"An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat"

A

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OF

Master of Science

IN

AGRICULTURAL CHEMISTRY AND SOIL SCIENCE

BY

PRITI K THAKOR

B.Sc. (Chemistry)

Department of Agricultural Chemistry and Soil Science

B. A. College of Agriculture

Anand Agricultural University

Anand – 388 110, Gujarat (India)

2014

REGISTRATION NO. : 04-1061-2009

PRITI K THAKOR M.Sc. (Agri.) AGRICULTURAL CHEMISTRY AND SOIL SCIENCE 2014

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Name of the Student **Major Advisor**

Priti K. Thakor **Dr. P. G. Shah**

DEPARTMENT OF AGRIL. CHEMISTRY & SOIL SCIENCE
B. A. COLLEGE OF AGRICULTURE
ANAND AGRICULTURAL UNVERSITY
ANAND – 388 110
GUJARAT (INDIA)

ABSTRACT

An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat was conducted at Pesticide Residue Laboratory during 2012-13**.** A laboratory study was carried out at AINP on pesticide residues, and Micronutrient Project, ICAR Unit -9, AAU, Anand. Total 203 samples were collected from Kheda, Anand and Vadodara districts of Middle Gujarat area having intensive cultivation of vegetable and cotton.

Soil samples were collected from the depth (0-15 cm) by auger. For making composite sample, small portions of soil were collected from 15-20 well distributed spots, moving in a zigzag manner from each individual sampling site. Soil bulk was reduced to about 500 g by quartering method and kept it in a clean cloth bag. Prior the ground water sample collection tube well was switched on for 15-20 minutes for purging. Water sample filtered through muslin cloth was collected into a 2.5 Litre amber coloured glass bottle.

The ground water samples analysed for micronutrients revealed presence of Cu (0.002 to 0.024 ppm), Fe (0.044-5.924 ppm), Mn (0.002-0.082 ppm) and Zn (0.03-0.268 ppm). The Cd content in ground water ranged from 0.002-0.032 ppm. Similarly Co content ranged from 0.022-0.168 ppm. The concentration of Cr in the ground water sample ranged from 0.006-0.488 ppm, Ni from 0.016-0.264 ppm and Pb in the ground water samples ranged from 0.006-0.192 ppm. Surface water samples (10) were analysed to know the micronutrient status (Cu, Fe, Mn, and Zn). The Cu content in the surface water samples ranged from Tr to 0.036 ppm, Fe from 0.528-9.576 ppm, Mn from 0.018-0.104 ppm, Zn from 0.018-0.074 ppm, Cd from Tr-0.022 ppm, Co from 0.018-0.102 ppm, Cr from Tr-3.256 ppm and Ni from 0.028-0.144 ppm and Pb from Tr-0.064 ppm. There were differences in EC of ground and surface water samples. The EC of the ground water samples was higher than surface water samples. However, differences in pH among ground water and surface water were marginal. The NO_3-N^- content in the ground water samples ranged from 0.018-1.182 ppm. The phosphorous content of surface water ranged from 0.117-0.737 ppm.

Out of 80 soil samples analysed for pesticide residues only 5.0 % samples showed residues above detectable limit. Of the 22 soil samples collected from vegetable growing area, only 3 were found contaminated with pendimethalin. The micronutrient content in soil samples collected from cotton growing area revealed presence of Cu (0.84-22.90 ppm), Fe (0.74-30.44 ppm), Mn (3.34-113.34 ppm) and Zn (Tr-46.4 ppm). The heavy metal content in these samples showed presence of Cd (Tr-0.26 ppm), Co (0.22-1.28 ppm), Cr (Tr-1.06), Ni (0.06-3.00 ppm) and Pb (Tr-2.88 ppm) were detected. Soil samples collected from vegetable growing area found to contain Cu (1.02 to 15.42 ppm), Fe (3.56-28.80 ppm), Mn (4.44-89.76 ppm) and Zn

(0.6-22.06 ppm) with respect to the heavy metal content in soil samples collected from vegetable growing area Cd (Tr-0.18 ppm), Co (0.22-0.84 ppm), Cr (Tr-1.38 ppm), Ni (0.28-1.40 ppm) and Pb (Tr-1.76 ppm) were detected. The percentage O.C content for soil samples of cotton growing area ranged from 0.03-0.95%. The percentage O.C content of soil samples of vegetable growing area ranged from 0.25-0.97%. The EC and pH of soil samples collected from cotton growing area varied from 0.126-2.260 dS m^{-1} and 7.02-8.67, respectively. The EC and pH of soil samples collected from vegetable growing area ranged from 0.292-2.680 dS m^{-1} and 7.32-8.67, respectively.

Total 21 vegetable samples comprising brinjal (5), tomato (2), capsicum (5) and okra (9) collected from farmer's field were monitored for pesticide residues by Gas Chromatography – Mass Spectrometry techniques. None of the brinjal sample was contaminated with pesticide residues. In okra out 9 samples and in capsicum out of five samples one each contained acephate 0.141 ppm and 0.087 ppm, respectively with respect to the micronutrient status, vegetable samples found to contain Cu (5.5 to 16.0 ppm), Fe (89.5 – 295.5 ppm), Mn (11.0 - 44.0 ppm) and Zn (14.0 -53.5 ppm). Vegetable samples also revealed presence of heavy metals e.g. Cd (Tr-2.0 ppm), Co (Tr- 3.5 ppm), Cr (Tr-10.5), Ni (0.6-8.5 ppm) and Pb (Tr-18.5 ppm).

Total of 29 cotton lint samples were analyzed for the pesticide residues. The samples were contaminated with chlorpyriphos (0.011 and 0.015 ppm) and quinalphos (0.054 ppm) with respect to the micronutrient, lint samples revealed presence of Cu (1.5-4.0 ppm), Fe (46.0-144.5 ppm), Mn (5.0-10.5 ppm) and Zn (2.5-7.5 ppm). Cotton lint samples also revealed presence of Cd (Tr-2.0 ppm), Co (Tr-2.0 ppm), Cr (Tr-9.5 ppm), Ni (1.5-4.5 ppm) and Pb (Tr-1.5 ppm).

Dr. P. G. SHAH
Residues Analyst
ICAR, Unit - 9
Anand Agricultural University
Anand-388 110.

C E R T I F I C A T E

This is to certify that the thesis entitled **"An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat"** submitted by **Thakor Pritiben K., Reg. No. 04-1061-2009** in partial fulfillment of the requirements for the award of the degree of **Master of Science** in **Agri. Chemistry and Soil Science** of the Anand Agricultural University is a record of bonafide research work carried out by her under my personal guidance and supervision. The thesis has not previously formed the basis for award of any degree, diploma or other similar title.

Place: Anand **(P. G. Shah)**
Date : / /2014 **Major Guide**

DECLARATION

This is to declare that the whole of the research work reported here in the thesis entitled **"An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat"** for the partial fulfillment of the requirement for the award of the degree of **MASTER OF SCIENCE** in **Agri. Chemistry and Soil Science** by the undersigned is the result of investigation done by her under the direct guidance and supervision of **Dr. P. G. Shah,** Residue Analyst, AINP on Pesticide Residues, ICAR Unit-9, Anand Agricultural University, Anand and no part of the research work has been submitted for any other degree so far.

Place: Anand **(PRITI K. THAKOR)**
Date : / /2014

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As an amateur investigator, it is a matter of great pride and pleasure to present this thesis, which is the climax of my dedication, devotion and ardor to my field of interest.

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PLACE: ANAND

DATE: - -2014 ***(Pritiben K. Thakor)***

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Abbreviations and Acronyms

%	:	Per cent
&	:	And
/, $^{-1}$	:	Per
µg	:	Microgram
µV	:	Micro Volt
^{0}C	:	Degree Celsius
AAS	:	Atomic Absorption Spectrophotometer
AAU	:	Anand Agricultural University
AINP	:	All India Network Project
Anon.	:	Anonymous
BIS	:	Bureau of Indian Standard
BDL	:	Below Determination Limit
Cd	:	Cadmium
Cm	:	Centimetre
Co	:	Cobalt
CPCB	:	Central Pollution Control Board
Cr	:	Chromium
CRMs	:	Certified Reference Materials
Cu	:	Copper
DCM	:	Dichloro methane / Methylene chloride
dS m^{-1}	:	Desi Siemens per Meter
DTPA	:	Diethylene Triamine Pentaacetic Acid
EC	:	Electrical Conductivity
ECD	:	Electron Capture Detector
et al.	:	*et allii* ; and co-workers

etc.	:	*Et cetera;* and rest, so on
EU	:	European Union
FAO	:	Food and Agriculture Organization
Fe	:	Iron
Fig.	:	Figure
FPD	:	Flame Photometric Detector
g	:	Gram
g/cc	:	Gram per Cubic Centimetre
GLC	:	Gas Liquid Chromatograph
GC MS	:	Gas Liquid Mass Spectrometry
h	:	Hours
ha^{-1}	:	Per hectare
HBs	:	Herbicides
HPLC	:	High Performance Liquid Chromatograph
ICMR	:	Indian Council of Medical Research
i.d.	:	Internal Diameter
i.e.	:	That is
ISI	:	Indian Standard Institution
ICAR	:	India Council of Agricultural Research
ISO	:	International Organization for Standardization
kg	:	Kilogram
μL	:	Micro Litre
L	:	Litre
LOD	:	Limit of Detection
LOQ	:	Limit of Quantification
M	:	Meter

Mg	:	Magnesium
Mn	:	Manganese
mL	:	Milliliter
MRL	:	Maximum Residue Limit
nA	:	Neno Ampere
ND	:	Not Detected
Ni	:	Nickle
No.	:	Number
OC	:	Organic Carbon
OCs	:	Organochlorines
OM	:	Organic Matter
OPs	:	Organophosphates
pA	:	Pico Ampere
Pb	:	Lead
pH	:	Potential of Hydrogen Ions
ppm	:	Parts Per Million
PSA	:	Primary and Secondary Amine
QuEChERS	:	Quick Easy Cheap Effective Rugged and Safe
rpm	:	Revolution Per Minute
RSD	:	Relative Standard Deviation
RSD_{WR}	:	Relative Standard Deviation Within-Laboratory Reproducibility
SD	:	Standard Deviation
SPs	:	Synthetic pyrathroids
USEPA	:	United States Environmental Protection Agency
viz.,	:	Namely
vs.	:	Versus
WHO	:	World Health Organization
Zn	:	Zinc

I. INTRODUCTION

According to 2010 FAO world agriculture statistics, India is the world's largest producer of many fresh fruits and vegetables, milk, major spices, selected fresh meats, selected fibrous crops such as jute, several staples such as millets and castor oil seed. India is the second largest producer of wheat and rice, the world's major food staples. India ranked within the world's five largest producers of over 80% of agricultural produce items, including many cash crops such as coffee and cotton, in 2010. As of 2011, India had a large and diverse agricultural sector, accounting, on average, for about 10 percent of export earnings. India has grown to become among the top three global producers of a broad range of crops, including wheat, rice, pulses, cotton, peanuts, fruits, and vegetables.

