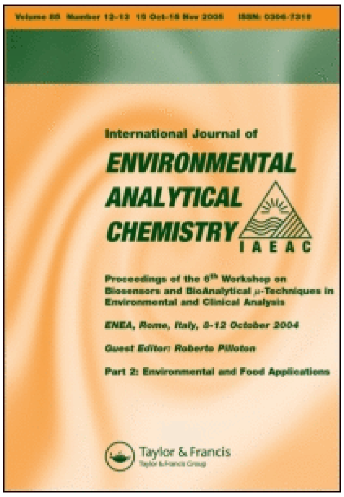

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DETERMINATION OF PESTICIDES IN WATER, SEDIMENTS AND SOILS BY GAS CHROMATOGRAPHY

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The determination of pesticides and their degradation products by gas chromatography has been reviewed and evaluated. The review covers the determination of organochlorine, nitrogen containing, organophosphorus and other pesticides in various water bodies, sediments and soils. The sources 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 summarized in tabular form.

Keywords: Pesticides, gas chromatography, water, sediment, soil

INTRODUCTION

Water is the most important and fundamental requirement for the existence of life and its pollution is the major problem nowadays. 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

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pesticide at 5.0 μg/L. The WHO has classified the pesticides into five groups on the basis of their hazardous nature (3).

Various reports have been published on pesticides analysis in different matrices in last few years. Literature survey indicates 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 (4–14) using various analytical techniques. In this paper an attempt has been made to review the gas chromatography technique for the estimation of pesticides in water, sediments and soils. The review covers sampling procedures, extraction and purification techniques, detectors used, applications and other experimental conditions. The present review covers the literature from 1981 to 1996. The references used in preparing this review are compiled by searching Chemical and Analytical Abstracts of the same period. In addition to this, some important Journals like J. Chromatogr., J. Chromatogr. Sci., Chromatographia, Analyst, Anal. Chem., Fresenius J. Anal. Chem., Wat. Res., Bull. Environ. Contam. Toxicol., Environ. Tech. Letters, and Intern. J. Environ. Anal. Chem., have also been reviewed.

SOURCE OF PESTICIDES POLLUTION

The major sources of pesticide pollution are industries, agricultural, forestry and domestic activities. However, the pesticides pollution through air have also been reported (15). The dust particles in air adsorbed the pesticides (due to pesticides spray in agriculture, forestry and domestic use) and then contaminate water bodies, sediments and soil through rain water (16).

SAMPLING TECHNIQUES

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. Different sampling devices have been used for the sampling of water, viz., air sampling pump (17), a ship board sampling apparatus (18), low speed submersible, peristaltic pump, bladder pumps (19). Besides, an apparatus to collect 28 L of water was designed to minimise the sample contamination and used for water sampling for pesticides analysis (20).

EXTRACTION AND CLEANUP

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. The most commonly used organic solvents include dichloromethane (21), hexane (22), acetone (23), acetic acid (24), benzene (25), toluene (26), methanol (27), acetonitrile (24), petroleum ether (28), ethyl ether (29), iso-octane (30), and pentane (29). These solvents are used as single, binary or ternary mixtures for pesticides extraction. The extraction is done either by simple shaking method or using Soxhlet extraction apparatus (31). The LLE technique is 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 (14).

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% (32). The use of SPE offers the advantages of convience, cost saving and minimal consumption of solvents (33). Various columns, disks and cartridges have been used for extraction purposes. The important ones are SS–401, XAD–2 (34), PL RD-S (35), HDG-C_{18} (34), RDS–18 (36), C_{18} cartridge (37) C_{18} silica bonded cartridge (38), SEP-Pak C_{18} (39), microextraction fibre (40), ODS Impregnated polymers (41), C_8 disk (36), and C_{18} Empore disk (41–42).

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 (43). 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 (44). 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. (45) has developed a micro processed optimization of SPE method for the extraction of various pesticides. Barcelo et al. (46) 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 (47), sulfonyl urea (48), phenyl urea (49, 50), organophosphorus (51), triazines (52), hydroxy triazines (53), anilines (54), dinitroaniline (55), organochlorines (44), and fungicides (56).

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 without any pH adjustment of the environmental water samples. Cyanuric acid, a well known degradation product of triazine is successfully extracted by GCB (57, 58). However, De Kok et al. (59) 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 (60–63).

Ion exchangers have also been used for the extraction of semi or polar pesticides (64–66) 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 (67, 68).

