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Trace Analysis of Pesticides by Gas Chromatography

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CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

The pesticides generally occur in water that have been affected by agricultural discharges. Pesticides are bioaccumulative and relatively stable, as well as toxic or carcinogenic, and, therefore, require close monitoring. The determination of pesticides and their degradation products by gas chromatography has been reviewed and evaluated. This report covers the literature part for the determination of organochlorine, nitrogen containing, organophosphorous and other pesticides in various water bodies, sediments and soils. The source of pollution, sampling procedures, extraction, purification and preconcentration techniques and other gas chromatographic conditions for pesticides determination have been discussed. The various gas chromatographic conditions used for the analysis of different pesticides are summarised in tabular form.

1.0 INTRODUCTION

Non-point source pollution caused by agricultural activities is an important source of water contamination. In rural areas, pesticides and nutrients are released in surface and ground water and may degrade the quality of drinking water and cause various health problems. Water is the most important and fundamental requirement for the existence of life and its pollution is the major problem now a days. Among various organic and inorganic water pollutants, pesticides are very dangerous and harmful because of their tissue degradation and carcinogenic in nature (1). The pesticides generally occur in water that have been affected by agricultural discharges. Pesticides are bioaccumulative and relatively stable, as well as toxic or carcinogenic, and, therefore, require close monitoring. Herbicides and nematicides are frequently water pollutants due to their direct application on to the plants. They are transported into ground water or leached to the surface water. The EEC Directive 80/778 (2) concerning the quality of water for human consumption, established the maximum concentration of each pesticide at 0.1 μg/L and total amount of pesticide at 5.0 μg/L. The WHO has classified the pesticides into five groups on the basis of their (LD_{50} values) hazardous nature (3). Most herbicides belong to class III (Table 1 and 2). The IARC (International Agency for Resaerch on Cancer) classified chemicals into working groups according to the degrees of evidence for carcinogenity towards human and animals. Some pesticides and their transformation products (chlorophenoxy acid herbicides, DDT, ETU, chlorophenols, some aniline derivatives etc.) are listed into group 2B-agents possibly carcinogenic to human (1). The persistence and mobility of pesticides are the key parameters responsible for the overall leaching potential of non-ionic compounds (4). Bottoni and Funari (5) evaluated the impact of 48 herbicides on ground water quality.

The EPA elaborated lists of pesticides properties which indicate their ground water contamination potential (Table 3). Barcelo (6) discussed the differences in priority lists of

Table 1: WHO Recommendations of Pesticides Hazards (3)

Sl. No.	Class	LD_{50} for rat, oral (mg/Kg body mass)	
		Solid	Liquid
I	Extremly Hazardous	≤5	≤20
II	Highly Hazardous	5-50	20-200
III	Moderately Hazardous	50-500	200-2000
IV	Slightly Hazardous	>500	>2000
V	Unlike to Present Hazard in Normal Use	>2000	>3000

Table 2: Distribution of Pesticides According to Hazardous Class (3)

Pesticides Group	Class					Total
	I	II	III	IV	V	
Rodenticids	11	10	4	-	-	25
Insecticides	26	51	62	13	13	165
Fungicides	6	5	21	30	67	129
Herbicides	-	5	26	57	119	20

Table 3: Properties of Pesticides Which Indicate Their High Groundwater Contamination Potential

Parameters	Value
Water Solubility	>30 mg L^{-1}
K_d	<5, usually <1
K_{oc}	<300
Henry's Law Constant	<10^{-2} atm. m^3 mol
Speciation	Negatively charged, fully or partially at ambient pH
Hydrolysis Half Time	>25 weeks
Photolysis Half Life	>1 week
Field Dissipation Half Life	>3 weeks

pesticides in water elaborated by the EEC and EPA. WHO requirements on pesticide residue concentrations have been reported (7), providing information about the contemporary requirements on ananlytical methods for the determination of priority pesticides and their transformation products in water samples.

Various reports have been published on pesticides analysis in different matrices in last few years. Literature survey indicates that there are only a few reports available on pesticides analysis in Indian context. It is also observed that among various analytical techniques, gas chromatography has been used frequently for pesticides analysis. Pesticide separation and identification in environmental and biological samples have been reviewed (8-18) using various analytical techniques. The pesticides pollution through air have also been reported (19). The dust particles in air adsorbed the pesticides (due to pesticides spray in agriculture and domestic use) and then contaminate water bodies, sediments and soil through rain water (20).

In view of the importance of pesticides analysis and the utility of Gas Chromatography, planning has been done to carry out the pesticides analysis in some of the Indian rivers and other water bodies. In this report attempt has been made to review the gas chromatography technique for the estimation of pesticides in water, sediments and soils. The report covers literature on sources of pesticides pollution, sampling procedures, extraction and purification, Gas Chromatogarphy as the techniques of pesticides ananlysis, detectors and other experimental conditions. The present report covers the literature from 1981 to 1997. The references used in preparing this report are compiled by searching Chemical and Analytical Abstracts of the same period. In addition to this, some important Journals like J. Hydrology, J. Environmental Engineering, Water Res., J. Chromatogr., J. Chromatogr. Sci., Chromatographia, Analyst, Anal. Chem., Fresenius J. Anal. Chem., Bull. Environ. Contam. Toxicol., Environ. Tech. Letters, Intern. J. Environ. Anal. Chem., etc. have also been reviewed.

2.0 GAS CHROMATOGRAPHY

The Gas Chromatography (GC) is one of the important technique because of its wide applicability for the determination of a variety of pollutants. The GC is applicable for the classes of pollutants which are volatile at the working temperature. Most of the pesticides are volatile at the working temperature of GC and, therefore, a large number of reports are available in literature for the determination of pesticides in various matrices by GC.

The separation of the compounds by GC is carried out by the phenomenon of adsorption or partition or both by adsorption and partition between mobile and stationary phases. It depends upon the nature of the stationary phase. The stationary phase is made up of a large number of theoretical plates and the separation efficiency is directly proportional to the number of theoretical plates. The equilibrium of the pollutants being analysed exits between the two neighbouring theoretical plates as the pollutants travel in the column. As a results of this sort of equilibrium among the million and billion theoretical plates pollutants get separated and retained in the stationary phase for different times.

There are two phases in GC i.e. mobile phase which carry the compounds forward through the column and the stationary phase packed in the column. The different components of the Gas Chromatographic system are shown in Fig. 1. The various parts of GC and their functions are discussed below.

Mobile Phase : The most commonly used mobile phases are nitrogen, hydrogen, helium, methane etc. These gases were used at different rates to achieve the best resolution of pesticides. The function of the mobile phase is to carry out the volatilised pollutants through the column and separate them by adsorption or partition or both by adsorption and partition phenomenon.