The continuous use of agricultural land for growing different types of crops has lead to reduction in fertility of the soil and therefore soil requires additional nutrients and agro-chemical products for the growing of healthy food crops. Moreover agro chemical products such as insecticides, fungicides and herbicides are also used for crop protection from harmful insects and other pests. With the growing population, the demand for food crops is also increasing and for efficiently meeting the demand farmers have to constantly take the help of chemicals products. Agro-chemical products are boon to the farmers, as with the use of these products farmers can increase the agricultural productivity that efficiently help to provide food for the ever growing population and also contribute towards eradicating poverty.

The basic objective of most agricultural development programmes is to increase productivity and thereby increase the total agricultural production. The major inputs which have been promoted as a result of these programmes are high response varieties, fertilizers and plant protection chemicals. The impact of these inputs is called "Green Revolution".

Pesticides continue to be a significant input in modern agriculture and are used for the management of pests that are noxious, destructive or troublesome organisms. Pesticides are chemicals substance used for controlling, preventing, destroying, repelling or mitigating any pest (Oudejans, 1994). Ever since man organized agriculture as a profession, he used pesticides in some form or other e.g. nicotine, rotenone, natural pyrethrins and inorganic sulphur. Recent research and development brought about hundreds of new synthetic chemicals of varying toxicity which do benefit the agriculture but they are also the potential source of environmental pollution and could pose negative consequences for human health.

Judicious use of chemicals has played an important role in improving the lives of millions of people worldwide. In fact they are integrated into every aspect of human life. Agrochemicals are resultant of the interaction of biology, chemistry, and environmental stewardship/ sustainability and have created unique opportunities to meet societal needs for food, feed, fiber, and fuel. Crop productivity and crop protection will remain the most important factors in modern agriculture, especially as the population grows and available land for agriculture is reduced.

In agriculture, the safe and effective use of agrochemicals has successfully been carried out to solve various problems. However, these are also responsible for some important environmental and ecological imbalances. Excessive use of agrochemicals lead to the contamination of soil and groundwater with heavy metal. These heavy metals become a matter of concern as it get into the human food chain through higher uptake by crops and vegetables, grown for human consumption. Leaching of agrochemicals to water-bodies lead to the contamination of groundwater with nitrate, rendering it unfit for consumption by humans or livestock and even for crop production.

India is the largest producer of pesticides in Asia and ranks 12th in the world for the use of pesticides. A majority of population in India is engaged in agriculture and is therefore exposed to the pesticides used in agriculture. Although Indian average consumption of pesticide is far lower than many other developed economies, the problem of pesticide residue is high thereby affecting also affected the export of agricultural commodities in the last few years (Abhilash and Nandita Singh 2009).

India is the second largest producer of vegetables in the world (ranks next to china) & accounts for about 12 per cent of world production of vegetables with the productivity of 15 tonnes per ha which is quite low compared to other countries. The current production level is over 87.5 million tonnes and total area under vegetable cultivation is around 6.2 mha which is about 2.8 per cent of the total area under cultivation in the country (Sharma, 2008). In the case of vegetables potato, tomato, onion, cabbage & cauliflower account for around 60 per cent of the total vegetable production in the country. Vegetables are typically grown in India in field conditions.

Gujarat is one of the leading states whose agricultural growth rate is much higher than National agricultural growth rate. Intensive farming, proper use of chemical input and use of advance crop production technology by the farming community are some of the factors which contributed immensely in higher growth rate of agriculture in Gujarat. Abusive use of agrochemicals had certain drawbacks which are detrimental for sustainability of agricultural production. There are enough evidences at national and international level which signifies that abusive use of agrochemicals has lead to drastic reduction in quality of the soil. As in the case of Punjab soil productivity reduced due to this.

Though, Gujarat is far ahead in agricultural growth rate, certain important issues have been posing threat. Scarcity of good quality irrigation water is one such issue. Major part of Gujarat lies under rainfed condition where every drop of water has immense importance. To cope the growing inter and intra-sectoral competition for water and declining fresh water resources, the utilisation of “marginal quality water” for agriculture has posed a new challenge for environmental management (Abdulraheem, 1989). In water scarce areas there are competing demands from different sectors on the limited available water resources. Though industrial use of water is very low as compared to agricultural use, the disposal of industrial effluents on land and/or on surface water bodies make water (ground and surface) resources unsuitable for other uses (Burman *et al*., 2001 and Bahera & Reddy, 2002). Industry is a small user of water in terms of quantity, but has a significant impact on quality. Over three-fourth of fresh water draw by the domestic and industrial sector, return as domestic sewage and industrial effluents which inevitably end up in surface water bodies or in the groundwater, affecting water quality. The “marginal quality water” could potentially be used for other uses like irrigation. Hence, the reuse of wastewater for irrigation using domestic sewage or treated industrial effluents has been widely advocated by experts and is practiced in many parts of the world, particularly in water scarce regions. However, the environmental impact of reuse is not well documented, at least for industrial effluents, particularly in developing countries like India where the irrigation requirements are large.

OBJECTIVES:

Taking these points under consideration, an investigation entitled "An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat" was proposed with below following objectives:

(1) Estimation of nitrate-nitrogen, heavy metals and pesticide residues in ground water collected from cotton and vegetable growing areas of Padra, Karjan, Nadiad and Kheda of middle Gujarat.

(2) Status of phosphorus, heavy metals and pesticide residues in surface water.

(3) Estimation of heavy metals and pesticide residues in soil.

(4) Estimation of heavy metals and pesticide residues from vegetables and cotton lint.

II. REVIEW OF LITERATURE

In agriculture, the safe and effective use of agrochemicals has successfully been carried out to solve various problems. Consumer concerns on food safety and society awareness of chemical contaminants in the environment have increased in the past few years. As a consequence, more restrictions in the use of chemical products have been imposed at national and international levels. Though use of agrochemicals has helped India and other countries in achieving self-sufficiency in food production, their indiscriminate use has considerably polluted the environment.

Attempts have been made to collect literature on various aspects of present investigation pertaining to agrochemical contamination in the environment. The available literature related to present studies has been reviewed and presented hereunder.

2.1 Status of inorganic and organic contaminants in water

Kumarasamy *et al*. (2011) studied the levels of 17 organochlorine pesticides residues (OCPs) in surface water and sediments from Tamiraparani river basin, South India were investigated to evaluate their potential pollution and risk impacts. A total of 96 surface water and sediment samples at 12 sampling stations were collected along the river in four seasons during 2008–2009. The ΣOCP concentrations in surface water and sediments were in the range of 0.1 to 79.9 ng L^{-1} and 0.12 to 3,938.7 ng g^{-1} dry weights (dw), respectively. Among the OCPs, the levels of dichlorodiphenyltrichloroethanes (DDTs), aldrin, dieldrin, cis-chlordane, transchlordane, and mirex were dominant in the sediments. The dominant OCPs in water samples were heptachlor, *o,p′*-DDE, dieldrin, *o,p′*-DDD, and mirex, which showed different source of contamination pattern among sampling seasons.

Pujeri *et al*. (2010) evaluated seven water samples collected from Bijapur lakes. The water samples from Bijapur lakes contained significantly higher levels of endosulfan, 4-bromo-2-chlorophenol and chlorpyriphos. From the results it can be seen that endosulfan was detected in the range of 0.00025 to 0.005 mg/L. The concentration of 4-bromo-2-chlorophenol ranged from 0.01 to 0.009 mg/L. The concentration of captan was below the limit of WHO. The concentration of chlorpyriphos ethyl ranged from 0.0002 to 0.004 mg/L which was above the WHO limit. The fipronil was detected in only one sample i.e. 0.004 mg/L. The concentration of oxyfluorfen was 0.0025 mg/L which was below the limit, while monocrotophos was not detected in any of the samples.

Shadma and Pandey (2010) studied the effect of industrial waste water as irrigation water on status of heavy metals. They analysed the lettuce root and shoot tissues for heavy metals *viz*., Nickel (Ni), Chromium (Cr), Copper (Cu) and Zinc (Zn).They found that the concentration of Cu and Zn in industrial waste water was lower than Ni and Cr. The diluted (25 and 50%) and undiluted (100%) waste water was used to irrigate the lettuce plants grown in alluvial soils. Plants accumulated heavy metals in their shoot and root in high concentrations after irrigation with undiluted industrial waste water. Maximum accumulation of heavy metals was found in the root than the shoot.

Reddy *et al.* (2009) studied the ground water pollution due to nitrate contamination in north eastern part of Anantapur District, A.P., India. They analysed ground water samples during pre monsoon and post monsoon seasons. They found that the ground water contamination by nitrate was higher in pre monsoon samples than post monsoon.

Tagma *et al*. (2009) studied the ground water pollution due to nitrate by agricultural diffusion in Souss-Massa basin, Morocco and they observed that the western and south western parts of the Souss-Massa were highly contaminated by nitrate due to exceed application of nitrogenous fertilizers for increasing crop yield and economical purpose.

Schipper *et al.* (2008) studied leaching of heavy metal in surface water by agricultural soil in Netherland. They found that the levels of copper and zinc metal were higher in water due to manures and fertilizers use in agricultural soils.

Akan *et al.* (2008) conducted study on the quality of waste water and vegetable and the samples were collected from the Jakarta wastewater channel the Airport Road Bridge, Kano metropolis. They found that levels of nitrate, nitrite, sulphate and phosphate were higher than the maximum permissible limits set by Federal Environmental Protection Agencies (FEPA) of Nigeria. The concentrations of the metals in the wastewater and vegetables samples were higher than limits set by WHO and the maximum contaminant levels (MCL) due to the wastewater used for the irrigation of these vegetables.

Kaushik *et al*. (2008) studied Yamuna, a prominent river of India covers an extensive area of 345,843 km^2 from Yamunotri glacier through six Indian states. They found the residues of organochlorine pesticides (OCPs) namely, isomers of HCH and endosulfan, DDT and its metabolites, aldrin, dieldrin, were analysed in water samples of river Yamuna along its 346 km stretch passing through Haryana–Delhi– Haryana and the canals originating from it. They found that all samples contained β-HCH, *p,p′*-DDT, *p,p′*-DDE and *p,p′*-DDD in maximum traceability while other were γ- HCH, α-HCH, *o,p′*-DDD, *o,p′*- DDT, δ-HCH, *o,p′*-DDE, aldrin, dieldrin, α and β endosulfan remained below determination limits (BDL). In canals the values were found between 12.38–571.98 ng/L and 109.12–1572.22 ng/L for ΣHCH and ΣDDT, respectively. Water of Gurgaon canal and Western Yamuna canal contained maximum and minimum concentration, respectively both of ΣHCH and ΣDDT residues.