During recent years, supercritical fluid extraction (SFE) has received wide spread attention for the extraction of pesticides from environmental samples. However, a very few reports are available (14, 69–72) on the extraction of pesticides by SFE from water sample directly. This is due to high cost, incapability of simultaneous and parallel extraction (73) and good solubility of water in supercritical carbon dioxide which make it unfit for routine applications (63). 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. (74) 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% (75). Clean up and purification of the extracted pesticides is carried out by column chromatography, gel permeation chromatography, sweep codistillation (76), cartridges and disks. The columns used for this purpose are silica (77, 78), XAD–2 (79), alumina (80), florisil (81), while the cartridges and disks used are C_8 (82), C_{18}(83), carbon black (70), etc. The use of membrane disks for clean up is increasing significantly in the past few years (84). A comparative study for the preconcentration of pesticides has been discussed (85). SPE membrane disks have also been used in conjunction with SFE in the preconcentration of pesticides (85). On line SPE was coupled to membrane disk for the concentration of pesticides in river water and ground water (86). Using a SFE/SPE methodology > 90% recoveries of pesticides have been reported (87).

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 (75, 81), organophosphorus (88) and silica gel column (10%) deactivated was used for the clean up of nitrogen-phosphorous pesticides (89). 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 (81). 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 (32).

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 (90, 91). In most of the methods, clean up and concentrations are achieved by SPE or column switching technique (92). Organochlorine and pyrethroid pesticides have been purified using HPLC system in water samples (61). 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 (93).

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 (76, 81) 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 (81). High performance GPC clean up of hexaflumuron residue in soil samples has also been reported (94). A comparison of GPC, sweep codistillation and florisil column chromatography for organochlorine has been discussed (42). Further, a comparison of various types of GPC columns for the clean up of twelve selected pesticides from soil samples has been reported (93).

DETECTORS AND DETECTION LIMITS

Generally electron capture detectors (ECD) have been used for organochlorine pesticides detection. However, other detectors such as NPD (27), FID (70), AFID (95), MS (15, 21), EIMS (29), EPN (96), hall electrolytic conductivity detector (97), AED (98), AAS (99), FT-IR(100), FPD(101), ECD/NPD (26, 27), NPD/ITD (21,102) have also been used for the detection of other classes of pesticides. The detection limit of these detectors ranged from ppm level to ppb level (27, 29, 34, 95–103). The detection of NPD was found to be 0.1 to 0.02 μg/L while ECD is 20 to 100 times more sensitive (26, 27, 104). Detection with reductive electrolytic conductivity detector has been achieved up to 30 ng/L (78).

During 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 I) with He, H_2, N_2, Ar and CH_4 gases as the mobile phases (105). Gas chromatography with ECD, NPD, FPD detectors have frequently been used for volatile and semivolatile pesticides (101,106). Pre-derivatisation is also required for some of the pesticides due to the thermal instability of phenyl urea pesticides (107, 108), high polar nature and low volatility of phenoxy acid herbicides (109) and hydrolytic instability of N-methyl carbamate pesticides (110). Post column derivatisation of pesticides has also been reported due to the use of highly sensitive fluorescence detector (53).

CONCLUSION

The gas chromatography 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 article. 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.

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

TABLE I 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 × 2 mm)	N_2	ECD	26
2.	α-HCH, β-HCH, γ-HCH & δ-HCH	Ground water	Fused silica DB–17 (30m × 0.32 mm)	He	ECD	30
3.	α-BHC, β-BHC, γ-BHC, o,p'-DDE, p,p'-DDT, heptachlor, heptachlorepoxide, endo.I, endo. II, endrin, aldrin & dieldrin	River water	GP 1.5% SP–2250–1.95% SP–2401 on Supelcoport	N_2	ECD	22
4.	α-HCH, β-HCH, γ-HCH, 4,4'-DDT, 4,4'-DDE, 4,4' -DDD & aldrin	Rain Water	DB-Fused Silica	–	MS	111
5.	α-HCH, β-CH, γ-HCH, 4,4'-DDE, 2,4'-DDD, 4,4'-DDD, 2,4'-DDT, 4,4'-DDT, dichlorvos, heptachlor, heptaepoxide, & dieldrin	Water	CP SIL–5	–	MS	112
6.	o,p'-DDE, p,p'-DDE, o,p'-DDT, p,p'-DDT, trifluralin, lindane, alachlor, captan, folpet, oxyluorfen, bromopropylate, dizofol, tetradifen & deltamethrin	Ground water	Ultra capillary (25m × 0.32 mm) with 5%phenyl methyl	He	-	37
7.	α-BHC, β-BHC, δ-BHC, p,p'-DDD, p,p'-DDE, p,p'-DDT, aldrin, chlordane, heptachlor & endrin	Water	DB–1, 4 m long & OV–17, 30 m long fused silica	He	ECD	114
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 (1.8 m × 4 mm) or Supelcoport with 3% OV (1.8 m × 4 mm)	Ar & CH_4	ECD	75
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 (27 m × 0.32 mm)	He	ECD	124
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	44
11.	p, p'-DDT, α-BHC, β-BHC, γ-BHC, δ-BHC, chloridazon, methoxychlor & trifluralin,	River water	DB–1701	He	MS	89
12.	α-HCH, β-HCH, δ-HCH & γ-HCH	Rain water	GasChrom Q with 5% silicone OV–17 or ULBON silica (50 m × 0.25 mm)	N_2	ECD or MS	15

