, R.B. Swanson and P.R. Jordan, *Water Res. Bull.*, 30 (1994) 823.
- [344] D.R. Green and D. Kowalski, Amberlite XAD-2 Resin Columns. Documentation of their Performance in the Extraction of Organics From Water, Seastar Instr. Ltd., Sidney (Canada), September 1984.
- [345] A. Di Corcia, S. Marchese and R. Samperi, *J. Chromatogr.*, 642 (1993) 175.
- [346] A. Di Corcia, A. Marconini and R. Samperi, *Anal. Chem.*, 65 (1993) 907.
- [347] M. Battista, A. Di Corcia and M. Marchetti, *Anal. Chem.*, 61 (1989) 935.
- [348] V. Adamović, *Hrana i Ishrana*, 24 (1983) 9.
- [349] F. Zilio Grandi, L. Szpyrkowicz and J. Naumczyk, *Toxicol. Environ. Chem.*, 28 (1990) 195.
- [350] R.A. Moore and F.W. Karasek, *Int. J. Environ. Anal. Chem.*, 17 (1984) 187.
- [351] G.A.V. Rees and L. Au, *Bull. Environm. Contam. Toxicol.*, 22 (1979) 561.
- [352] G.E. Carlberg and L. Martinsen, *Sci. Total Environ.*, 25 (1982) 245.
- [353] A. Bruchet, K. Alben, W.-H. Ding and K. Aldous, *Proc. 1986 Water Qual. Technol. Conf. AWWA, AWWA, Denver*, 1987, p. 905.
- [354] B. Zygmunt, A.T. Brinkman and R.W. Frei, *Toxicol. Environ. Chem.*, 7 (1983) 1.
- [355] R.L. Jolley, L.W. Condie, J.D. Johnson, S. Katz, R.A. Minear, J.S. Mattice and V.J. Jacobs. (Editors), *Water Chlorination. Chemistry. Environmental Impact and Health Effects.*, Vol. 6, Lewis Publishers, Chelsea, 1990.
- [356] B.W. Lykins, J.A. Goodrich, W.E. Koffskey and M.H. Griesse, *Proc. Annual Conf. AWWA, Philadelphia*, June 23–27, 1991, AWWA, Denver, 1991, p. 897.
- [357] P. Van Rossum and R.G. Webb, *J. Chromatogr.*, 150 (1978) 381.
- [358] M. Kilarska, R. Rajtar, W. Solarski and E. Zieliński, *Chem. Anal.*, 37 (1992) 279.
- [359] A. Przyjazny, *J. Chromatogr.*, 346 (1985) 61.
- [360] B. Wigilius, H. Borén, G.E. Carlberg, A. Grimvall, B.V. Lundgren and R. Sävenhed, *J. Chromatogr.*, 391 (1987) 169.
- [361] K.M.S. Sundaram, S.Y. Szeto and R. Hindle, *J. Chromatogr.*, 177 (1979) 29.
- [362] J.K. Fawell, M. Fielding and J.W. Ridgway, *J. Instit. Water Environ. Manag.*, 1 (1987) 61.
- [363] H. Horth and co-workers, I.H. Suffet and M. Malaiyandi (Editors), *Techniques for the Fractionation and Identification of Mutagens Produced by Water Treatment Chlorination, w Organic Micropollutants in Water. Sampling, Analysis and Toxicity Testing*, Amer. Chem. Soc., Washington, 1987, p. 659.
- [364] S.A. Daignault, D.K. Noot, D.T. Williams and P.M. Huck, *Water. Res.*, 22 (1988) 803.
- [365] J.P. Franke, J. Wijsbeek, J.F. Greving and R.A. de Zeeuw, *Arch. Toxicol.*, 42 (1979) 115.
- [366] W.R. McKoen, J.J. Muldowney and B.S. Aptowicz, *Proc. Annual Conf. AWWA, 1986, AWWA, Denver*, 1986, p. 967.

- [367] B. Nouri, G. Toussaint, P. Chambon and R. Chambon, *Analyst*, 120 (1995) 2683.
- [368] M.P. Maskarinec and D.L. Manning, Application of XAD-4 Solid Sorbent to the Collection of Pesticides from Water Samples, Final Report, Oak Ridge National Laboratory, Martin Marietta, Springfield (USA), October 1987.
- [369] V.N. Mallet, J.M. Francoeur and G. Volpé, *J. Chromatogr.*, 172 (1979) 388.