Kumari Beena *et al.* (2008) studied twelve samples each of soil and ground water collected from paddy-wheat, paddy-cotton, sugarcane fields and tube wells from same or near by fields around Hisar, Haryana, India during 2002–2003 to monitor pesticide residues. In soil, HCH (0.002–0.051 μg g^{-1}), DDT (0.001–0.066 μg g^{-1}), endosulfan (0.002–0.039 μg g^{-1}) and chlordane (0.0002–0.019 μg g^{-1}) among organochlorines, cypermethrin (0.001–0.035 μg g^{-1}) and fenvalerate (0.001–0.022 μg g^{-1}) among synthetic pyrethroids and chlorpyriphos (0.002–0.172 μg g^{-1}), malathion (0.002–0.008 μg g^{-1}), quinalphos (0.001–0.010 μg g^{-1}) among organophosphates were detected. Dominant contaminants were DDT, cypermethrin and chlorpyriphos from the respective groups. In water samples, HCH, DDT, endosulfan and cypermethrin residues were observed frequently. Only chlorpyriphos among organophosphates was detected in 10 samples. On consideration of tube well water for drinking purpose, about 80% samples were found to contain residues above the regulatory limits.

Kar *et al.* (2008) collected 96 surface water samples collected from river Ganga in West Bengal during 2004-05 and analyzed for pH, EC, Fe, Mn, Zn, Cu, Cd, Cr, Pb and Ni. The pH was found in the alkaline range (7.21-8.32), while conductance was obtained in the range of 0.225-0.615 mmhos/cm. The Fe, Mn, Zn, Ni, Cr and Pb were detected in more than 92% of the samples in the range of 0.025-5.49, 0.025-2.72, 0.012-0.370, 0.012-0.375, 0.001-0.044 and 0.001-0.250 mg/L, respectively, whereas Cd and Cu were detected only in 20 and 36 samples (Cd: 0.001-0.003 and Cu: 0.003-0.032

mg/L). Overall seasonal variation was significant for Fe, Mn, Cd and Cr. The maximum mean concentration of Fe (1.520 mg/L) was observed in summer, Mn (0.423 mg/L) in monsoon but Cd (0.003 mg/L) and Cr (0.020 mg/L) exhibited their maximum during the winter season. The Fe, Mn and Cd concentration also varied with the change of sampling locations. The highest mean concentrations (mg/L) of Fe (1.485), Zn (0.085) and Cu (0.006) were observed at Palta, those for Mn (0.420) and Ni (0.054) at Berhampore, whereas the maximum of Pb (0.024 mg/L) and Cr (0.018 mg/L) was obtained at the downstream station, Uluberia. All in all, the dominance of various heavy metals in the surface water of the river Ganga followed the sequence: Fe > Mn > Ni > Cr > Pb > Zn > Cu > Cd. A significant positive correlation was exhibited for conductivity with Cd and Cr of water but Mn exhibited a negative correlation with conductivity.

Sankararamakrishnan *et al*. (2008) studied nitrate- nitrogen and fluoride concentration in shallow and unconfined ground water aquifers of Kanpur district along the Ganges Alluvial Plain of Northern India. They divided Kanpur district into three zones namely, Bithore, Kanpur city and Beyond Jajmau and sampling was carried out in three seasons (summer, moon and winter). The water samples from around 99 India Mark II hand Pumps, were analyzed for summer monsoon and winter seasons. In Bithore zone, 19% of the samples exceeded the BIS (Bureau of India Standards) limit of 10.2 mg/L nitrate–N and as high as 166 mg/L as nitrate–N was observed. About 10% and 7% samples in Kanpur city and beyond Jajmau zone respectively, exceeded the BIS limit. The Frequency distribution histogram of nitrate–N revealed a skewed (non-normal) distribution. Both point and non-point sources contribute to the ground water contamination. Especially in Bithore zone, the point sources could be attributed to the animal wastes derived from cows and buffaloes and non point sources could be due to the extensive agricultural activity prevalent in that area.

Kumari Beena *et al.* (2007) studied that presence of pesticide residues in rain water during 2002 employing multi residue analysis method by Gas Liquid Chromatography equipped with ECD and NPD detectors and capillary columns. The presence of pesticide residues in surface aquatic system triggered the investigation of the presence of pesticides in rain water. A total of 13 pesticides were detected in rain water samples. Among the different groups of pesticides, organochlorines were present in the range of 0.041–7.060 ppb with maximum concentration of *p,p′*-DDT up to 7.060 μg L^{-1}. Synthetic pyrethroids were present ranging from 0.100 to 1.000 μg L^{-1} and organophosphates in the range of 0.050–4.000 μg L^{-1} showing maximum contamination with cypermethrin (1.000 μg L^{-1}) and monocrotophos (4.000 μg L^{-1}) of the respective groups. Almost 80% samples showed the residues above MRL of 0.5 ppb fixed for multi residues and on the basis of single pesticide, 16–50% samples contained residues above the MRL value of 0.1 ppb.

Ahad *et al*. (2006) determined the residues of pesticides from the drinking water of Rawal Lake, Islamabad, Pakistan in 2004. It was found that residues of methyl parathion, fenitrothion, azinphosmethyl and alpha-cypermethrin were detected in the concentrations of 0.38 μg L^{-1}, 0.62 μg L^{-1}, 3.3 μg L^{-1} and 5.82 μg L^{-1} by GC (NPD and ECD), respectively. The pyrethroid pesticide residues values were four times higher than EEC standards for drinking water.

Deka *et al*. (2005) collected twenty drinking water samples from different pockets of greater Jorhat district of Assam, India during 2001-2002 to monitor pesticide residues. Total residues of HCH, endosulfan and DDT ranged from 0.019-0.031, 0.005-0.007 and 0.025-0.035 ng mL^{-1}, respectively. Pesticides were only detected in the pond and stream samples but not in the tube well and deep well water samples. All the water samples contained pesticide residues below the regulatory limit prescribed by European Union (EU).

Liu *et al*. (2006) studied the Oraganochlorine residues in water and soil in agriculture product in China Xiaogan. The residues of DDT, HCH were determined according to the national sanitary GB 5009 (1985). Detection of DDT and HCH was in 100% soil and 94.44% water samples. The residue of HCH was highest in the vegetables, planting soil and in crop. Planting soil contained highest (0.510 µg kg^{-1}) β-HCH and (19.86) *p,p'*-DDT µg kg^{-1} respectively. In surface water *p,p'*-DDE was highest (0.062 µg L^{-1}). The residues of organochlorine were found even in the deep well water with 70 meter depth.

Sankararamakrishnan *et al.* (2005) undertook a survey in Kanpur, northern India, and reported the presence of high concentrations of both organochlorine and organophosphorous pesticides in the surface and ground water samples. Among various pesticides analyzed, high concentrations of γ-HCH (0.259 µg/L) and malathion (2.618 µg /L) were detected in the surface water samples collected from the River Ganges in Kanpur. In the ground water samples collected from the various hand pumps located in agricultural and industrial areas, apart from γ -HCH and malathion, dieldrin was also detected. The maximum concentration values of γ -HCH, malathion and dieldrin were 0.900, 29.835 and 16.227 µg/L, respectively. Especially, the concentration of malathion was found to be much higher than the EC water quality standards in the ground water samples from industrial area posing a high risk to the common people. Pesticides like DDE, DDT, aldrin, ethion, methyl parathion and endosulfan were not detected in both the surface and ground water samples.

Tariq *et al*. (2004) evaluated pesticides from ground water in four intensive cotton growing districts of Pakistan. Water samples were collected from 37 rural open wells in areas of Bahwalnagar, Muzafargarh, D. G. Kanh and Rajan pur districts of Punjab, Pakistan and detected eight pesticides viz., bifenthrin, cyhalothrin, carbofuran, endosulfan, methyl parathion and monocrotophos. And respective concentration were 13.5%, 5.4%, 59.4%, 8%, 5.4% and 35.1% in July; 16.2%, 13.5%, 43.2%, 8% and 24.3% in October.

Kumazawa Kikuo (2002) studied nitrate nitrogen (NO_3-N) concentrations in groundwater in Japan and showed that it increased steadily due to the development of intensive agriculture. In some areas, they content reached or even exceeded the unacceptable level for drinking water, i.e. 10 mg L^{-1}. In 2000, the Environment Agency showed that 5.6% (173 of 3,374) tested wells and 4.7% (64 of 1,362) wells used for drinking water exceeded the standard level in 1999. The highest value of NO_3-N in the wells was 100 mg L^{-1}. Many researchers have shown that NO_3-N pollution of groundwater was widely observed in Japan, except the paddy field regions. Farming practices in Kagamigahara city of Gifu prefecture have been typical ones for reducing NO_3-N pollution in groundwater. In the east district of the city, NO_3-N concentration was low in 1966, but reached 27.5 mg L^{-1} in June, 1974. The farmers in this district began to reduce the nitrogen fertilizers in carrot cultivation, going from 256 kg N ha^{-1} in 1970 to 153 kg N ha^{-1} in 1991. The use of controlled release fertilizer increased fertilizer-nitrogen use efficiency compared with common compound fertilizer and NO_3-N concentration in the groundwater began to decrease steadily. It was discussed that in order to decrease the NO_3-N pollution of groundwater, it is necessary to refocus not only on agricultural technology but also agricultural policy, toward sustainable agriculture and rural development.

2.2 Status of inorganic and organic contaminants in soil

Kumar *et al*. (2011) conducted the study to monitor contamination of organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) in agricultural soils from the northern states of India. The average concentration of OCPs was 37.67 ± 0.33 ng/g (dry weight − DW), while HCHs alone accounted for 93% followed by DDT (4.27%) and endosulphan (2.51%). The α/γ ratio of HCH (< 0.01–8.64) reflects the use of technical as well as lindane formulations. The ratio of *p,p′*-DDT/*p,p′*-DDE (0.16) and *o,p′*-DDT/*p,p′*-DDT (<0.01) indicates the contamination of soils with the past use of technical DDT. The mean concentrations of endosulphan and dieldrin were 0.95 ± 0.53 ng/g (DW) and 0.16 ± 0.07 ng/g (DW), respectively. The average concentration of PCBs was 13.44 ± 0.06 ng/g (DW). The toxic equivalency (TEQ) calculated using WHO 2005-TEFs ranged from 0.01 to 105.40 pg WHO 2005-TEQ/g (DW) with the mean of 13.78 ± 0.11 pg WHO 2005-TEQ/g (DW). PCB-105 (25%), PCB-114 (18%), and PCB-118 (18%) were the dominant congeners and accounted for 61%, while non ortho PCBs contributed only 18% to total DL-PCBs.