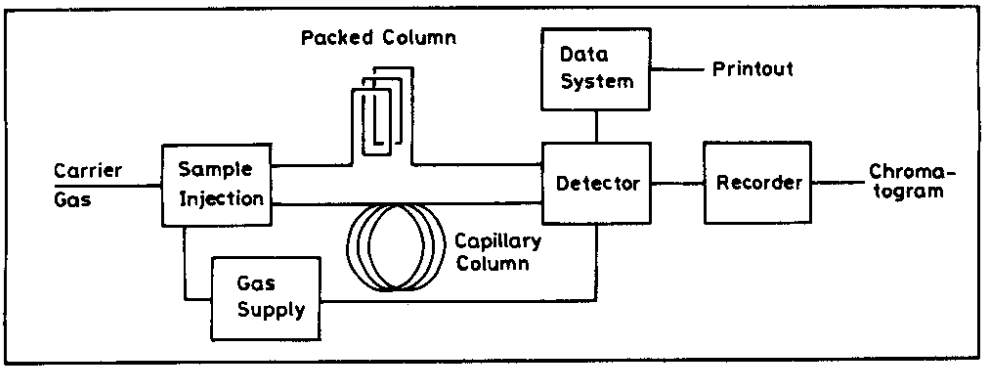

Fig. 1: Different Components of a Gas Chromatograph.

Column (Stationary Phase) : The stationary phase is packed in the steel or glass columns. The columns used in GC are of two types i.e. packed columns and capillary columns. The capillary columns are more sensitive and effective than the packed columns. The separation efficiency of the columns depends upon the number of their theoretical plates. The glass columns are essential to minimize compound decomposition on the column.

Sample Injection : It is a device in which the sample to be analysed is injected with the help of microsyringe. The temperature of this point is to be kept greater than the boiling point of the pollutants to be analysed.

Detectors : The sensitivity, reproducibility and efficiency of a Gas Chromatoraph depends upon the quality of the detectors. The most commonly used detectors for pesticides analysis are Electron Capture Detector (ECD), Flame Ionisation Detector (FID), Thermal Conductivity Detector (TCD) and Mass Spectroscopy (MS) detector. However, other detectors such as NPD, AFID, EIMS, EPN, AED, AAS, FT-IR, FPD, ECD/NPD, NPD/ITD etc. have also been used for the detection of other classes of pesticides.

Electron Capture Detector (ECD) is a pollutant specific detector which is sensitive to a certain types of pollutants which contain atoms or groups of high electron affinity such as halogens, carbonyls, nitrogenous etc. The detector contain the source of pollutants ionisation (radioisotopes). The carrier gas is ionised by the detector and a constant ion current is amplified and displayed as the base line. When a compound having an affinity for the electrons enter the detector, negative ions are formed resulting in a decrease in the ion current which can be measured by having the familiar peaks by the recorder.

Thermal Conductivity Detector (TCD) is widely used for its high reliability, simplicity and ease of operation. The TCD measures the difference in thermal conductivity between the carrier gas flowing through a reference cell and a carrier gas/sample component mixture flowing through a measuring cell.

Flame Ionisation Detector (FID) yields an excellent sensitivity and a wide linear dynamic range. It operate on the principle that the electrical conductivity of gas is directly proportional to the concentration of charged particle within the gas. The sample component in the effluent gas is mixed with hydrogen and burned in air. The extent of ionisation depends on the nature of the pollutants and temperature of the flame. A collected electrode with a D.C. potential applied is placed across the flame and measure the conductivity of the flame which is recorded by the recorder.

In Mass Spectroscopy (MS) detector the analysed pollutant is bombarded with a beam of very high energy electrons which resulted into the positively charged ions. These ions further decomposed into various small cations due to their high energy contents. These ions are allowed to pass through the magnetic field and the ions of low mass are deflected more than the heavier ions which are recorded by the recording device.

Data System : The data system is a computerised device. The signal from the detector is sent to the data system where it is converted in the form of peak. The data system also display various parameters such as retention times, number of peaks, area of peaks, etc. These information are stored in the data system. The size of peaks may be changed if required.

Recorder : It is a simple printer and always remain attached with data system. The chromatogram from the data system can be printed with the help of the recorder.

The specific conditions for the most commonly used pesticides in India are listed in Table 4.

Table 4. Retention Times (minutes) and Other Chromatographic Conditions of Most Commonly Used Pesticides in India

Pesticides	Liquid Phase: 1.5% OV-17 +1.95% QF-1 Column Temp.: 200°C Mobile Phase: Argon/Methane (60 mL/min)	Liquid Phase: 5% OV-210 Column Temp.: 180°C Mobile Phase: Argon/Methane (70 mL/min)
α-BHC	0.54	0.64
PCNB	0.68	0.85
Lindane	0.69	0.81
Dichloran	0.77	1.29
Heptachlor	0.82	0.87
Aldrin	1.00	1.00
Heptachlor Epoxide	1.54	1.93
α-Endosulfan	1.95	2.48
β-Endosulfan	3.59	4.59
p.p'-DDE	2.23	2.10
Dieldrin	2.40	3.00
Captan	2.59	4.09
Endrin	2.93	3.56
o.p'-DDT	3.16	2.70
p.p'-DDT	3.48	3.75
p.p'-DDD	3.48	3.75
Mirex	6.10	3.78
Methoxychlor	7.60	6.50

(Glass column: 180 cm x 4 mm, Gas Chrom Q, 100/200 mesh)

3.0 SAMPLING TECHNIQUES, APPARATUS AND CHEMICALS

The water samples should be collected in pesticide grade solvent prewashed, glass containers closed with teflon lined caps or heavy aluminium foil. Teflon is normally preferred, however, if aluminium foil is used, it should be precleaned with analytical grade acetone followed by pesticide grade ethyl acetate and hexane. At no time sample should be in contact with plastic materials, nor any solvent should come in contact with plastic during analysis, since pesticizing agent may leach out and subsequently interfere with the analysis. Sample storage should be for minimal period at 3-5°C. Immediate extraction is recommended where possible, at which point the organic phase can be stored at -15°C for two or three weeks.. Different sampling devices have been used for the sampling of water, viz., air sampling pump (21), a ship board sampling apparatus (22), low speed submersible, peristaltic pump, bladder pumps (23). Besides, an apparatus to collect 28 L of water was designed to minimise the sample contamination and used for water sampling for pesticides analysis (24).

All glassware used for sample collection and pesticide residue analysis should be cleaned throughly and should be rinsed with the pesticide grade solvents. Glassware should be stored to prevent accumulation of dust or other contaminants. It is a good practice to segregate glassware used for higher level analysis (such as for coloured water, agricultural runoff and industrial effluents) from that used for lower level analysis (such as for surface water). The important instruments/glasswares used for the analysis of pesticides include rotary evaporation or Kuderna-Danish assembly, Separatory funnels, packed and capillary columns, gas chromatograph with desired detectors etc.

Solvents, reagents and other materials for pesticide analysis should be free from interferences under the condition of the analysis. Specific selection of reagents and distillation

of solvents in an all-glass system may be required. Pesticide grade solvents usually do not require redistillation, however, a blank should be run before use. The most commonly used solvents and chemicals include acetenitrile, acetone, n-hexane, iso-octane, benzene, methyl chloride, touluene, silica gel, sodium sulphate anhydrous, high purity gases (nitrogen, hydrogen, zero air, methane etc.).