- [370] J. Alberti and W. Stock, *Gewässerschutz-Wasser-Abwasser*, 106 (1989) 204.
- [371] D. Spuziak-Salzenberg and W. Schröer, *Chemosphere*, 17 (1988) 2407.
- [372] M. Biziuk, J. Czerwiński and E. Kozłowski, *Int. J. Environ. Anal. Chem.*, 50 (1993) 109.
- [373] M. Giabbai, L. Roland, M. Ghosal, J.H. Reuter and E.S.K. Chian, *J. Chromatogr.*, 279 (1983) 373.
- [374] G.R. Aiken, in F. Frimmel and R.F. Christman (Editors), *Humic Substances and their Role in the Environment*, John Wiley and Sons, S. Bernhard, 1988, pp. 15–28.
- [375] M.O. Rivett, D.N. Lerner and J.W. Lloyd, *Ground Water Monit. Rev.*, 10 (1990) 127.
- [376] A.P. Bianchi, M.S. Varney and J. Phillips, *J. Chromatogr.*, 557 (1991) 429.
- [377] J. Freudenthal, *Int. J. Environ. Anal. Chem.*, 5 (1978) 311.
- [378] M. Biziuk and J. Tronczyński, Związki organiczne w środowisku i metody ich oznaczania, Proceedings of the conference, Jachranka 18–21 mai 1993, Biblioteka Monitoringu Środowiska, Warszawa 1993, p. 187.
- [379] M. Biziuk and J. Tronczyński, Metody oznaczania związków organicznych w wodach, Proceedings of the conference, Politechnika Gdańska, 22 april 1993, Komitet Chemii Analitycznej PAN, Katedra Chem. Anal. Polit. Gdańskiej, IMGW, Warszawa 1993, pp. 28–47.
- [380] M. Biziuk and J. Tronczyński, *Int. J. Environ. Anal. Chem.*, in print.
- [381] M.W. Brooks, J. Jenkins, M. Jimenez, T. Quinn and J.M. Clark, *Analyst*, 114 (1989) 405.
- [382] J. Manez Vanuesa, J.C. Molto Cortes, C. Igualada Canas and G. Font Perez, *J. Chromatogr.*, 472 (1989) 365.
- [383] M. Guidotti and G. Macilenti, *Boll. Chim. Igien.*, 46 (1995) 97.
- [384] S. Bengtsson and A. Ramberg, *J. Chromatogr. Sci.*, 33 (1995) 554.
- [385] T.S. Albanis and D.G. Hela, *J. Chromatogr. A*, 707 (1995) 283.
- [386] A. Janssen, M. Keuter, D.M. Zu Altenschiedesche, A. Ritzkopf and W. Treder, *GIT Fachz. Lab.*, (1995) 417.
- [387] A. Geissler and H.F. Schöler, *Chemosphere*, 23 (1991) 1029.
- [388] J.J. Saady and A. Poklis, *J. Anal. Toxicol.*, 14 (1990) 301.
- [389] J.P. Thome and Y. Vandaele, *Int. J. Environ. Anal. Chem.*, 29 (1987) 95.
- [390] S. Butz and H.-J. Stan, *J. Chromatogr.*, 643 (1993) 227.
- [391] C. de la Colina, F. Sánchez-Raero, G.D. Cancela, E. Romero and A. Pena, *Analyst*, 120 (1995) 1723.
- [392] S. Wang, M.J. Santos-Delgado and L.M. Polo-Diez, *Quim. Anal.*, 14 (1995) 84.
- [393] R. Infante and R. Pérez, *Int. J. Environ. Anal. Chem.*, 43 (1991) 165.
- [394] W.E. Johnson, N.J. Fendinger and J.R. Plimmer, *Anal. Chem.*, 63 (1991) 1510.
- [395] M.T. Meyer, M.S. Mills and E.M. Thurman, *J. Chromatogr.*, 629 (1993) 55.
- [396] H.-J. Brauch, *Acta Hydrochim. Hydrobiol.*, 21 (1993) 84.
- [397] L.L. Signorella, J.J. Delfino and D.H. Powell, *Bull. Environ. Contam. Toxicol.*, 51 (1993) 72.
- [398] K. Nick and H.F. Schöler, *Vom Wasser*, 84 (1995) 271.