Sl. No.	*Pesticides*	*Source*	*Stationary Phases*	*Mobile Phases*	*Det.*	*Ref.*
13.	o,p'-DDE, p,p'-DDE, p,p'-DDD, aldrin, endrin, lindane, trifluralin, heptachlor, chlorfenson, dicofol & tetradifon	Water	OV–101 (30 m × 0.53 m)	-	-	116
14.	p,p'-DDT, o,p' -DDD, p,p' -DDD & o,p'-DDE	River water	Ultra–1 methyl (50 m × 0.2 mm)	-	MS	119
15.	Dalapon-sod., dicamba, MCPA, MCPB, 2,4,5-T, TBA & mecoprop	Water	CP Sil 19 CB fused silica	-	MS	121
16.	Butachlor, chlormethoxynil & bifenox	River water	OV–17 CB-FSC (25 m × 0.32 mm)	-	ECD	79
17.	Chlorinated eight pesticides	-	DB–5 Fused silica (30 m × 0.25 mm)	He	NPD & ECD	113
18.	Alachlor & metolachlor	Ground water	Fused silica with SE–54	He	ECD & NPD	115
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 (15 m × 0.33 mm)	He	MS	128
20.	DDT, MCPA-methyl ester, MCPB methyl ester, lindane, alachlor, heptachlor, metolachlor, aldrin & metazachlor,	Drinking water	HP–101 methyl silicon (2 m × 0.2 mm)	-	MS	129
21.	DDE, DDT, aldrin, dieldrin, lindane, metazachlor & metalochlor	Water	DB- wax capillary (15 m × 0.32 m)	He	NSD	130
22.	Metolachlor & trifluralin	Soil	10% DC 200:2% OV 225 (2 m × 1.8 mm)	N_2	FID	117
23.	Aldrin, dieldrin & lindane	Water	HP–1 fused silica (25 m × 0.2 mm)	He	MS	151
24.	Lindane & alachlor	River and Ground water	Chromosorb WHP with 1:1 of 10% OV–101 & 15% OV–210	-	ECD & NPD	118
25.	Aldrin, dieldrin & lindane	Lake and River water	Chemosorb WHP with 1:1 of 10% OV–101 & 15% of OV–210	N_2	ECD	39
26.	Alachlor, chloridazon, molinate, trifluralin, propanil & isoproturon.	Soil	Fused silica (15 m × 0.15 mm)	H_2	NPD	93
27.	Lindane, dicofol, chlorfenson & tetradifon	Soil	Ultra–2 (30 m × 0.25 mm)	-	ECD & NPD	120
28.	Metolachlor	Run off water	DB–5, 5% phenyl–95% methyl (30 m × 0.25 mm)	He	NPD	138