Hexane is not a good solvent for the preparation of stock solutions of pesticides as it is easily evaporated upon storage even at 4°C and due to also complete insolubility of organochlorine pesticides in it. Certain batches of hexane also contain unsaturated hydrocarbons which can give the peak in Chromatograph. It has been observed in some of laboratories that the exposure of hexane to light may cause this problem. Isoctane pure or a mixture of isoctane and toulene are the best solvent for the preparation of stock solutions.

Stock solutions of pesticides can be kept at 4°C in the dark for a year or longer. However, they should be checked once in a while (every 2 to 4 months). The major problem in longer-term storage of these stock solutions is evaporation and not degradation. A 1000 ppm stock solution is normally recommended and can be prepared by dissolving 100 mg of pure analytical standard pestticide in the appropriate solvent and diluting to 100 mL in "low-actinic" volumetric flasks.

4.0 EXTRACTION, CLEANUP AND CONCENTRATION

The extraction of pesticides is normally being carried out by simple liquid-liquid extraction (LLE), in case of water, and solid-liquid extraction (SLE), in case of sediments and soils, techniques using organic solvents. Hexane is not effective as an extraction solvent for all the pesticides. In certain cases, when there are suspended solids in the water samples, hexane gives less satisfactory results even when it gives good results (80% recovery) from spiked distilled water. Therefore, hexane should not be used as a broad-spectrumn solvent for extraction of organochlorinated pesticides from water. Methylene chloride is a very effective solvent for multi-residue extraction of pesticides from water including those samples with considerable turbidity and organic content. It has low water solubility and is not too polar to extract excessive co-extractives as compared with more polar solvents, such as diethyl ether and ethyl acetate. Howewver, the most commonly used organic solvents include dichloromethane (25), hexane (26), acetone (27), acetic acid (28), benzene (29), toluene (30), methanol (31), acetonitrile (28), petroleum ether (32), ethyl ether (33), iso-octane (34) and pentane (33). These solvents are used as single, binary or ternary mixtures for pesticides extraction.

The general methods for extraction of organics from water samples involve manual or mechanical shaking, stirring with magnetic stirrers, continuous liquid-liquid extraction and homogenization method (35) using a high-efficiency dispenser which essentially contains high speed blades with a high frequency ultrasonic probe coupled to it, or adsorption-desorption columns such as the use of polyurethane plugs and XAD-type resins. In brief, the mechanical shaking method is efficient provided the shaking is sufficiently, vigorous and a suitable solvent is used. Different models of shaker can provide different results particularly with turbid waters. Thorough testing with one's own water samples is needed. The homogenization method can cause severe emulsion for certain samples (for example, some

industrial effluents). Under this condition, emulsion is difficult to break up even by high speed centrifugation. The analyst should carefully consider his/her own situation, e.g. water types and choice of solvent, before contemplating using this approach.

The LLE technique is a tedious, time consuming , and consumes large volume of costly solvents. The handling of solvents is hazardous from the health point of view. Besides, emulsion formation in LLE is another problem of pesticides extraction of more polar pesticides, viz., phenoxy acids and their metabolites (18). On the other hand solid phase extraction (SPE) technique is free from these drawbacks and is very fast, sensitive with the recovery of pesticides ranging from 90-95% (36,). The use of SPE offers the advantages of convience, cost saving and minimal consumption of solvents (37). Various columns, disks and cartridges have been used for extraction purposes. The important ones are SS-401, XAD-2 (37), PL RD-S (39), HDG-C_{18} (38), RDS-18 (40), C_{18} cartridge (41) C_{18} silica bonded cartridge (42), SEP-Pak C_{18} (43), microextraction fibre (44), ODS Impregnated polymers (45), C_8 disk (40), and C_{18} Empore disk (45-46).

SPE involved the use of non polar C_8 and C_{18} phases in the form of cartridges and disks. The disks are preffered to the cartridges, as the disks have high cross sectional area that provides several advantages which are not possible in cartridges. With the disk, the decreased back pressure allows greater flow rate while their wide bed decreases the chance of plugging. The embedding of the stationary phase into a disk prevent channeling and improves mass transfer (47). Off line SPE or on line SPE or solid phase micro extraction on various types of silica bonded, polymeric or carbon type phase progressed on LLE for LC and GC methods (48). Extraction efficiency in SPE has been optimized and increased by varying various parameters such as pH, ionic strength of the sample, elution solvents, content of organic modifier in the sample, elution gravity, etc. Progressiveness of the technique has been established by using polymer membrane containing the enmeshed sorbent particle in a web of polymer

micro fibrils called membrane extraction disks. Wells et al. (49) has developed a micro processed optimization of SPE method for the extraction of various pesticides. Barcelo et al. (50) has described the various aspects of SPE using C_{18} and styrene divinyl benzene Empore extraction disk with the influence of water type and matrix at low level. The isolation and preconcentration of most of the non polar or semi polar pesticides was achieved by C_{18} or C_8 bonded silica disks or cartridges from water samples, e.g., phenoxy acid herbicides (51), sulfonyl urea (52), phenyl urea (53,54), organophosphorus (55), triazines (56), hydroxy triazines (57), anilines (58), dinitroaniline (59), organochlorines (48), and fungicides (60).

The extraction of the polar pesticides is very difficult by the non polar (C_{18} and C_8) stationary phases and, therefore, polar pesticides are extracted successfully on graphatized carbon black (GCB). The porous character of GCB makes it faster in extraction with out any pH adjustment of the environmental water samples. Cyanuric acid, a well known degradation product of triazine is successfully extracted by GCB (61,62). However, De Kok et al. (63) extracted N-methyl carbamates and their degradation products by C_{18}/OH silica sorbents. Moreover, a number of workers recommended styrene divinyl benzene copolymer PLRP-S as the universal SPE system for the extraction of pesticides of even moderately polar in nature (64-67).

Ion exchangers have also been used for the extraction of semi or polar pesticides (68-69) but their range of utility is low due to low capacity (capacity decreases by the ions in water samples) and incapability for the extraction of the pesticides having similar acidic-basic moieties which determine the pH of the sample. However, ion exchangers have been successfully used for the selective extraction of interfering compounds, e.g., substituted anilines and their degradation products (71,72).

During recent years, supercritical fluid extraction (SFE) has received a wide spread attention for the extraction of

pesticides from environmental samples. However, a very few reports are available (18,73-76) on the extraction of pesticides by SFE from water sample directly. This is due to high cost, incapability of simultaneous and parallel extraction (77) and good solubility of water in supercritical carbon dioxide which make it unfit for routine applications (67). Another problem in SFE extraction is to optimize the experimental conditions by optimizing the temperature, pressure, amount and type of modifier, extraction time and cell volume. The coupling of SFE with SPE has resulted in the fast and sensitive extraction of pesticides with good recoveries. Alzaga et al. (78) compared SFE and LLE for some of the pesticides.