Oyekunle *et al*. (2009) conducted study to evaluate the levels and seasonal variations of some organochlorine pesticides (OCPs) in the cultivated land of Oke-Osun farm settlement, Osogbo, Nigeria. A field sampling programme was conducted in the rainy and dry seasons for 4 months each resulting in the analysis of a total of 40 samples. Soil samples collected at 20-m intervals were air-dried to a constant weight, sieved through a mesh of 2.0-mm pore size and selected by coning and quartering method. Solid–liquid extraction was used to extract OCPs from the soil. Qualitative identifications and quantitative evaluation of the OCPs were carried out with the aid of a Perkin Elmer gas chromatograph coupled with electron capture detector. Seasonal mean ranges of OCPs in soil (μg/kg) were 13.09± 21.66 *β*- BHC–42.01 ± 17.50 *p,p'*-DDT in rainy season and 30.74 ± 17.38 *α*-BHC–82.88 ± 32.24 *p,p'*-DDT in the dry season. The results obtained from this study revealed that agricultural soil samples of Oke-Osun farm settlement were contaminated with persistent organochlorine pesticides mainly as a result of their applications by farmers. Higher levels of OCPs were obtained for dry season than the rainy season. There were indications from this study that pesticides that have deleterious health effects on humans previously placed under legal restrictions by regulatory agencies were still being used by the farmers of Oke-Osun farm settlement and this gives cause for environmental concern.

Acosta *et al*. (2009) identified sources of heavy metal contamination by multivariable soil analysis. They found that the contamination source of Cr, Mn and Ni were associated with soil parent materials; Cd and Pb contents were related to anthropogenic activities, specifically industrial activities and traffic; Cu content due to controlled by long-term application of inorganic fertilizers in agricultural areas.

Oyekunle *et al*. (2009) conducted a study to evaluate the levels and seasonal variations of some organochlorine pesticides (OCPs) in the cultivated land of Oke-Osun farm settlement, Osogbo, Nigeria. This study revealed that agricultural soil samples of Oke-Osun farm were contaminated with persistent organochlorine pesticides mainly as a result of their applications by farmers. Higher levels of OCPs were obtained for dry season than the rainy season.

Toan *et al.* (2006) studied organochlorine pesticides residues in the surface soils of Hanoi, Vietnam. Sixty representative soil samples were collected from the centre of Hanoi and five surrounding districts in agricultural areas. They found that among OCPs, HCH isomers and Σ DDT in surface soil samples were the maximum.

Geng *et al*. (2006) measured the pollution levels of OC pesticides and PCBs in the soil samples collected from arable land, residential and vegetable lands. The values of PCBs in the soil were in a range of 3.06-14.88 ng g^{-1} with an average value of 8.04 ng g^{-1}.The residues of OC in the soil were in range of 5.07-87.77 ng g^{-1} with an average value of 32.58 ng g^{-1}. Σ HCHs and Σ DDTs accounted for over 12.13% and 81.37% of the total OC, respectively. The DDT and its metabolites were the main pollutants in the soil.

Jayshree and Vasudevan (2006) investigated the level of pesticides in agricultural soil of Thiruvalliur district (Kerala), India. Sample contained *p,p'*-DDT from 0.9-10.3 µg kg^{-1} of soil and HCH derivative such as HCH ranged from 0.9-43.6 µg kg^{-1}. Among the OCs, endosulfan sulfate content (29.2 µg kg^{-1}) was high in soils of few villages in the district. Among the different OCs, endosulfan sulfate was found almost in all the samples in very high concentration. The results showed that the soils of the district contained significant levels of DDT, HCH and endosulfan and their derivatives.

Gao *et al*. (2005) quantified the OCs like DDT, DDD, HCH, HCB, endosulfan, dieldrin and endrin to detect current level of OC in agricultural soil by GC (ECD). Thirteen OCs were detected and DDT was the main residue, while HCH ranked second.

Qiu *et al*. (2005) determined the DDT and its metabolites (DDTs) and hexa-chlorocyclohexane isomer (HCHs) in agricultural soils from north of Zhejiang province. The results showed that the concentration of Σ HCHs and Σ DDTs in the soil ranged from 0.20-20.1 ng g^{-1} and 1.50-326.8 ng g^{-1}, respectively and the mean of these were 1.73 ng g^{-1} and 44.68 ng g^{-1}, respectively. It was found that Σ DDTs concentration in some samples (81 out 919) exceeded the official Chinese norms for DDT.

Aydinalp and Marinova (2002) studied the distribution and forms of heavy metals *viz.*, Cd, Cu, Mn, Ni and Zn in the agricultural soils of the Busra city (Bulgaria) to ascertain the degree of pollution and found that the total heavy metal content of the soils was higher than the levels reported in literature for similar soil which indicate some degree of heavy metal pollution in soil.

FAO (2001) reported that 24 soil samples from two depths (1-6 and 6-12 inches) from 6 locations of cotton zone of Punjab were found contaminated with pesticide residues. The fenvalerate, lindane and azinphosmethyl were found higher in lower layer than the top layer, in the lower layer lindane residues were also found.

2.3 Status of heavy metal and pesticide residues in vegetable and cotton lint

Swarnam and Velmurugan (2013) studied the pesticide residues in vegetable samples *viz;* brinjal, okra, green chilli, crucifers and cucurbits collected from farmers' field. They found that 34% samples were contaminated with pesticides. Among them OC compounds (α-endosulfan, β-endosulfan and endosulfan sulfate) were detected in 14.5% samples. The SP compounds such as α-cypermethrin, fenvalerate-I, fluvalinate I, deltamethrin and λ-cyhalothrin were detected in 32% samples. The OP compound residues such as chlorpyriphos, profenophos, monocrotophos, triazophos, ethion, dimethoate and acephate were found in 54% of the samples. The15.3% samples were found to contain residues exceeding the prescribed maximum residue limit. Multiple residues of more than one compound were detected in 34.1% of samples containing residues.

Elbagermi *et al*. (2012) monitored the heavy metals *viz*., Pb, Cu, Zn, Co, Ni and Cd contents in fruits and vegetables collected from production and market sites in Misurata area of Libya. The results of this study showed that the average concentrations detected ranged from 0.02 to 1.824, 0.75 to 6.21, 0.042 to 11.4, 0.141 to 1.168, 0.19 to 5.143 and 0.01 to 0.362 mg/kg for Pb, Cu, Zn, Co, Ni and Cd, respectively.

Inoti *et al.* (2012) studied the consumption of leafy vegetables grown in urban areas contaminated with heavy metals which is a serious major source of health problems for both human and animals at Thika town situated in Central, Kenya. This study was conducted to analyze the heavy metal levels in tomato (*Lycopersicon esculentum* L.) and spinach (*Spinacia oleracea* L.) grown in Thika town. The mean concentrations of Pb, Zn and Cd in all the samples were more than the maximum permitted concentrations while there was no evidence of copper (Cu) contamination. Heavy metal uptake differences in the vegetables were attributed to plant differences in tolerance. Lead concentration in the vegetables was above the maximum limit of 0.3 mg kg^{-1} accepted for human health by WHO.

Osman *et al*. (2010) studied contamination of 23 pesticides from different chemical groups in 160 different domestic vegetables collected from four major big supermarkets located in Al-Qassim region, Saudi Arabia. Pesticides were identified by gas chromatography with mass spectrometry (GC–MS). Residues were found in 89 of the 160 samples and 53 samples were above the maximum residue levels (MRLs). The most frequently found pesticides were carbaryl followed by biphenyl and carbofuran. Cabbage was the most positive and violated MRLs (16 and 11 samples), followed by carrot and green pepper (12 and 7 samples), cucumber (12 and 6 samples), egg-plant (12 and 5 samples), squash (11 and 7 samples), lettuce (11 and 6 samples) and tomato (11 and 4 samples). The highest concentrations were found in lettuce (ethiofencarb, 7.648), followed by tomato (tolclofos-methyl, 7.312 mg/kg), cabbage (chlropyriphos, 6.207 mg/kg), carrot (heptanophos, 3.267 mg/kg), green pepper (carbaryl, 2.228 mg/kg) and egg-plant (carbaryl, 1.917 mg/ kg).

Owago *et al*. (2009) conducted the study to determine the residue levels of organochlorine pesticides (OCPs) in 34 samples of 19 varieties of vegetables collected from selected sites around Deyang city and Yanting County, Southwest China, in June and September 2007. The compounds targeted were: isomers of hexachlorocyclohexane or HCHs (α-HCH, β-HCH, γ- HCH or lindane and δ-HCH); isomers/metabolites of dichloro-diphenyl-trichloroethane (DDTs) namely *p,p'*-dichloro-diphenyl-dichloroethylene (*p,p'*-DDE), *p,p'*-dichloro-diphenyl-dichloroethane *(p,p'*-DDD), *p,p'*- and *o,p'*-dichloro-diphenyl- trichloroethane (*p,p'*-DDT and *o,p'*-DDT). The results indicated that all the vegetable samples had some levels of one or more OCPs in them. Residues of DDTs were found in 94.12% while HCHs were in 91.18% of all the samples analyzed indicating high incidence of these xenobiotics in the vegetables from the areas investigated. Among the HCH isomers, γ- HCH was the most prevalent but β-HCH was the most abundant indicating both old and fresh inputs of HCHs. The DDT metabolites *p,p'*-DDE and *p,p'*-DDD were more prevalent than the parent material, *p,p'*-DDT suggesting minimal fresh inputs of DDT. The OCPs residue levels in the vegetables were generally low (1.3 ng/ g wet weight) except in one sample of green pepper (*Capsicum annum L*) in which the concentrations (ng/g wet weight) of *o,p'*-DDT (82.59), *p,p'*-DDE (61.41) and total DDT (148.44), all exceeded the Chinese Extraneous Maximum Residue Limit of 50 ng/ g for DDTs in vegetables according to the guidelines of the Chinese quality standard for food (GB 2763-2005).

Baig *et al*. (2009) evaluated residues of three pesticides in vegetables, belonging to organophosphate group, which was extensively used in the Southern part of Punjab province in Pakistan. Triazophos, profenophos and chlorpyriphos were commonly used on the summer vegetables i.e., egg plant, pumpkins and okra cultivated in study area. They observed that 33.0% of the samples were contaminated with any of the above three pesticides. The results showed that 8% of the samples tested contained residues higher than the MRLs.

Anwar (2008) conducted survey to study the status of pesticide residues in water, oil, fruits and vegetables in cotton growing areas of Sindh and lower Punjab, Pakistan and found that 70% water samples, 37% vegetable samples and all soil samples were contaminated.

Bhanti and Taneja (2007) evaluated the residual concentration of selected OP pesticides (methyl parathion, chlorpyriphos and malathion) in vegetables grown in different seasons (summer, rainy and winter). Pesticide residues in vegetables of different seasons showed that winter vegetables were the most contaminated followed by summer and raining vegetables.