Sl. No.	Pesticides	Source	Stationary Phases	Mobile Phases	Det.	Ref.
29.	Alachlor, chlorpyrifos, metochlor, α-endo. & β-endo.	Marsh water	Supelco SPB–5 fused silica (30 m × 0.25 mm)	He	MS	43
30.	Permethrin & cyfluthrin	Water	DBS-MS fused silica (20 m × 0.32 mm)	He	MS	122
31.	Alachlor	Well water	SPB–20 (60 m × 0.75 mm)	He	NPD	123
32.	Alachlor, metalochlor & pendimeth-alin	Soil	BP–1 fused silica (12 m × 0.22 mm)	He	ITD	125
33.	Metolachlor	Soil	DBVAX (15 m × 0.52 mm)	N_2	NPD	27
34.	Alachlor, metolachlor & trifluralin	Clay	DB–5 (30 m × 0.3 mm)	-	MS	126
35.	Alachlor, chloridazon, chlortoluron, metolachlor, propachlor & triflura-lin	Water	DB–5 (30 m × 0.32 mm)	He	FID & ECD	127
36.	Alachlor & metolachlor	Water & soil	BP–1 fused silica (12 m × 0.22 m)	He	MS & ECD	21
37.	Captan, captafol & chlorothalonil	River water	DB–5 with 55 phe-nyl –95% methyl polysiloxane	He	MS & ECD	56
38.	Metazachlor, metalochlor, pretilachlor & propachlor	Water	PTE–5 Supelco (30 m × 0.32 mm)	-	NPD & FID	131
39.	Alachlor, propachlor, diuron, linuron, captan, MCPA ester & trif-luralin	River, lake & sea water	HP–5 fused silica with 5% phenyl–95% methyl polysiloxane (25 m × 0.25 mm)	He	MS & FID	148
Nitrogen Containing						
40.	Atrazine, simazine, cyanazine, & desethylatrazine	Rain water	DB–5 fused silica (30 m × 0.52 mm)	He	NPD	133
41.	Atrazine, prometon, propazine, simazine, prometryn, ametryn, sim-etryn, terbutryn, & irgarol,	Costal water	OV–1701 fused silica	He	NPD	38
42.	Atrazine and its degradation prod-ucts	Run off water	DB–5 with 5% phe-nyl –955 methyl (30 m × 0.25 mm)	He	NPD	32
43.	Atrazine, cyanazine & simazine	Marsh water	Supelco-SPB–5 fused silica (30 m × 0.25 mm)	He	MS	43
44.	Atrazine, desmetryn, metamitron, dimethoate, hexazinone & vami-dothion	Water	CP-Sil 19CB Chrompak (15 m × 0.32 m)	N_2	NPD	35
45.	Atrazine, simazine, terbuthylazine, desethylatrazine & desisopropylat-razine	Soil	DB–5 Ultra–1 (50 m × 0.25 mm)	-	ECD & NPD	134
46.	Atrazine & prometryn	Water	DB–17	He	NPD	139
47.	Atrazine & simazine	Water	HP–1 methyl silicone	He	MS	44

Sl. No.	Pesticides	Source	Stationary Phases	Mobile Phases	Det.	Ref.
48.	Atrazine, cyanazine, diethyltetrazine, deisopropylatrazine & hydroxyatrazine	Soil	DBVAX (15 m × 0.52 mm)	N_2	NPD	27
49.	Atrazine, simazine, cyanazine, & metribuzin	Clay	DB–5 (30 m × 0.25 mm)	-	MS	126
50.	Atrazine, cyanazine, simazine, amytrin, propazine & sebuthylazine	Water	DB–5 (30 m × 0.32 mm)	He	ECD, NPD & FID	127
51.	Atrazine, deethylatrazine, & pendimethali	Water & soil	BP–1 fused silica (12 m × 0.22 m)	He	MS & NPD	21
52.	Atrazine, simazine & cyanazine	Well water	SPB–20 (60 m × 0.75 mm) siloxane	He	NPD	123
53.	Atrazine & simazine	River water	DB–1701	He	MS	89
54.	Atrazine, desisopropylatrazine, desethylatrazine, desethylterbutyline, simazine, propazine & terbutrylin	Water	HP–101 methyl silicone (2 m × 0.22 mm)	-	MS	129
55.	Atrazine, simazine & terbuthylazin	Water	DB-wax (15 m × 0.32 mm)	He	NSD	130
56.	Atrazine, simazine, propazine, prometon, ametryn, prometryn, & terbutyrin	Ground water	DB–1 (30 m × 0.32 mm)	He	NPD	135
57.	Atrazine, ametryn, desmetryn, prometryn, propazine, sebuthylazine, simazine, simytrin, terbuthylazine & terbutryn	Water	PTE–5 Supelco (30 m × 0.32 mm)	-	NPD & FID	131
58.	Atrazine, simazine, propazine, cyanazine & prometryn	River, lake & sea water	HP–5 fused silica with 5% phenyl–95% methyl polysiloxane (25 m × 0.25 mm)	He	MS & FID	148
59.	Atrazine Prometon, atraton, propazine terbuthylazine, secbumeton, prometryn, terbutryn, simazine, ametryn, simetryn	Industrial & municipal water	Supelcoport coated with 5% carbovax (1.8 m × 2 mm)	He	MS & T.I.D.	78
60.	Atrazine, cyperazine & Carbaryl,	Water & sediment	Fused silica with 5% phenyl methyl silicone	H_2, N_2 & CH_4	NPD, FPD, ECD & MS	138
61.	Atrazine, propazine, simazine, prometon, prometryn, ametryn, terbutryn & irgarol	Costal water	OV–1701 fused silica (25 m × 0.25 mm)	He	NPD	38
62.	Atrazine, simazine, propazine, terbutylazine, trietazine, & sechumeton	Drinking water	DB–1 (15 m × 0.32 mm)	He	FID, NPD & FPD	136
63.	Simazine, dimethoate	Water	HP–1 (12 m × 0.2 mm)	He	NPD	106
64.	Bromacil, deet, hexazinone, metribuzin, terbacil, triadimefon, & tricyclazole	Industrial & municipal waste water	Supelcoport with 3% SP 2250 (1.8 m × 2 mm)	N_2	Therm-ionic	132
65.	Betazone	Water	CP Sil 19 CB fused silica (25 m × 0.25 mm)	-	MS	121