Clean-up procedures may not be necessary for a relatively clean sample matrix. The clean up procedures are recommended for the analysis of various industrial and municipal effluents. If clean-up is required, the recovery of each compound of interest for the clean up procedure should not less than 85% (79). Clean up and purification of the extracted pesticides is carried out by column chromatography, gel permeation chromatography, sweep codistillation (80), cartridges and disks. The columns used for this purpose are silica (81,82), XAD-2 (83), alumina (84), florisil (85), while the cartridges and disks used are C_8 (86), C_{18} (87), carbon black (74), etc. The use of membrane disks for clean up is increasing significantly in the past few years (88). A comparative study for the preconcentration of pesticides has been discussed (89). SPE membrane disks have also been used in conjunction with SFE in the preconcentration of pesticides (89). On line SPE was coupled to membrane disk for the concentration of pesticides in river water and ground water (90). Using a SFE/SPE methodology > 90% recoveries of pesticides have been reported (91).

Among various forms of chromatography, column chromatography has been used extensively with different types of adsorbents, e.g., florisil column was used for organochlorine, PCBs, PCNBs, strobane, trifluralin (79,85), organophosphorus (92) and silica gel column (10%) deactivated was used for the clean up of nitrogen-phosphorous pesticides (93). The clean up of

organophosphorus pesticides in water samples was achieved by C_{18} Empore disk, disposal C_{18} cartridges and C_{18} Empore disk plus florisil (85). Although different non polar cartridges and disks have been used for SPE technique but reports are available on the clean up of the pesticides using two SPE cartridges (C_{18} and SCX) installed vertically in series for efficient extraction and clean up of atrizent and its degradation products (36).

For the determination of polar pesticides, HPLC appears to be the most appropriate technique. In the recent years numerous methods for the determination of polar pesticides have been published (94,95). In most of the methods, clean up and concentrations are achieved by SPE or column switching technique (96). Organochlorine and pyrethroid pesticides have been purified using HPLC system in water samples (65). Since the introduction of size exclusion chromatography (SEC) for the clean up of environmental samples it could not achieved a reputation as an universal and most applicable analytical technique. This is because of the insufficient advancement of the technique and, therefore, further advancement is required in the technique such as liquid chromatographic pump and the detectors. Besides, SEC columns are not currently available as disposal cartridges. Moreover, SEC is not destructive, highly reproducible and can isolate a variety of compounds in the same fraction (97).

The use of gel permeation chromatography (GPC) in clean up process is limited as it is not capable to clean up the pesticides or any other contaminants of the similar sizes. However, a very few reports (80,85) are available for the clean up of pesticides by GPC. A Bio-Beads S-X3 column was used for the clean up of organo phosphorous pesticides (85). High performance GPC clean up of hexaflumuron residue in soil samples has also been reported (98). A comparison of GPC, sweep codistillation and florisil column chromatography for organochlorine has been discussed (46). Further, a comparison of various types of GPC columns for the clean up of twelve selected pesticides from soil samples has been reported (97).

Extraction Procedure : Extraction is normally carried out by adding 50 mL of suitable solvent to 1 L of water sample in a separatory funnel for 30 minutes. The procedure may be repeated three times. Mix all the three organic phases into a flask. Place a layer of at least 5 cm of anhydrous sodium sulfate in a 125 mL sintered glass funnel and set it up so that the funnel will drain into a 500 mL round-bottom flask. Arrange for vacuum filtration. Drain the organic layer in the 500 mL separatory funnel into the filtration column. The extracted organic solvent is concentrated by a Kuderna denish apparatus on a gentle heating on rotaroevaporator water bath. Alternatively, evaporate the combined sample extracts under vacuum by a rotary evaporator at 30-35°C to 5-6 mL. Transfer the concentrate with 4x2 mL rinsings to a 15 mL graduated glass tube with a conical bottom. Finish the evaporation to 3 mL under a gentle stream of nitrogen at 50 to 60°C (water bath) at atmospheric pressure. Some losses of the low level pesticides will occur if evaporation is carried out above 40°C in a rotary evaporator or below 40°C using the nitrogen method.

A known amounts of pesticides is added (such as 10 to 50 μ litre of the intermediate mixed standard solutions) to a 1 litre water sample and carry through the same procedure as the samples. If a larger amount of the mixed standard solution is to be used, make sure the standard solution is prepared in suitable solvents such as acetone, ethyl acetate or methanol. In any event, do not use more than 200 litres of the mixed standard solution in order to reduce the effect of introducing another variable. Call the peak height from the standard "a" and the peak height from the sample to which pesticide was added "b", whereupon the extraction, efficiency, E, equals b/a. Periodically determine extraction efficiency and a control blank to test the procedure. Also analyse one set of duplicates with each series of samples as a quality control check.

5.0 DETECTION

The experimental conditions used for the detection of pesticides by various workers have been summarised in Table 5. This include name and source of pesticides, columns, mobile phases and detectors used in Gas Chromatography. A basic gas chromatograph is required that has oven temperature control of ± 0.5°C and separate injection and detector temperature controls. The instrument must be capable of accommodating glass-lined injection parts, columns etc. Suitable carrier gas and flow controls are essential. Zero leak is necessary for electron capture detectors and there must be zero oxygen in the carrier.

A suitable detection system of high sensitivity and selectivity is required to achieve the best results. Generally, the electron capture detector is supposed to be the best sensitive detector for pesticides determination. However, other detectors such as NPD (31), FID (74), AFID (99), MS (19,25), EIMS (33), EPN (95), hall electrolytic conductivity detector (96), AED (102), AAS (103), FT-IR(104), FPD(105), ECD/NPD (30,31), NPD/ITD (25,106) have also been used for the detection of other classes of pesticides. Either tritium, ^{3}H or nickel, ^{63}Ni, is satisfactory. The nickel detector affords the advantage of high operating temperature. If available, the linearized electron capture system is highly recommended because of its extended linear and dynamic range capabilities. Readout of detector response requires a 25 cm (10 inch) strip chart recorder, generally 1 mv full scale, with variable chart speed. Mechanical integrators are available to assist in the evaluation of response versus concentration. Different electronic integrators are also commercially available. The detection limit of these detectors ranged from ppm level to ppb level (31,33,38,99-107). The detection of NPD was found to be 0.1 to 0.02 µg/L while ECD is 20 to 100 times more sensitive (30,31,108). Detection with reductive electrolytic conductivity detector has been achieved upto 30 ng/L (82).

During the last few years various reports have been published on the separation and identification of pesticides by gas chromatography. Literature indicates that the analysis of pesticides has been carried out using non polar or semi polar stationary phases (Table 5) with He, H_2, N_2, Ar and CH_4 gases as the mobile phases (109). Gas chromatography with ECD, NPD, FPD detectors have frequently been used for volatile and semivolatile pesticides (105,110). Pre-derivatisation is also required for some of the pesticides due to the thermal instability of phenyl urea pesticides (111,112), high polar nature and low volatility of phenoxy acid herbicides (113) and hydrolytic instability of N-methyl carbamate pesticides (114). Post column derivatisation of pesticides has also been reported due to the use of highly sensitive fluorescence detector (57).