Hassan *et al*. (2007) analyzed 124 vegetable samples for the pesticide residue of 7 commonly used pesticides. It was observed that 89 vegetable sample were found to be contaminated. Out of which 47 samples exceeded MRLs. Cypermethrin was found in 39 samples, methamidophos in 27, fenvalerate in 22, malathion in 20, chlorpyriphos in 9, endosulfan in 7 and methyl parathion was found only in one sample of vegetable.

Quintero *et al.* (2006) studied the residues of seven organophosphorus pesticides *viz*., methamidophos, diazinon, chlorpyriphos, parathion-methyl, dimethoate, malathion and tetrachlorvinphos in some vegetables like: potato, lettuce, tomato, onion, red pepper and green onion cultivated in Jose Maria Vargas County in Tachira State, Venezuela. They observed that 48.0% of the samples were contaminated with some of the pesticides. They found that the methamidophos residues were higher compared to other pesticide in the vegetable. The results showed that 16.7% of the samples tested had residues higher than the maximum limits permitted.

Bhattachrya *et al*. (2005) studied the residue levels of OC pesticides in marketable samples from an agricultural field, of West Bengal. Results revealed that in some samples the residue levels were much higher, above the acceptable daily intake value and few of them contained residues levels above the tolerance levels. The endrin, aldrin, dieldrin, endosulfan and DDT pesticide residues concentrations were found to be as high as 0.594, 0.620, 0.712, 1.892 and 0.122 ppm, respectively, which were above the tolerance limit.

Deka *et al*. (2005) monitored the samples of cabbage (22), cauliflower (25), ridge gourd (8), knotholes (4), okra (14), tomato (18), chilli (8), brinjal (25), potato (12), pointed gourd (7), bottle gourd (8), bitter gourd (6), carrot (8) and sweet beets (5) collected from 6 different agro-climatic zones of Assam for pesticide residues. Out of 170 samples124 samples were found to be contaminated but did not exceed the MRL.

Sardana *et al*. (2005) studied the pesticide residues in okra and brinjal fruits from non-IPM and IPM fields before their delivery to market. Samples were tested for chlorpyriphos, cypermethrin, monocrotophos, dimethoate and deltamethrin, widely used on these crops in Raispur village, Ghaziabad districts (UP, India) during 2003-04 and 2004-05. The soil samples from fields before sowing and after harvesting of both the crops were also analyzed for chlorpyriphos residue during 2003-04. Residue level of chlorpyriphos in soil was 0.41 $\mu g\ g^{-1}$ before start of okra trial and increased to 4.22 and

1.41 µg g^{-1} at harvest time in non-IPM and IPM fields of okra crop, respectively, while in brinjal crop the chlorpyriphos was non detectable (ND) in soil of both IPM and non-IPM fields before and after trial. During 2003-04 the residues of chlorpyriphos and cypermethrin in okra fruits were found to be 0.10 mg g^{-1} and could not be detected in non IPM trials and 5.75 and 0.63 µg g^{-1} for non-IPM fields while monocrotophos in brinjal fruits was not found in IPM and 1.24 µg g^{-1} detected in non-IPM respectively.

Blossom and Singh (2004) estimated the residues of different insecticides in lint samples collected from cotton growing areas of Punjab. Majority of the samples were contaminated with endosulfan residues. Ethion and cypermethrin residues were detected in 80% of samples. The residues of chlorpyriphos, fenvalerate, triazophos, deltamethrin and profenophos were also present in 40, 28, 14, 8 and 8% of the lint samples, respectively.

FAO (2001) discussed the presence of the pesticide residues from Pakistan. In vegetable samples collected from different locations around multan city. It was observed that all the vegetable samples analyzed were contaminated, out of which 63% samples found exceeding MRLs for carbofuran, dichlorvos, methyl parathion, fenitrothion and quinalphos methyl, while the pyrethroids, deltamethrin and cypermethrin were not detected in any samples.

Ahuja *et al.* (1998) monitored cauliflower, cabbages, tomatoes, brinjal, okra, cucumber, french beans and field beans at harvest for residues of HCH and its isomers, endosulfan, dimethoate, monocrotophos, quinalphos, fenvalerate, cypermethrin and carbendazim. The residues of α, β and τ isomers of HCH, endosulfan, monocrotophos, quinalphos, dimethoate and carbendazim were detected in most of the analyzed samples. However, the residues of Monocrotophos on tomatoes, brinjal and okra and those of carbendazim were found above the MRL.

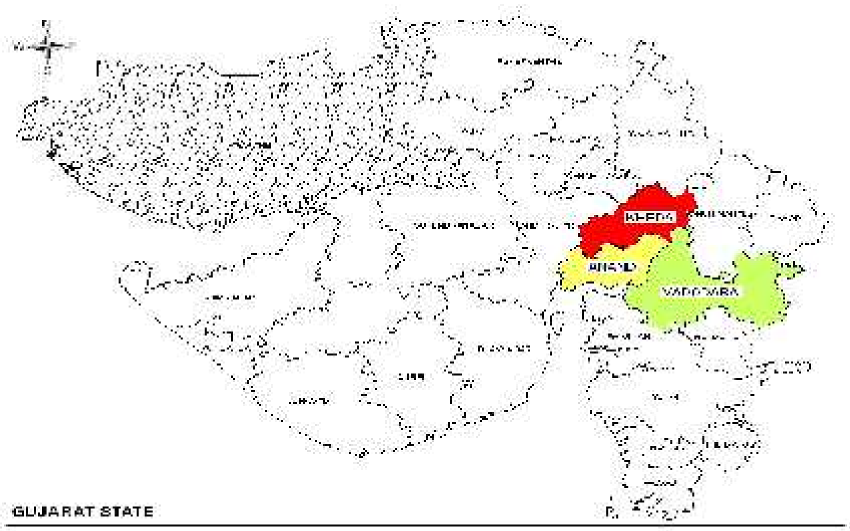
III. MATERIALS AND METHODS

This chapter deals with the particulars of materials used during the course of exploration and methods adopted in conducting the present investigation entitled "**An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat ".**

3.1 Experimental details

3.1.1 Experimental setup: A laboratory study was carried out at laboratory AINP on pesticide residues, and Micronutrient Project, ICAR Unit -9 , AAU, Anand.

3.1.2 Sampling area: Kheda, Anand and Vadodara districts of Middle Gujarat area having intensive cultivation of vegetables and cotton.



3.1.3 No. of samples: Total 203 samples were collected from intensive agricultural area of middle Gujarat. Every information on the nature of soil, crop history, pesticide and fertilizers applied, water table depth, industrial activity nearby if any, etc were recorded.

Soil samples	: 80 soil samples collected from cotton growing area and 22 soil samples collected from vegetable growing area
Ground/ surface water samples	: 41 ground water samples and 10 surface water were collected.
Vegetable samples + cotton lint	: 21 vegetable samples and 29 cotton lint samples were collected.

3.1.4 Sampling procedure: Standard sampling procedure recommended for water and soil was followed

Soil: Samples were collected from the depth of 0-15 cm by auger. For making composite sample, small portions of soil were collected from 15-20 well distributed spots, moving in a zigzag manner from each individual sampling site. Soil bulk was reduced to about 500 g by quartering method and kept it in a clean cloth bag.

Ground water: Before ground water sample collection, tube well was switched on for 15-20 minutes for purging. Water sample filtered through muslin cloth was collected in to a 2.5 Littre amber colour glass bottle.

3.2 Solvents and other chemicals

3.2.1 Acetone

Analytical grade acetone (2.5 L) was refluxed with $KMnO_4$ (0.5 g) till violet colour persisted. The acetone was then glass distilled at 58 °C.

3.2.2. Methylene chloride/Dichloromethane (DCM)

Analytical grade methylene chloride (dichloromethane) was glass distilled at 39 °C.

3.2.3 Petroleum spirit (40-60 ºC)

Analytical grade petroleum spirit was distilled in glass assembly. The suitability of petroleum spirit was checked by concentrating 50 mL of the solvent to 1.0 mL, which was subjected to Gas Liquid Chromatograph- Electron Capture Detector (GLC-ECD) for the detection of possible impurities.

3.2.4 Toluene

Analytical grade toluene was distilled in glass assembly for the preparation of the working standards. Its suitability was checked on GLC-ECD.

3.2.5 Acetonitrile

Analytical grade acetonitrile (purity ≥ 99.9%) was used as such without distillation although its suitability was checked on GLC-ECD by completely evaporating 50 mL of solvent and then made up the volume to 1.0 mL with a mixture of petroleum spirit : acetone (1:1, v/v).

3.2.6 Acetic Acid

HPLC grade acetic acid was used as such without distillation for the extraction.

3.2.7 Cotton

Cotton was purified by Soxhlet extraction using petroleum spirit : acetone (1:1, v/v) for 6.0 h followed by drying in oven at 60°C for 1.0 h. It was stored in a closed glass container.

3.2.8 Sodium chloride (NaCl)

Phthalate esters, present in reagent grade sodium chloride generally interfere in GLC-ECD analysis. Therefore, to remove the phthalate esters, sodium chloride was subjected to 540°C in muffle furnace for 6 h and stored in glass container.

3.2.9 Sodium sulfate (Na_2SO_4)

Anhydrous, granular, reagent grade sodium sulfate was purified in the same way as sodium chloride and stored in glass container.

3.2.10 Magnesium sulfate ($MgSO_4$)

Anhydrous, fine powdered, reagent grade magnesium sulfate (≥99.5%) was used as such for analysis and stored in desiccators at room temperature.

3.2.11 PSA (Primary Secondary Amine)

Primary and Secondary Amine (Ethylenediamine-N-propyl**,** Bond Elut™ PSA) was used as anion exchanger for dispersive column cleanup and was kept in a desiccator at room temperature. PSA removes fatty acids, sugars and other H-bonding matrix co-extractives. Its particle size is 40 μm.

3.2.12 Sodium acetate (CH_3COONa)

Anhydrous, fine powdered, reagent grade sodium acetate was used as such for analysis and stored in desiccator at room temperature.

3.3 Equipments and instruments

3.3.1 Equipments

3.3.1.1 Homogeniser

'Robot coupe, Blixer 6 v.v' mixer was used to homogenize the vegetable samples.

3.3.1.2 Weighing balances

'Contech' make digital weighing balance (1000 g capacity and 0.01 g sensitivity was used for weighing the samples and reagents while 'Sartorious basic plus' balance (maximum capacity 210 g and sensitivity 0.01 mg) was used for weighing pesticide standards.

3.3.1.3 Vacuum rotary evaporator

'Heidolph' make vacuum rotary evaporator was used to concentrate the large volume of the sample extract.