- [399] Y. Keun-Sung, H. Sa-Moon and K. Jong-Ho, *Anal. Sci. Technol.*, 7 (1994) 441.
- [400] Ch.-Y.L. Hsu and R.R. Walters, *J. Chromatogr.*, 629 (1993) 61.
- [401] A.J. Debets and co-workers, *Analyst*, 117 (1992) 1355.
- [402] Ch.H. Marvin, I.D. Brindle, C.D. Hall and M. Chiba, *Anal. Chem.*, 62 (1990) 1495.
- [403] C.E. Werkhoven-Goewie, U.A.Th. Brinkman and R.W. Frei, *Anal. Chem.*, 53 (1981) 2072.
- [404] F. Moja and co-workers, *Giornale Italiano di Chimica Clinica*, 13 (1988) 145.
- [405] F. Moja and co-workers, *Giornale Italiano di Chimica Clinica*, 13 (1988) 287.
- [406] R. Schuster and A. Gratzfeld-Hüsgen, HPLC Analysis of Pesticides Traces in the ppt-Range, Application Note, Hewlett Packard, Pub. No. 12-5952-1550, 1990.
- [407] R.W. Frei, *Swiss Chem.*, 6 (1984) 55.
- [408] U. Oehmichen, *Neue DELIWA-Zeit.*, (1989) 352.
- [409] C. Schlett, *Acta Hydrochim. Hydrobiol.*, 21 (1993) 102.
- [410] J. Schülein, D. Martens, P. Spitzauer and A. Kettrup, *Fresenius J. Anal. Chem.*, 352 (1995) 565.
- [411] A. Di Guardo, R.J. Williams, P. Matthiessen, D.N. Brooke and D. Calamari, *Environ. Sci. Pollut. Res.*, 1 (1994) 151.
- [412] K.M. Moore, S.R. Jones and C. James, *Water Res.*, 29 (1995) 1225.
- [413] B. Nouri, B. Fouillet, G. Toussaint, P. Chambon and R. Chambon, *Analyst*, 120 (1995) 1133.
- [414] S. Chiron, S. Papilloud, W. Haerdi and D. Barceló, *Anal. Chem.*, 34 (1995) 1637.
- [415] Bakerbond spe, Application Notes, J.T. Baker, Philipsburg 1992.
- [416] S. Chiron, A.F. Alba and D. Barceló, *Environ. Sci. Technol.*, 27 (1993) 2352.
- [417] D. Barceló, G. Durand, N. de Bertrand and J. Albaigés, *Sci. Total. Environ.*, 132 (1993) 283.
- [418] E.A. Hogendoorn and U.A.Th. Brinkman, *J. Chromatogr.*, 644 (1993) 307.
- [419] T. McDonnell and J. Rosenfeld, *J. Chromatogr.*, 629 (1993) 41.
- [420] S.A. Senseman, T.L. Lavy, J.D. Mattice, B.M. Myers and B.W. Skulman, *Environ. Sci. Technol.*, 27 (1993) 516.
- [421] R.E. Hendriks, *LC-GC*, 6 (1993) 296.
- [422] S. Bengtsson, T. Bergolf, S. Grant and G. Jonsall, *Pestic. Sci.*, 41 (1994) 55.
- [423] J.S. Ho and W.L. Budde, *Anal. Chem.*, 66 (1994) 3716.
- [424] A. Di Guardo, D. Calamari, G. Zanin, A. Consalter and D. Mackay, *Chemosphere*, 28 (1994) 511.

- [425] J.S. Ho, P.H. Tang, J.W. Eichelberger and W.L. Budde, *J. Chromatogr. Sci.*, 33, (1995) 1.
- [426] V.S. Ong and R.H. Hites, *Environ. Sci. Technol.*, 29 (1995) 1259.
- [427] L. Braunstein and K. Spengler, *Vom Wasser*, 75 (1990) 1.
- [428] J.J. Vreuls and U.A.Th. Brinkman, *J. High Resolut. Chromatogr.*, 14 (1991) 455.
- [429] M.E. Rosen and J.F. Pankow, *J. Chromatogr.*, 537 (1991) 321.
- [430] B.N. Zegers, H.J. Gens, S.H.J. Wildenburg, H. Lingeman and U.A.Th Brinkman, *J. Chromatogr. A*, 677 (1994) 141.
- [431] J.B. Pawliszyn and N. Aleksandru, *Water Pollut. Res. J. Can.*, 24 (1989) 207.