Calibration : The gas chromatograph and detector system should be adjusted as per the operating conditions of the equipment with desired column. Column selection is a matter of choice for the analyst and depends on the pesticides being analyzed. Examples of separations obtained for different columns under different operation conditions have been given in Table 5.

The elution position of individual known compound is determined for a given system by injection of pure standards in the solvent of choice and their elution times recorded. Absolute time or time relative to another known compound may be used. For each compound of interest, a series of standards can be injected under closely controlled conditions and the peak height or peak area determined is plotted against concentration of pesticide in the injected sample (at which point injection volume is to be kept constant) or as total mass (ng or μg) of pesticide injected which allows some variability in injection volume. Calibration curves will allow assessment of the linear range and the dynamic range of the detector for each compound.

Analysis : Appropriate volume of the sample is injected into the gas chromatograph and examined for peaks that correspond in retention time to the standards. Peak height or peak area is then used to assess the individual compound concentrations in the

sample. Overlapping peaks, shoulders and other anomalies complicate qualitative and quantitative analysis and may require additional cleanup procedures and/or alternative columns for separation.

Confirmation of Identity and Quantity : The identity of a pesticide residue is based on comparing its retention time with that of an external standard. It is preferable to use an internal standard. Since there are other pesticides, artifacts and sample co-extractives that could give the same or similar retention time as the pesticide in question, confirmation of its identity is necessary. Dual, or better yet multiple, GC column technique is the most popular and practical. If the retention time of the pesticide in question matches that of the standard using two or more GC columns of different polarity, its identity is reasonably ascertained. It may be pointed out that mass spectrometry has been often advocated as the tcol for confirmation of identity. Furthermore, in the presence of co-extractives in some water sample extracts, and at the sub-ppb levels, mass spectrometry can lack the required sensitivity in certain cases. Its sensitivity for real world samples and for pure standards can be quite different. Sometimes a peak in a sample can be comprised of two or more different compounds. This situation will lead to over estimation of the compound in question. A common practice is to re-analyse the sample in another GC column of different polarity. If the quantity of a pesticide analysed in one GC column matches that analysed in another GC column of different polarity, the quantity of this pesticide in a sample is fairly ascertained.

Calculation : The concentration of each compounds of interest is detrmined by the comparison of peak area or height of the samples with those of standards. This can be done by using the following equation:

$$X_{sam} = \frac{H_{sam}}{H_{std}} \times \frac{V_{inj_{std}}}{V_{inj_{std}}} \times X_{std} \times \frac{V_{ext}}{V_{sam}}$$

where,

X_{sam} = concentration of organic compound in original water sample (μg/L)

H_{sam} = peak height (or area) of sample

H_{std} = peak height (or area) of standard

$V_{inj\ std}$ = volume of standard injected (μL)

$V_{inj\ san}$ = volume of sample injected (μL)

X_{std} = concentration of organic compound in standard solution (pg/μL)

V_{ext} = final volume of sample extract (mL) and

V_{sam} = volume of original water sample extracted (mL).

6.0 CONCLUSION

The identification and quantification of pesticuides in different water bodies is very important part of water ananlysis because of the carcinogenic nature of pesticides. Wastewater should be monitored for pesticides prior to its discharge in a river or on land. The gas chromatographic technique is supposed to be the instrumental technique of choice for pesticides analysis, as most of the pesticides are volatile at the working temperature of gas chromatography. However, a prederivatization of a very few pesticides may be required prior to their GC determination. The choice of the separation and identification of pesticides depends on the polarity, ionic character and stability of the pesticides. Classical liquid-liquid extraction procedure has been replaced by the solid phase extraction method due to various advantages highlighted in this report. During the last few years, a number of publications on pesticide residue analysis increased significantly and important advances were made in the development of multiclass, multiresidue; single-class, multiresidue; and single residue methods for a wide variety of sample types. Most pesticides analysis have been reported using multi residue method involving solvent extraction of the analytes from the sample matrix and quantitative determination by GC.

Table 5: Summary of Experimental Conditions for the Determination of Pesticides by Gas Chromatography

Sl. No.	Pesticides	Source	Stationary Phases	Mobile Phases	Det.	Ref.
	Organochlorine Pesticides					
1.	α-BHC, β-BHC, p,p'-DDE, p,p'-DDT, o,p'-DDD,lindane, heptachlor, aldrin, endo. I & metoxichlor	Artificial polluted water	Supelcoport with 1.5% SP (3 m x 2mm)	N_2	ECD	30
2.	α-HCH, β-HCH, γ-HCH & δ-HCH	Ground water	Fused silica DB-17 (30m x 0.32mm)	He	ECD	34
3.	α-BHC, β-BHC, γ-BHC, o,p'-DDE, p,p'-DDT, heptachlor, heptachlor-epoxide,endo.I, endo. II,endrin, aldrin & dieldrin	River water	GP 1.5% SP-2250-1.95% SP-2401 on Supelcoport	N_2	ECD	26
4.	α-HCH,β-HCH,γ-HCH,4,4'-DDT, 4,4'-DDE, 4,4'-DDD & aldrin	Rain Water	DB-Fused Silica	-	MS	115
5.	α-HCH,β-CH,γ-HCH,4,4'-DDE,2,4'-DDD, 4,4'-DDD,2,4'-DDT,4,4'-DDT,dichl-orvos,heptachlor, heptaepoxide, & dieldrin	Water	CP SIL-5	-	MS	116
6.	o,p'-DDE,p,p'-DDE,o,p'-DDT,p,p'-DDT, trifluralin, lindane, alachlor, captan, folpet, oxyluorfen, bromopropylate,dizofol,tetrad-ifen & deltamethrin	Ground water	Ultra capillary (25m x 0.32mm) with 5%phenyl methyl	He	-	41