3.3.1.4 Turbo Vap LV (Concentrate workstation)

'Caliper' make nitrogen based evaporator was used for reducing the final volume of the sample under inert atmosphere.

3.3.1.5 Centrifuge

A high speed digitally controlled centrifuge model 'R-23' obtained from Remi Instruments Ltd. was used.

3.3.1.6 Vortex mixer

A simple vortex mixer from Tarson Ltd. was used to mix the solvent in test tube.

3.3.1.7 Water bath

Nova water bath was used to evaporate the water from porcelain dish for determination of Nitrate-Nitrogen.

3.3.1.8 Hot plate

Nova hot plate was used to evaporate the water, vegetable and cotton lint samples after di-acid digestion.

3.3.1.9 Mechanical shaker

A mechanical shaker from Orbital Shaking Incubator used to shake soil samples for heavy metal analysis.

3.3.2 Instruments

3.3.2.1 Gas Liquid Chromatograph (GLC)

Two different makes of Gas Liquid Chromatographs (GLC) were used for the qualitative and quantitative estimation of pesticide residues *viz.*, Shimadzu GC 2010, Varian 450 GC equipped with Electron Capture Detector (ECD) and Thermo Trace Ultra GC equipped with Flame Photometry Detector (FPD).

3.3.2.2 Gas Chromatograph- Mass Spectrometer (GC-MS)

Confirmation of detected pesticides was performed on gas chromatograph-mass spectrometer namely Shimadzu GCMS-QP2010 plus equipped with quadruple.

3.3.2.3 Atomic Absorption Spectrophotometer (AAS)

'Perkin Elmer' AAS 3110 was used for the qualitative and quantitative estimation of heavy metal.

3.3.2.4 UV/VIS Spectrophotometer

'ELico® SL 177' make UV/VIS Spectrophotometer was used for measurement of organic carbon, nitrate-nitrogen and phosphorous.

3.3.2.5 EC meter

'EUTECH' make EC meter was used for EC measurement of water and soil samples.

3.3.2.6 pH meter

'EUTECH' make pH meter was used for pH measurement of water and soil samples.

3.4 Certified Reference Standards (CRMs)

Certified reference standards (97% purity) for organophosphates, organochlorines, synthetic pyrethroids, herbicides and other pesticides were obtained from Sigma-Aldrich Ltd. (USA). All organic solvents used in the standard preparation were analytical grade.

3.4.1 Primary standard

A technical grade pesticide standard was accurately weighed on Sartorious basic plus balance (maximum capacity 210 g and sensitivity 0.01 mg). It was then transferred to volumetric flask (A grade). The standard was initially dissolved with distilled toluene and final volume was made up.

3.4.2 Secondary/Intermediate standard

The primary standard was diluted to 25 mL with distilled toluene in volumetric flask. This gave a concentration of 10 to 20 ppm.

3.4.3 Final working standards

The intermediate standards were used for the preparation of final working standards. Suitable aliquots were diluted to required final volume using distilled toluene, to obtain final concentration of 0.01, 0.025, 0.05, 0.1, 0.25, 0.5 and 1.0 ppm.

3.5 Linearity study

A linearity study was performed to determine the performance of ECD/ FPD detector. For the linearity study, a graph of response (height/area) v/s concentration was plotted. To establish the linearity of individual detector of each gas chromatograph, seven different concentrations of the standards *viz.*, 0.01, 0.025, 0.05, 0.1, 0.25, 0.5 and 1.0 ppm were injected and their response (μV) was recorded. A correlation coefficient and equation was determined by using best fit model of linear relationship.

3.6 Residue analysis

3.6.1 Pesticide residues from water samples

Method of analysis

Solvents and Regents: Petroleum spirit, Acetone, NaCl, Methylene chloride/Dichloromethane (DCM), Na_2SO_4, Cotton

Apparatus: Weighing Balance, Rotary vacuum evaporator and Turbo Vap LV

Instrument: Varian GC 450 with TSD/ ECD and Thermo Trace Ultra GC with FPD.

Table: 3.1 Scope of the pesticides for water, soil, vegetable and cotton lint samples

Class	Pesticides
OCs	α-HCH, γ-HCH, β-HCH, heptachlor, δ-HCH, aldrin heptachlor epoxide, chlordane-*cis*, chlordane-*trans*, α-endosulfan, *p,p*'-DDE, dieldrin, endrin *p,p*'-DDD, β-endosulfan, *p,p*'-DDT, endosulfan sulfate, endrin ketone, methoxychlor
OPs	DDVP, phorate, dimethoate, parathion methyl, fenitrothion, phorate sulphone, malathion, phorate sulphoxide, chlorpyrifos, chlorfenvinphos, profenophos, ethion, quinalphos, triazophos, phosalone
SPs	bifenthrin, fenpropathrin, λ-cyhalothrin, β-cyfluthrin, α-cypermethrin, fenvalerate, deltamethrin
HBs	trifluralin, fluchloralin, alachlor, metolachlor, butachlor, pendimethylene

Extraction of Water sample (AOAC Method)

A 750 mL representative water sample was taken in to 1000 mL separatory funnel followed by 150 g NaCl. The residue was extracted thrice with methylene chloride (DCM) 100 mL, 50 mL and 50 mL each and shaken vigorously. The lower organic phase was collected each time passing through bed of anhydrous Na_2SO_4. The combined DCM phase was completely evaporated using a rotary vacuum evaporator (below 40 °C). The concentration step was repeated thrice in the presence of petroleum spirit to remove the traces of methylene chloride. Final volume was made by petroleum spirit:acetone (1:1;v/v) and subjected to analysis on a Varian GC 450 and FPD analysis on a Thermo Trace Ultra GC for quantitation.

3.6.2 Pesticide residues from soils

Method of analysis

Solvents and Regents: Acetonitrile, Petroleum spirit, Acetone, NaCl, $MgSO_4$ and PSA (Primary Secondary Amine)

Apparatus: Weighing Balance, Centrifuge and Turbo Vap LV, Vortex mixer

Instrument: Varian GC 450 with TSD/ ECD and Thermo Trace Ultra GC with FPD.

Extraction of soil sample (QuEChERS Method)

Ten gramme representative soil sample was taken in to a 50 mL polypropylene centrifuge tube, and 20 mL acetonitrile followed by mixture of 1 g NaCl + 4 g $MgSO_4$ was added and shaken vigorously by hand (1 min.) well before centrifuge. The tubes were centrifuged at 3500 rpm for 3 minutes. A 10 mL aliquot from the supernatant was transferred by auto pipette in to a 15 mL polypropylene centrifuge tube containing 1.5 g $MgSO_4$ and 0.25 g PSA, followed by centrifugation at 3500 rpm for 2 minute. A 4.0 mL aliquot was transferred into a glass test tube and acetonitrile was completely evaporated on a Turbo Vap LV. Final volume was made by 1:1; petroleum spirit:acetone and subjected to ECD analysis on a Varian 450 GC and FPD analysis on a Thermo Trace Ultra GC.

3.6.3 Pesticide residues from vegetables

Method of analysis

Solvents and Regents: Acetonitrile, Petroleum spirit, Acetone, $MgSO_4$ and PSA (Primary Secondary Amine), Sodium acetate

Apparatus: Weighing Balance, Centrifuge and Turbo Vap LV, Vortex mixer

Instrument: Varian 450 GC with TSD/ ECD and Thermo Trace Ultra GC with FPD.

Extraction of vegetable sample (QuEChERS Method)

Measured 15 g representative vegetable sample in to a 50 mL polypropylene centrifuge tube, and 15 mL 1% acetic acid in acetonitrile followed by mixture of 6g $MgSO_4$ + 1.5 g Sodium acetate was added and shaken vigorously by hand (1 min.) well before centrifuge. The tubes were centrifuged at 3500 rpm for 2 minute. A 6.0 mL aliquot from the supernatant was transferred by auto pipette in to a 15 mL polypropylene centrifuge tube containing 0.9 g $MgSO_4$ and 0.3 g PSA, followed by centrifugation at 2500 rpm for 2 minute. A 2.0 mL aliquot was transferred into a glass test tube and acetonitrile was completely evaporated on a Turbo Vap LV. Final volume was made by 3:1; petroleum spirit:acetone and subjected to ECD analysis on a Varian GC 450 and FPD analysis on a Thermo Trace Ultra GC.

3.6.4 Extraction of Pesticide residues from cotton lint

Method of analysis

Solvents and Regents: Petroleum spirit, Acetone

Apparatus: Weighing Balance, Rotary vacuum evaporator and Turbo Vap LV

Instrument: Varian GC 450 with TSD/ ECD and Thermo Trace Ultra GC with FPD.

Extraction of cotton lint sample (AOAC Method)

A 20 g cotton lint sample was weighed in a wide mouth bottle; 200 mL of mixture of acetone: pet- spirit (1:1, v/v) was added and kept for overnight at room temperature. It was filtrated and collected in 250 mL capacity conical flask. The filtrate was concentrated on vacuum rotary evaporator at 45°C. The residues were transferred with the repetitive washings mixture of pet-spirit: acetone (1:1, v/v). Finally the volume was reconstituted to 5.0 mL with pet-spirit: acetone (1:1, v/v) after complete evaporation on TurboVap and subjected to ECD analysis on a Varian GC 450 and FPD analysis on a Thermo Trace Ultra GC for quantitation.