Sl. No.	Pesticides	Source	Stationary Phases	Mobile Phases	Det.	Ref.
66.	3-Indolyacetic acid, 3-indolyl butyric acid, 3-indolyl propionic acid, bentazone, bromoxynil, fluazifop, & fluazifop-p-butyl	Water	HP-5 (1.5 m × 0.32 mm)	He	MS	128
Organophosphorous						
67.	Mevinophos, diazinon, fenitrothion, coumaphos & carbofenthion	Drinking water	DB-1 (15 m × 0.32 mm)	He	FID, NPD & FPD	136
68.	Dimethoate, fonopos, diazinon, formothion, malathion, fenthion, methidathion, phosmate, azinphos-methyl & phosolone	Ground water	Ultra with 5% phenyl-methyl silicone (25 m × 0.32 mm)	He	-	37
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 (1.8 m × 2 mm)	He, N_2	FID	137
70.	Malathion, parathion, fenintrothion, & diazinon	Water	HP-1 methyl silicone	He	MS	44
71.	Malathion, tetrachlorvinphos, molinate, carbofuron & primicarb	Water	DB-17	He	NPD	139
72.	Malathion, parathion, methylparathion, ethyl parathion, dizinon, terbufos, propanil, carbofuron & monocrotophos	River, lake & sea water	HP-5 fused silica with 55 phenyl-95% methyl polysiloxane (25 m × 0.25 mm)	He	MS & FID	148
73.	Diazinone, malathion & dichlorvos	Water	HP-1 fused silica (25 m × 0.2 mm)	He	MS	151
74.	Parathion	River & ground water	Chromosorb WHP with 1:1 of OV-101 & 15% OV-210	-	ECD & NPD	118
75.	Methyl parathion, profenofos, propanil, norflurazon & pendimethalin	Clay	DB-5 (30 m × 0.25 mm)	-	MS	126
76.	Parathion, malathion, pflanzenbe, & handlungsmittle	Water	DB-wax (15 m × 0.32 mm)	He	NSD	130
77.	Parathion methyl, parathion methyl & phorate	Lake & river water	DB-wax (15 m × 0.32 mm)	N_2	ECD	39
78.	Parathion, parathion methyl, dimethoate, chlorpyrifos, trichlorfen & phorate	River water	Two fused silica columns; Ultra-2 (25 m × 0.32 mm) & SGE-1701 (25 m × 0.25 mm)	He	NPD	150
79.	Dimethoate, fenitrothion, & methidathion	Soil	Ultra-2 (30 m × 0.25 mm)	-	ECD & NPD	120
80.	Phorifos & methidathion	Marsh water	Supelco SPB-5 (30 m × 0.25 mm)	He	MS	43