7.	α-BHC, β-BHC, δ-BHC, p,p'-DDD, p,p'-DDE, p,p'-DDT, aldrin, chlordane, heptachlor & endrin	Water	DB-1, 4m long & OV-17, 30m long fused silica	He	ECD	118
8.	α-BHC, β-BHC, δ-BHC, γ-BHC, 4,4'-DDE, 4,4'-DDD, 4,4'-DDT, aldrin, captan, carbophenothion, chlordane, dichloran, dicofol, dieldrin, endo.I, endo.II, endosulphate, endrin, endrin aldehyde, heptachlor, isodrin, methoxychlor & mirex.	Industrial waste water	Supelcoport with 1.5%SP 2250/1.95% SP 2401(1.8m x4mm) or Supelcoport with 3% OV (1.8m x 4mm)	Ar & CH_4	ECD	79
9.	α-HCH, β-HCH, δ-HCH, γ-HCH, p,p'-DDE, p,p'-DDE, o,p'-DDT, p,p'-DDT, quintozene, vinchlozolin, heptachlor, aldrin, β-hep-, α-heptachlor, epoxide, captan, α-endo., dieldrin & β-endo.	Surface water	DB-5 fused silica (27m x 0.32mm)	He	ECD	128
10.	α-HCH, β-HCH, δ-HCH, 4,4'-DDE, 4,4'-DDT, α-endo., β-endo., aldrin, dieldrin, lindane, heptachlor, epoxyheptachlor & endo.sulphate	Water	HP-1 methyl silicone	He	MS	48
11.	p,p'-DDT, α-BHC, β-BHC, γ-BHC, δ-BHC, chloridazon, methoxychlor & trifluralin,	River water	DB-1701	He	MS	93
12.	α-HCH, β-HCH, δ-HCH & γ-HCH	Rain water	GasChrom Q with 5% silicone OV-17 or ULBON silica (50m x 0.25mm)	N_2	ECD or MS	19
13.	o,p'-DDE, p,p'-DDE, p,p'-DDD, aldrin, endrin, lindane, trifluralin, heptachlor, chlorfenson, dicofol & tetradifon	Water	OV-101 (30m x 0.53m)	-	-	120

14.	p,p'-DDT, o,p'-DDD, p,p'-DDD & o,p'-DDE	River water	Ultra-1 methyl (50m x 0.2mm)	-	MS	123
15.	Dalapon-sod., dicamba, MCPA, MCPB, 2,4,5-T, TBA & mecoprop	Water	CP Sil 19 CB fused silica	-	MS	125
16.	Butachlor,chlormethoxynil & bifenox	River water	OV-17 CB-FSC (25m x 0.32mm)	-	ECD	83
17.	Chlorinated eight pesticides	-	DB-5 Fused silica (30m x 0.25mm)	He	NPD & ECD	117
18.	Alachlor & metolachlor	Ground water	Fused silica with SE-54	He	ECD & NPD	119
19.	2,4'-D, 2,4'-DB, 2,4,5'-T, MCPB, MCPA, 4-chlorophenoxy acetic acid, chlorifibric acid, dichlorprop, fenoprop, mecoprop, aciflurfen, benazolin acid, chloramben, chlorfenaclopyralid, dicamba, chlorobenzoic acid, flamprop, fluroxypyr,haloxyfop, picloram, triclopyr,chlorflurenol & methyl flamprop-isopropyl as their pentaflurobenzylic derves.	Water	HP-5 capillary (15m x 0.33mm)	He	MS	132
20.	DDT, MCPA-methyl ester, MCPB methyl ester, lindane, alachlor, heptachlor, metolachlor, aldrin & metazachlor,	Drinking water	HP-101 methyl silicon (2m x 0.2mm)	-	MS	133
21.	DDE, DDT, aldrin, dieldrin, lindane, metazachlor & metalochlor	Water	DB- wax capillary (15m x o.32m)	He	NSD	134

22.	Mmetolachlor & trifluralin	Soil	10% DC 200:2% OV 225 (2m x 1.8mm)	N_2	FID	121
23.	Aldrin, dieldrin & lindane	Water	HP-1 fused silica (25m x 0.2mm)	He	MS	155
24.	Lindane & alachlor	River and Ground water	Chromosorb WHP with 1:1 of 10% OV-101 & 15% OV-210	-	ECD & NPD	122
25.	Aldrin, dieldrin & lindane	Lake and River water	Chemosorb WHP with 1:1 of 10% OV-101 & 15% of OV-210	N_2	ECD	43
26.	Alachlor, chloridazon, molinate, trifluralin, propanil & isoproturon.	Soil	Fused silica (15 m x 0.15mm)	H_2	NPD	97
27.	Lindane, dicofol, chlorfenson & tetradifon	Soil	Ultra-2 (30m x 0.25mm)	-	ECD & NPD	124
28.	Metolachlor	Runn off water	DB-5, 5% phenyl-95% methyl (30m x 0.25mm)	He	NPD	142
29.	Alachlor, chlorpyrifos, metochlor, α-endo. & β-endo.	Marsh water	Supelco SPB-5 fused silica (30m x 0.25mm)	He	MS	47
30.	Permethrin & cyfluthrin	Water	DBS-MS fused silica (20m x 0.32mm)	He	MS	126
31.	Alachlor	Well water	SPB-20 (60m x 0.75mm)	He	NPD	127

32.	Alachlor,metalochlor & pendimethalin	Soil	BP-1 fused silica (12m x 0.22mm)	He	ITD	129
33.	Metolachlor	Soil	DBVAX (15m x 0.52mm)	N_2	NPD	31
34.	Alachlor, metolachlor & trifluralin	Clay	DB-5 (30m x 0.3mm)	-	MS	130
35.	Alachlor, chloridazon, chlortoluron, metolachlor, propachlor & trifluralin	Water	DB-5 (30m x 0.32mm)	He	FID & ECD	131
36.	Alachlor & metolachlor	Water & soil	BP-1 fused silica (12m x 0.22m)	He	MS & ECD	25
37.	Captan, captafol & chlorothalonil	River water	DB-5 with 55 phenyl -95% methyl polysiloxane	He	MS & ECD	60
38.	Metazachlor, metalochlor, pretilachlor & propachlor	Water	PTE-5 Supelco (30m x 0.32mm)	-	NPD & FID	135
39.	Alachlor, propachlor, diuron, linuron, captan, MCPA ester & trifluralin	River, lake & sea water	HP-5 fused silica with 5% phenyl-95% methyl polysiloxane (25m x 0.25mm)	He	MS & FID &	152
Nitrogen Containing						
40.	Atrazine, simazine, cyanazine, & desethylatrazine	Rain water	DB-5 fused silica (30m x 0.52mm)	He	NPD	137
41.	Atrazine, prometon, propazine, simazine, prometryn, ametryn, simetryn, terbutryn, & irgarol,	Costal water	OV-1701 fused silica	He	NPD	42

42.	Atrazine and its degradation products	Run off water	DB-5 with 5% phenyl -955 methyl (30m x 0.25mm)	He	NPD	36
43.	Atrazine, cyanazine & simazine	Marsh water	Supelco-SPB-5 fused silica (30m x 0.25mm)	He	MS	47
44.	Atrazine, desmetryn, metamitron, dimethoate, hexazinone & vamidothion	Water	CP-Sil 19CB Chrompak (15m x 0.32m)	N_2	NPD	39
45.	Atrazine, simazine, terbuthylazine, desethylatrazine & desisopropyl-atrazine	Soil	DB-5 Ultra-1 (50m x 0.25mm)	-	ECD & NPD	138
46.	Atrazine & prometryn	Water	DB-17	He	NPD	143
47.	Atrazine & simazine	Water	HP-1 methyl silicone	He	MS	48
48.	Atrazine, cyanazine, diethyltetrazine, deisopropylatrazine & hydroxyatrazine	Soil	DBVAX(15m x 0.52mm)	N_2	NPD	31
49.	Atrazine, simazine, cyanazine, & metribuzin	Clay	DB-5(30m x 0.25mm)	-	MS	130
50.	Atrazine, cyanazine, simazine, amytrin, propazine & sebuthylazine	Water	DB-5(30m x 0.32mm)	He	ECD, NPD.& FID	131
51.	Atrazine, deethylatrazine, & pendimethali	Water & soil	BP-1 fused silica (12m x 0.22m)	He	MS & NPD	25
52.	Atrazine, simazine & cyanazine	Well water	SPB-20(60m x 0.75mm) siloxane	He	NPD	127
53.	Atrazine & simazine	River water	DB-1701	He	MS	93