3.7 GLC/GC-MS parameters

3.7.1 GLC parameters of Varian GC-450

GLC	:	Varian 450-GC TSD-ECD
Auto sampler	:	Varian CP-8400
Column	:	GsBP 5 MS, 25 m, 0.32 mm id, 0.25µm FT
Detector	:	ECD- Ni^{63}
Carrier gas	:	Nitrogen
Oven programming	:	160 ºC (0.0 min) $\xrightarrow{7^{\circ}C/min}$ 250 ºC (1.0 min) $\xrightarrow{5^{\circ}C/min}$ 290 ºC (3.0 min)
Column flow mode	:	Constant Flow
Column flow	:	1.5 mL min^{-1}
Injection mode	:	Split
Split ratio	:	1:5
Injection volume	:	1.0 µL
Injector temperature	:	250 ºC

Detector temperature	:	335 ºC
Make up gas and Flow	:	N_2 and 30 mL min^{-1}

3.7.2 GLC parameters of Thermo Trace Ultra GC

GLC	:	Thermo Trace Ultra GC
Auto sampler	:	AS-3000
Column	:	GsBP-5MS, 30 m, 0.25 mm id, 0.25µm FT
Detector	:	FPD
Carrier gas	:	Nitrogen
Oven programming	:	120 ºC (0.0 min) $\xrightarrow{5^{o}C/min}$ 200 ºC (3.0 min) $\xrightarrow{7^{o}C/min}$ 290 ºC (2.14 min)
Column flow mode	:	Constant Flow
Column flow	:	1.0 mL min^{-1}
Injection mode	:	Splitless
Injection volume	:	1.0 µL
Injector temperature	:	250 ºC
Detector temperature	:	300 ºC
FPD temperature	:	150 ºC
PMT voltage	:	Low
Make up gas and Flow	:	N_2 and 30 mL min^{-1}
H_2	:	90 mL min^{-1}
Air	:	115 mL min^{-1}
Retention Time	:	31.865 min

3.7.3 GC-MS parameters of Shimadzu GC-MS QP2010 Plus

GC-MS	:	Shimadzu GC-MS QP 2010
Auto sampler	:	AOC 20*i*
Column	:	GsBP-5 ms, 30 m, 0.25 mm id, 0.25µm FT
Carrier gas	:	Helium
Oven programming	:	100 ºC (0.5 min) $\xrightarrow{5.0^{o}C/min}$ 290 ºC (5.0 min)
Column flow mode	:	Constant Flow
Column flow	:	1.5 mL min^{-1}
Injection mode	:	Split
Split ratio	:	1:5
Injection volume	:	1.0 µL
Injector temperature	:	250 ºC
Ion Source temperature	:	250 ºC
Interface temperature	:	290 ºC
Solvent Delay Time	:	5.0 min
Scan Mode	:	TIC 50m/z to 450m/z

3.7.4 Quantitative analysis

Quantitative estimation of pesticide was performed on Varian GC equipped with TSD/ECD and Thermo Trace Ultra GC equipped with FPD. Quantitation was carried out by external standard method using different concentration of working standard of pesticides.

$$\text{Pesticide Residues } (\mu g\ g^{-1}) = \frac{A_1}{A_2} \times \frac{V}{W} \times C$$

Where,

A_1 = Peak area/height of sample

A_2 = Peak area/height of standard

V = Volume of sample extract

W = Weight of soil sample for extraction

C = Concentration of pesticides ($\mu g\ mL^{-1}$) standard

3.7.5 Method validation

Validation of method was performed in terms of recovery studies before the analysis of unknown sample. The recovery study was carried out at LOQ and 5X LOQ.

Table: 3.2 Scope of the micronutrient and heavy metal for water, soil, vegetable and cotton lint samples

Class	Elements
Micronutrient	Zinc (Zn),Manganese (Mn), Iron (Fe), Copper (Cu)
Heavy metal	Nickel (Ni), Cobalt (Co), Lead (Pb), Cadmium (Cd), Chromium (Cr)

3.8 Micronutrient and heavy metal

3.8.1 Determination of micronutrient and heavy metal from water

Method of analysis

Solvents and Regents: 6N HCl, Deionzed water

Apparatus: Polypropylene bottle, Measuring cylinder, Mechanical shaker, Whatman filter paper No. 42

Instrument: Atomic Absorption Spectrophotometer

A 250 mL water sample was evaporated to near dryness in either a hot air oven or a low heat hot plate. The residue was dissolved in 6N HCl (5 mL) and 10 mL volume was made up with deionzed water. The resultant solution was used for analysis on AAS (Lindsay and Norvell, 1978).

3.8.2 Determination of micronutrient and heavy metal from soil

Method of analysis

Solvents and Regents: DTPA extraction solution

Apparatus: Polypropylene bottle, Measuring cylinder, Mechanical shaker, Whatman filter paper No. 42

Instrument: Atomic Absorption Spectrophotometer

A 10 g air dried soil sample was weighed in a 150 mL capacity conical flask or polypropylene bottle and then 20 mL the DTPA extracting solution was added. The bottle was shaken for two hours with a speed of 120 cycles per minute and the suspension filtered through Whatman No 42 filter paper. The filtrate was kept in polypropylene bottles and analysed with an AAS (Lindsay and Norvell, 1978).

3.8.3 Determination of micronutrient and heavy metal from plant (vegetable and cotton lint)

Method of analysis

Solvents and Regents: HNO_3, Diacid (HNO_3 and $HClO_4$),

Apparatus: Conical flask,Polypropylene bottle, Hot plate, Whatman filter paper No. 42

Instrument: Atomic Absorption Spectrophotometer

One gramme plant sample was weighed in 150 mL capacity conical flask. Ten millilitre concentrated HNO_3 was added and a funnel was placed on the flask and kept for about 6-8 hrs or overnight at covered place/ chamber for pre digestion. After pre-digestion when the solid sample was no more visible, conc. 10 mL HNO_3 and 2-3 mL $HClO_4$ were added and kept on a hot plate in acid- proof digestion chamber having fume exhaust system and heated at about 100°C for first hour and then raised the temperature to about 200°C. Digestion was continued until the contents became colourless and only white dense fume appeared. The acid contents were reduced to about 2-3 mL by continuous heating at the same temperature. The flask was removed from hot plate, cooled it and about 30 mL distilled water was added. The contents were filtrated through Whatman No. 42 filter paper in to a 100 mL capacity volumetric flask by repetitive washing of distilled water and made the final volume to 100 mL. This solution was used for analysis of heavy metal on AAS.

3.9 Nitrate-nitrogen and phosphorus

3.9.1 Determination of nitrate-nitrogen from water

Method of analysis

Solvents and Regents: Phenol disulphonic acid, Calcium hydroxide $Ca(OH)_2$, 6N NH_4OH

Apparatus: Volumetric flask, Pipette, Porceline dish, Glass rod, measuring cylinder

Instrument: Spectrophotometer, Steam bath

A 25 mL water sample was taken in a porcelain dish; a pinch of $Ca(OH)_2$ was added. The porcelain dish was kept on the steam bath for evaporation up to dryness. After evaporating dish was allowed to cool. Phenol disulphonic acid (2 mL) was added and the dish was rotated. The reagent was allowed to act for 10 minutes. Then 15 mL cold distilled water was added and the solution was stirred with glass rod. After few minutes 6 N NH_4OH was added, slowly until the solution became alkaline as indicted by development of a yellow colour and again 6 N NH_4OH (3 mL) was added. The contents were cooled and transferred quantitatively to a 100 mL capacity volumetric flask. The intensity of the yellow colour was measured at 430 nm (blue filter) with spectrophotometer (Jaiswal, 2008).

3.9.2 Determination of phosphorus from water samples

Method of Analysis

Solvents and Regents: Ammonium molybdate solution, Freshly prepared $SnCl_2$

Apparatus: Volumetric flask, Pipette

Instrument: Spectrophotometer

A 10 mL water sample was taken into 50 mL capacity volumetric flask and 10 mL ammonium molybdate solution was added and shaken it well. Freshly prepared 4 mL of working $SnCl_2$ solution was added and volume was made up to 25 mL with distilled water. The transmittance was measured at 660 nm wavelength using spectrophotometer (Olsen's method; Jackson, 1973).

3.10 Organic Carbon from Soil

Method of analysis

Solvents and Regents: 1N Potassium dichromate solution, Conc. Sulphuric acid

Apparatus: Beaker, measuring cylinder,

Instrument: Spectrophotometer

One gramme soil sample was weighed into 100 mL beaker, and10 mL 1N $K_2Cr_2O_7$ and 20 mL conc.H_2SO_4 were added to it was kept overnight and supernant was taken in a conical flask and intensity was read of colour at 660 nm wavelength.

3.11 EC (Electrical conductivity) and pH

3.11.1 Determination of EC

Method of analysis

Solvents and Regents: 0.01 N Potassium chloride solution

Apparatus: Beaker, glass rod

Instrument: Conductivity meter

A 20 g soil was weighed in 250 mL beaker and 50 mL distilled water was added to it. The contents were stirred. The 5 mL was allowed to settle down and conductivity was measured with the help of conductivity meter (Jackson, 1973).

3.11.2 Determination of pH

Method of analysis

Solvents and Regents: Buffer solution of 4.0, 7.0 and 9.2

Apparatus: Beaker, glass rod

Instrument: pH meter

A 20 g soil sample was weighed in 250 mL beaker, 50 mL distilled water was added and contents, stirred for five minutes and kept for half an hour. Again stirred before immersing the electrodes readings were taken on pH meter (Jackson, 1973).

3.11.3 Statistical Analysis

The simple statistical analysis was carried out in the Microsoft Excel programme with the help of computer. The average, S.D., regression equation, R^2 value and µg g^{-1} (ppm) were calculated in excel software (Hoskins, 1961).

4.2 Water

4.2.1 Pesticide residues

4.2.1.1 Pesticide residues in ground water

Forty one samples of ground water were collected from tube wells of vegetable and cotton growing areas of middle Gujarat and analyzed for pesticide residues. None of the water sample showed pesticides above reporting level of 0.1 ppb. The results are presented in Table 4.43.

Although in present study ground water samples were not contaminated with pesticides, Kumari Beena *et al.* (2008) reported that ground water samples showed the presences of HCH, DDT, endosulfan and cypermethrin residues. About 80 per cent samples of the tube well water used for drinking purpose were found to contain residues above the regulatory limits. However, it is unlikely that pesticides present in soil can leach down the ground to the depth of 60 feet or more, the average depth of the tube-well in the region. Pesticides and/ or their metabolites having higher water solubility could leach down the ground but the reported pesticides are almost non-polar. In fact the authors have reported only GLC results without confirmation by mass spectroscopy.

The results obtained in the present study show that it is unlikely that ground water is contaminated with the pesticides used in cotton and vegetable growing areas.

Table: 4.43 Pesticide residues in ground water

Sr. No.	Sample Id	Pesticide Detected	ppb	Sr. No.	Sample Id	Pesticide Detected	ppb
1	Devaliya	Nil	BRL	22	Handod	Nil	BRL
2	Agas Station, Anand	Nil	BRL	23	Handod-1	Nil	BRL
3	Hingalla	Nil	BRL	24	Handod-2	Nil	BRL
4	Padaria	Nil	BRL	25	Ghora-1	Nil	BRL
5	Umara	Nil	BRL	26	Ghora-2	Nil	BRL
6	Galogamdi	Nil	BRL	27	Ghora-3	Nil	BRL
7	Rajupura	Nil	BRL	28	Jantral	Nil	BRL
8	Ode-2	Nil	BRL	29	Jahagirpura, Anand	Nil	BRL
9	Ode-4	Nil	BRL	30	Sarbhan	Nil	BRL
10	Mahli	Nil	BRL	31	Ranu-1	Nil	BRL
11	Surasamal	Nil	BRL	32	Ranu-2	Nil	BRL
12	Pipalsat	Nil	BRL	33	Khandha Farm	Nil	BRL
13	Gulabpura	Nil	BRL	34	Nadiad-1	Nil	BRL
14	Jadavpura, Chaklashi-1	Nil	BRL	35	Nadiad-2	Nil	BRL
15	Jadavpura, Chaklashi-2	Nil	BRL	36	Latipura-1	Nil	BRL
16	Jadavpura, Chaklashi-3	Nil	BRL	37	Anandi-A	Nil	BRL
17	Jadavpura, Chaklashi-5	Nil	BRL	38	Anandi-B	Nil	BRL
18	Radod-T.W.	Nil	BRL	39	Sahera	Nil	BRL
19	Virod	Nil	BRL	40	Bhoj	Nil	BRL
20	Atladara	Nil	BRL	41	Dabhasa	Nil	BRL
21	Sombhoi	Nil	BRL				

BRL- Below Reporting Level = 0.1 μg L^{-1}

4.2.1.2 Pesticide residues in surface water

Ten surface water samples were collected from the ponds of cotton and vegetable growing areas and analysed for pesticide residues. Pesticides were not detected above reporting limit of 0.1 ppb in any of the sample. The results are presented in Table 4.44.