Sl. No.	Pesticides	Source	Stationary Phases	Mobile Phases	Det.	Ref.
81.	Ethylazinophos, methylazinophos, bromophos, chlormephos, coumaphos, diazinon, dichlorvos, dimethoate, fenitrothion, fenthion, isofenphos, malathion, ethylparathion, methylparathion, phorate, phosmet, terbufos & triazofos	Water & Sediment	Fused silica with 5% phenyl-methyl silicone	H_2, N_2.	NPD, FPD, MS & ECD	138
82.	Azinophosethyl, azinophosmethyl, coumaphos, malathion, demeton, diazinon, dichlorvos, dimethoate, endrinaldehyde, etrimphos, fenitrothion, methamidophos, mevinophos, parathionmethyl, parathionethyl, propetamophos & triazophos	River water	DB–1701	He	MS	89
83.	Fonfos, diazinon, formothion, primicarb, fenitrothion, malathion, fenthion, chlorpyrifos, methidathion, propiconazole, phosmet, azinophosmethyl & phosalone	Water	HP–1 (12 m × 0.2 mm)	He	NPD	106
84.	Carbofuron, molinate, primicarb, diazinon, prometryn, tetradifon, & thioben	Soil	DB–17	He	NPD & ECD	73
85.	Ethoprophos, dicrotophos, sulfotep, diazinon, cyanophos, etrimophos, parathion-methyl, primiphos-methyl, parathion-ethyl, bromophos-methyl, chlorothion, bromophos-ethyl, jodfenphos & prothiophos,	Water	PTE–5 (30 m × 0.25 mm)	-	NPD	40
86.	IBP, MEP, MPP, DDP, EPN, α-CVP, β-CVP, diazinon, malathion, isoxathion, methidathion, salithion, phosalone & phosmet	River water & sediment	Ultra–2 with 25% phenyl-methyl silicone (25 m × 0.32 mm)	He	MS	140
87.	Triethylphosphorothioate, thionazin, tributylphosphate, sulfotep, phorate, dimethioate, disulfoton, methylparathion & famphur	Ground water	DB–5 (60 m × 0.32 mm)	He	NPD	135
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	141
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 Gas-Chrom Q (18 m × 2 mm)	N_2	Ms	149
Dinitroaninlie						
90.	Benfluralin, ethafluralin, isopropalin, profluralin & trifluralin	Industrial & municipal water	GasChrom Q with 1.5% OV–17/1.95% OV–210 (1.8 m × 2 mm)	$Ar:CH_4$ & N_2	ECD & MS	104
91.	Acifluorfen, bentazon, dacthal, dicamba, dichlorobenzoic acid, dichlorprop, dinoseb, 5-hydroxy-dicamba, pentachlorophenol, picloram & silvex	Water	DB–5 fused silica (30 m × 0.32 mm)	He	ECD	143

Sl. No.	*Pesticides*	*Source*	*Stationary Phases*	*Mobile Phases*	*Det.*	*Ref.*
92.	Cypermethrin, diphenamide, fensulfothion, metribuzin, pebulate, phosmate, terbufos & trifluralin	Marsh water	SPB–5 fused silica (30 m × 0.3 mm)	He	MS	43
93.	Dikegulac-sod. & endothal-sod.	Water	CP Sil 19CB fused silica (25 m × 0.25 mm)	-	MS	121
Phenoxy acid Herbicides						
94.	MCPA, MCPP & 2,4-D	Soil	BP–1 fused silica (12 m × 0.22 mm)	He	Finnigan ion	145
95.	MCPA, 2,4-D, Silvex, 2,4,5-T & 2,4'-DB	-	DB–5 (25 m × 0.25 mm)	He	MS	146
96.	Thiobencarb residues	Water	DB–17 (30 m × 0.25 m)	He	NPD	147
97.	Alicarb, benazolin, bromofenoxim, carbaryl, metribuzin, terbacil, ethofumesate, fenamiphos, pendimethalin, phenmedipham, terbuthylazine, triadimefon & vinclozolin	Water	DB–5 (30 m × 0.32 mm)	He	FID ECD & NPD	127
98.	Carbendazim, ethirimol, metalaxyl, folpet & vinclozolin	River water	DB–5 with 5% phenyl–95%methyl polysiloxane	He	MS, ECD & NPD	56
Urea Pesticides						
99.	Isoproturon, chlorotoluron, linuron & diuron	Water	BP–1 fused silica (25 m × 0.22 mm)	H_2, N_2	NPD	142
100.	Linuron & monolinuron	River water	DB–1701	He	MS	89
101.	Linuron, monuron & isoproturon	Soil	Fused silica (15 m × 0.15 mm)	H_2	NPD	93
102.	Chlorimuron, cinosulfuron, primisulfuron, thifensulfuron, triasulfuron & tribenuron	Water & soil	HP–5 (25 m × 0.2 mm)	He	ECD & NPD	144
103.	Diuron, carbofuron, linuron, monouron,	Water	DB–5 (30 m × 0.32 mm)	He	FID ECD & NPD	127
Carbamate Pesticides						
104.	Benomyl & carbaryl	Water	DB-wax (1.8 m × 0.32 mm)	He	NSD	130
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 (27 m × 0.32 mm) & SE–54 fused silica (22 m × 0.32 mm)	-	ECD	124

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