54.	Atrazine, desisopropylatrazine, desethylatrazine, desethylterbutyline, simazine, propazine & terbutrylin	Water	HP-101 methyl silicone (2m x 0.22mm)	-	MS	133
55.	Atrazine, simazine & terbuthylazin	Water	DB-wax (15m 0.32mm)	He	NSD	134
56.	Atrazine, simazine, propazine, prometon, ametryn, prometryn, & terbutyrin	Ground water	DB-1 (30m x 0.32mm)	He	NPD	139
57.	Atrazine, ametryn, desmetryn, prometryn, propazine, sebuthylazine, simazine, simytrin, terbuthylazine & terbutryn	Water	PTE-5 Supelco (30m x 0.32mm)	-	NPD & FID	135
58.	Atrazine, simazine, propazine, cyanazine & prometryn	River, lake & sea water	HP-5 fused silica with 5% phenyl-95% methyl polysiloxane (25m x 0.25mm)	He	MS & FID	152
59.	Atrazine Prometon, atraton, propazine terbuthylazine, secbumeton, prometryn, terbutryn, simazine, ametryn, simetryn	Industrial & municipal water	Supelcoport coated with 5% carbovax (1.8m x 2mm)	He	MS & T.I.D.	82
60.	Atrazine, cyperazine & Carbaryl,	Water & sediment	Fused silica with 5% phenyl methyl silicone	H_2, N_2 & CH_4	NPD, FPD, ECD & MS	142
61.	Atrazine, propazine, simazine, prometon, prometryn, ametryn, terbutryn & irgarol	Costal water	OV-1701 fused silica (25m x 0.25 mm)	He	NPD	42
62.	Atrazine, simazine, propazine, terbutylazine, trietazine, & sechumeton	Drinking water	DB-1 (15m x 0.32mm)	He	FID, NPD & FPD	140

63.	Simazine, dimethoate	Water	HP-1 (12m x 0.2mm)	He	NPD	110
64.	Bromacil, deet, hexazinone, metribuzin, terbacil, triadimefon, & tricyclazole	Industrial & municipal waste water	Supelcoport with 3% SP 2250 (1.8m x 2mm)	N_2	Therm-ionic	136
65.	Betazone	Water	CP Sil 19 CB fused silica (25m x 0.25mm)	-	MS	125
66.	3-Indolyacetic acid, 3-indolyl butyric acid, 3-indolyl propionic acid, bentazone, bromoxynil, flurazifop, & fluazifop-p-butyl	Water	HP-5(1.5m x 0.32mm)	He	MS	132
Organophosphorous						
67.	Mevinophos, diazinon, fenitrothion, coumaphos & carbofenthion	Drinking water	DB-1 (15m x 0.32mm)	He	FID, NPD & FPD	140
68.	Dimethoate, fonopos, diazinon, formothion, malathion, fenthion, methidathion, phosmate, azinphos-methyl & phosolone	Ground water	Ultra with 5% phenyl-methyl silicone (25m x 0.32mm)	He	-	41
69.	Azinophos methyl, bolstar, chlorpyrifos, chlorpyrifos methyl, coumaphos, demoton, diaznon, dichlorvos, disulfoton, ethoprop, fensulfothion, nerphos, mevinphos, naled, parathion methyl, phorate, ronnel, stirofos, tokuthion, trichloronate & fensulfothion	Industrial & Municipal waste water	Supelcoport with SP-2401 (1.8m x 2mm)	He, N_2	FID	141
70.	Malathion, parathion, fenintrothion, & diazinon	Water	HP-1 methyl silicone	He	MS	48

71.	Malathion, tetrachlorvinphos, molinate, carbofuron & primicarb	Water	DB-17	He	NPD	143
72.	Malathion, parathion, mehtyl-parathion, ethyl parathion, dizinon, terbufos,propanil, carbofuron & monocrotophos	River, lake & sea water	HP-5 fused silica with 55 phenyl-95% methyl polysiloxane 25m x 0.25mm)	He	MS & FID	152
73.	Diazinone, malathion & dichlorvos	Water	HP-1 fused silica (25m x 0.2mm)	He	MS	155
74.	Parathion	River & ground water	Chromosorb WHP with 1:1 of OV-101 & 15% OV-210	-	ECD & NPD	122
75.	Methyl parathion, profenofos, propanil, norflurazon & pendimethalin	Clay	DB-5(30m x 0.25mm)	-	MS	130
76.	Parathion, malathion, pflanzenbe, & handlungsmittle	Water	DB-wax(15m x 0.32mm)	He	NSD	134
77.	Parathion methyl,parathion methyl & phorate	Lake & river water	DB-wax(15m x0.32mm)	N_2	ECD	43
78.	Parathion, parathion methyl, dimethoate, chlorpyrifos, trichlorfen & phorate	River water	Two fused silica culumns; Ultra-2 (25m x 0.32mm) & SGE-1701 (25m x 0.25mm)	He	NPD	154
79.	Dimethoate, fenitrothion, & methidathion	Soil	Ultra-2(30m x 0.25mm)	-	ECD & NPD	124
80.	Phorifos & methidathion	Marsh water	Supelco SPB-5 (30m x 0.25mm)	He	MS	47

81.	Ethylazinophos, methylazinophos, bromophos, chlormephos, coumaphos, diazinon, dichlorvos, dimethoate, fenitrothion, fenthion, isofenphos, malathion, ethylparathion, methyl-parathion, phorate, phosmet, terbufos & triazofos	Water & Sediment	Fused silica with 5% phenyl-methyl silicone	H_2, N_2	NPD, FPD, MS & ECD	142
82.	Azinophosethyl, azinophosmethyl, coumaphos, malathion, demeton, diazinon, dichlorvos, dimethoate, endrinaldehyde, etrimphos, fenitrothion, methamidophos, mevinophos, parathionmethyl, parathionethyl, propetamophos & triazophos	River water	DB-1701	He	MS	93
83.	Fonfos, diazinon, formothion, primicarb, fenitrothion, malathion, fenthion, chlorpyrifos, methidathion, propiconazole, phosmet, azinophos-methyl & phosalone	Water	HP-1 (12m x 0.2mm)	He	NPD	110
84.	Carbofuron, molinate, primicarb, diazinon, prometryn, tetradifon, & thioben	Soil	DB-17	He	NPD & ECD	77
85.	Ethoprophos, dicrotophos, sulfotep, diazinon, cyanophos, etrimophos, parathion-methyl, primiphos-methyl, parathion-ethyl, bromophos-methyl, chlorothion, bromophos-ethyl, jodfenphos & prothiophos,	Water	PTE-5(30m x 0.25mm)	-	NPD	44
86.	IBP, MEP, MPP, EDDP, EPN, α-CVP, β-CVP, diazinon, malathion, isoxathion, methidathion, salithion, phosalone & phosmet	River water & sediment	Ultra-2 with 25% phenyl-methyl silicone (25m x 0.32mm)	He	MS	144

87.	Triethylphosphorothioate, thionazin, tributylphosphate, sulfotep, phorate, dimethioate, disulfoton, methyl-parathion & famphur	Ground water	DB-5(60m x 0.32mm)	He	NPD	139
88.	Trifluoroacetyl or methyl derves. of BPMC, MTMC, PHC, MIPC, & MBC	River water & sediment	Ultra-2-cross linked with 5% phenyl-methyl silicone	He	MS	145
89.	Organophosphorous pesticides and their degradation products e. g. dialkyl phosphorothioate and dialkyl phosphorodithioate	Pond water	Chromosorb W/AW with 20% Tritan X-305 with SE-30+6% OV-210 on GasChrom Q (18m x 2mm)	N_2	MS	153
Dinitroaninlie						
90.	Benfluralin, ethafluralin, isopropalin, profluralin & trifluralin	Industrial & municipal water	GasChrom Q with 1.5% OV-17/1.95% OV-210(1.8m x 2mm)	Ar:CH_4 & N_2	ECD & MS	108
91.	Acifluorfen, bentazon, dacthal, dicamba, dichlorobenzoic acid, dichlorprop, dinoseb, 5-hydroxydicamba, pentachlorophenol, picloram & silvex	Water	DB-5 fused silica (30m x 0.32mm)	He	ECD	147
92.	Cypermethrin, diphenamide, fensulfothion, metribuzin, pebulate, phosmate, terbufos & trifluralin	Marsh water	SPB-5 fused silica (30m x 0.3mm)	He	MS	47
93.	Dikegulac-sod. & endothal-sod.	Water	CP Sil 19CB fused silica (25m 0.25mm)	-	MS	125

Phenoxy acid Herbicides

94.	MCPA, MCPP & 2,4-D	Soil	BP-1 fused silica (12m x 0.22mm)	He	Finnigan ion	149
95.	MCPA, 2,4-D, Silvex, 2,4,5-T & 2,4'-DB	-	DB-5(25m x 0.25mm)	He	MS	150
96.	Thiobencarb residues	Water	DB-17(30m x 0.25m)	He	NPD	151
97.	Alicarb,benazolin,bromofenoxim, carbaryl, metribuzin, terbacil, ethofumesate, fenamiphos, pendimethalin, phenmedipham, terbuthylazine, triadimefon & vinclozolin	Water	DB-5(30m x 0.32mm)	He	FID ECD & NPD	131
98.	Carbendazim, ethirimol, metalaxyl, folpet & vinclozolin	River water	DB-5 with 5% phenyl-95%methyl polysiloxane	He	MS, ECD & NPD	60

Urea Pesticides

99.	Isoproturon, chlorotoluron, linuron & diuron	Water	BP-1 fused silica (25m x 0.22mm)	H_2, N_2	NPD	146
100.	Linuron & monolinuron	River water	DB-1701	He	MS	93
101.	Linuron, monuron & isoproturon.	Soil	Fused silica (15 m x 0.15mm)	H_2	NPD	97
102.	Chlorimuron, cinosulfuron, primisulfuron, thifensulfuron, triasulfuron & tribenuron	Water & soil	HP-5(25m x 0.2mm)	He	ECD & NPD	148
103.	Diuron, carbofuron, linuron, monouron,	Water	DB-5(30m x 0.32mm)	He	FID ECD & NPD	131

Carbamate Pesticides					
104. Benomyl & carbaryl	Water	DB-wax (1.8m x 0.32mm)	He	NSD	134
Pyrithroid Insecticides					
105. Fenpropathrin, c-permethrin, t-permethrin, t-D-cypermethrin, c-A-cypermethrin, c-β-cypermethrin, t-c-cypermethrin & deltamethrin	Surface water	DB-5 fused silica (27m x 0.32mm) & SE-54 fused silica (22m x 0.32mm)	-	ECD	128

List of Abbreviations Used

AAS:	Atomic Absorption Spectrometry
AED:	Atomic Emission Detection
AFID:	Alkali Flame Ionization Detection
BHC:	Hexachloro cyclohexane
BPMC:	3-(sec-butyl)phenyl-N-methyl carbamate
c:	Cis
CVP:	2-Chloro-1-(2,4-dichlorophenyl)venyl diethylphosphate
2,4-D:	2,4-Dichlorophenoxy acetic acid
2,4-DB:	4-(2,4-Dichlorophenoxy) butyric acid
DDD:	Dichloro diphenyl dichloro ethane
DDE:	Dichloro diphenyl ethylene
DDT:	Dichloro diphenyl trichloro ethane
ECD:	Electron capture detection
EDDP:	o-Ethyl-s,s-diphenyl phosphorodithioate
Endo:	Endosulfan
EPN:	o-Ethyl o,p-nitrophenyl phenyl phosphorothioate
FID:	Flame Ionization Detection
FPD:	Flame Photometric Detection
FTIR:	Fourier Transform Infra Red
GC:	Gas Chromatography
GCB:	Graphatized Carbon Black
GPC:	Gel Permeation Chromatography
HPLC:	High Performance Liquid Chromatography
HCH:	Hexa chloro cyclohexane
ITD:	Ion Trape Detection
LC:	Liquid Chromatography
MBC:	Methyl benzimidazol-2-ylcarbamate
MCPA:	(4-Chloro-o-tolyloxy) acetic acid
MCPB:	(4-Chloro-o-tolyloxy) butyric acid
MCPP:	(4-Chloro-o-tolyloxy) propionic acid
MEP:	o,o-Dimethy-o-4-nitro-m-tolyl phosphorothioate
MIPC:	2-Isopropyl phenyl-N-methyl carbamate
MTMC:	3-Methyl phenyl-N-methyl carbamate
MS:	Mass Spectrometry
NPD:	Nitrogen Phosphorous Detection
PCB:	Poly chlorinated biphenyl
PCNB:	Polychlorinated Nitrobiphenyl
PHC:	2-Isopropoxy phenyl-N-methyl carbamate
SEC:	Size Exclusion Chromatography
SFE:	Super Critical Fluid Extraction
SPE:	Solid Phase Extraction
t:	Trans
2,4,5-T:	2,4,5-Trichloro phenoxy acid
TBA:	2,3,6-Trichlorobenzoic acid
TID:	Thermo Ionic Detection
WHO:	World Health Organisation

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