Table: 4.44 Pesticide residues in surface water

Sr. No.	Village	Pesticide Detected	ppb
1	Saran	Nil	BRL
2	Chitral	Nil	BRL
3	Dhanora	Nil	BRL
4	Rarod W.R.	Nil	BRL
5	Chametha	Nil	BRL
6	Alwa	Nil	BRL
7	Koteshwer	Nil	BRL
8	Vav	Nil	BRL
9	Thuvavi	Nil	BRL
10	Kandha Pond	Nil	BRL

BRL-Below Reporting Level=0.1 $\mu g\ L^{-1}$

Contrary to the present findings, Ahad *et al*. (2006) reported the residues of methyl parathion, fenitrothion, azinphosmethyl and α-cypermethrin residues in water samples and found that pyrethroid pesticide residues were present in higher concentration.

Though, many workers have reported higher concentration of pesticides in the ground water as well as surface water, the findings need to be reconfirmed as results without confirmation on Mass Spectrometer (GC-MS/MS and /or LC-MS/MS) should not be reported as there is every possibility of misidentifiying impurities as pesticide. In the present study all the water samples were analysed by GLC instruments followed by verification through GC-MS/MS and/or LC-MS/MS.

4.2.2 Micronutrient and heavy metal content

4.2.2.1 Micronutrient contents in ground water

Forty one water samples collected from tube wells were analysed to rate the status of micronutrients in ground water (ppm). The micronutrients like copper, iron, manganese and zinc were analysed by AAS. The results are shown in Table 4.45. The Cu content in the ground water samples ranged from 0.002 to 0.024 ppm. In some ground water samples collected from the regions like Handod, Pipalsat, Gora-1 and Jahangirpura, only traces of Cu was recorded. The highest concentration of Cu of 0.024 ppm was recorded in the ground water sample collected from Padaria region. As per the BIS standard the safe limit of Cu in drinking water is 0.05 ppm. It is evident from the results that ground water samples contained Cu within the safe limit.

Similarly, the Fe content in the ground water samples ranged from 0.044-5.924 ppm. The highest Fe content (5.924 ppm) was in the sample collected from Padaria region & lowest (0.044) from Ranu-5 region. Out of total 41 samples analysed, 37 samples (90.24 %) exceeded the prescribed limit of Fe as per the BIS standard (i.e., 0.3 ppm). The Mn concentration in ground water samples ranged from 0.002-0.082 ppm. The highest content was in the samples collected from Padaria region (0.082 ppm) and lowest from Ghora-3 (0.002 ppm).

The Zn content in ground water ranged from 0.03-0.268 ppm. Of this, the highest zinc content was recorded in Nadiad-2 region (0.268ppm) and the lowest in Jadavpura, Chaklashi-2 (0.03 ppm). As per BIS the permissible limit for Zn in drinking water is 5.0 ppm. This leads to the conclusion that the Zn content in all the ground water samples was below the permissible limit (Fig: 5).

Table: 4.45 Micronutrient and heavy metal contents in ground water

Sr. No	Village	Micronutrient content (ppm)				Heavy metal (ppm)				
		Cu	Fe	Mn	Zn	Cd	Co	Cr	Ni	Pb
1	Devaliya	0.012	1.862	0.026	0.046	0.002	0.024	0.216	0.046	0.022
2	Agas Station, Anand	0.020	2.232	0.06	0.084	0.004	0.038	0.378	0.078	0.010
3	Hingalla	0.012	1.240	0.032	0.054	0.032	0.080	0.370	0.116	0.088
4	Padaria	0.024	5.924	0.082	0.136	0.004	0.038	0.426	0.066	0.006
5	Umara	0.014	2.894	0.028	0.040	0.006	0.100	0.694	0.164	0.096
6	Galogamdi	0.012	1.216	0.024	0.104	Tr	0.060	0.488	0.082	Tr
7	Rajupura	0.008	2.160	0.032	0.060	Tr	0.028	0.240	0.048	Tr
8	Ode-2	0.006	4.676	0.050	0.038	0.004	0.038	0.426	0.066	0.006
9	Ode-4	0.004	2.104	0.022	0.044	0.012	0.030	0.180	0.046	Tr
10	Surasamal	0.006	5.250	0.054	0.052	0.012	0.042	0.436	0.074	0.012
11	Pipalsat	Tr	0.788	0.006	0.038	Tr	0.024	0.028	0.034	0.074
12	Gulabpura	0.006	0.758	0.010	0.062	Tr	0.022	0.04	0.036	0.056
13	Sarbhan	0.010	1.040	0.028	0.090	0.022	0.092	0.226	0.112	0.082
14	Jadavpura, Chaklashi-1	0.008	2.020	0.030	0.034	0.004	0.046	0.148	0.068	0.004
15	Jadavpura, Chaklashi-2	0.004	0.272	0.014	0.030	0.006	0.022	Tr	0.016	0.026
16	Jadavpura, Chaklashi-3	0.004	0.566	0.008	0.064	0.006	0.024	0.076	0.036	Tr
17	Jadavpura, Chaklashi-5	0.004	0.724	0.024	0.060	0.006	0.080	0.158	0.114	0.070
18	Radod T.W.	0.002	0.764	0.020	0.058	0.004	0.046	0.104	0.072	Tr
19	Virod	0.006	0.684	0.008	0.054	0.006	0.018	Tr	0.028	Tr
20	Atladara	0.010	0.842	0.030	0.078	0.012	0.168	0.384	0.264	0.192
21	Mahli	0.006	0.460	0.010	0.056	0.006	0.034	0.080	0.036	Tr
22	Handod	Tr	2.900	0.032	0.048	Tr	0.044	0.302	0.06	0.07
23	Handod-1	0.006	1.130	0.014	0.048	Tr	0.030	0.080	0.042	Tr
24	Handod-2	0.006	0.482	0.018	0.042	0.002	0.054	0.054	0.070	0.076
25	Dabhasa	0.006	1.072	0.020	0.032	0.004	0.020	0.094	0.032	0.058
26	Ghora-1	Tr	0.322	0.014	0.048	0.004	0.044	0.034	0.050	Tr
27	Ghora-2	0.008	0.486	0.014	0.046	0.010	0.028	0.012	0.038	0.062
28	Ghora-3	0.002	0.088	0.002	0.050	Tr	0.030	0.006	0.040	0.088
29	Jahagirpura	Tr	0.480	0.016	0.050	0.010	0.038	0.120	0.058	0.096
30	Nadiad-1	0.006	0.674	0.016	0.066	0.012	0.030	0.024	0.040	0.012
31	Nadiad-2	0.004	0.520	0.008	0.268	0.016	0.046	0.036	0.062	0.046
32	Ranu-1	0.006	0.044	0.006	0.062	0.008	0.032	0.002	0.034	0.014
33	Ranu-2	0.012	0.914	0.014	0.046	0.016	0.030	0.024	0.048	0.034
34	Jantral	0.006	0.342	0.016	0.048	0.008	0.048	0.050	0.054	0.034
35	Latipura-1	0.010	0.814	0.020	0.052	Tr	0.026	0.018	0.036	Tr
36	Khandha Farm	0.010	0.472	0.012	0.056	Tr	0.040	0.018	0.064	0.020
37	Anandi-A	0.002	0.790	0.010	0.036	0.008	0.028	0.010	0.036	Tr
38	Anandi-B	0.008	3.704	0.020	0.058	0.010	0.026	0.238	0.052	Tr
39	Sahera	0.004	0.146	0.012	0.068	0.008	0.044	0.036	0.052	Tr
40	Bhoj	0.004	0.868	0.016	0.052	Tr	0.052	0.100	0.060	Tr
41	Sombhoi	0.014	0.716	0.008	0.088	0.006	0.034	Tr	0.040	0.066

Tr-Traces

4.2.2.2 Heavy metal contents in ground water

The heavy metal content in 41 ground water samples was analysed and data are presented in Table 4.45. The heavy metals like Cd, Co, Cr, Ni and Pb were analysed by AAS. The Cd content in ground water samples ranged from 0.002-0.032 ppm, wherein the highest Cd content of 0.032 ppm was recorded in the ground water sample collected from Hingalla cotton growing area. Nearly 28.83 per cent of the samples crossed the prescribed limit of 0.01 ppm for Cd by BIS. Similarly Co content of the ground water sample ranged from 0.022-0.168 ppm, where the highest Co content was recorded (0.168 ppm) in the sample collected from Atladara region and the lowest (0.022 ppm) in ground water sample collected from Gulabpura region.

The concentration of Cr in the ground water samples ranged from 0.006-0.488 ppm. The highest Cr content was recorded in the sample collected from Golagamdi region (0.488 ppm) and the lowest in the ground water sample collected from Ghora-3 region (0.006 ppm). Twenty two (53.66 %) ground water samples out 41 samples exceeded the prescribed limit of 0.05 ppm for Cr as per the BIS standard.

The Ni content in the ground water samples ranged from 0.016-0.264 ppm, wherein the highest Ni content was recorded in the ground water sample collected from Atladara region (0.264 ppm) and the least from Jadavpura, Chaklashi-2 regions (0.016 ppm). As per Environment Protection Agency (EPA) the safe limit for Ni is 20 ppm.

The concentration of Pb in the ground water samples ranged from 0.006-0.192 ppm, wherein the highest Pb content was recorded in the sample collected from Vadodara region i.e. Atladara (0.192 ppm). These results revealed that Atladara water sample is contaminated with Pb and unsafe as a potable water, whereas the rest of the water samples are safe for drinking. As per the BIS standard the safe limit of Pb is 0.1ppm.

Ghanem *et al.* (2011) also reported that 85 % in ground water samples collected from Tulkarem and Jenin in North West Bank were polluted with Pb (Fig: 6).

4.2.2.3 Micronutrient contents in surface water

Ten surface water samples were analysed to know the micronutrient status (Cu, Fe, Mn, and Zn) and the results are presented in Table 4.46. The Cu content in the surface water samples ranged from Tr to 0.036 ppm. In some of the surface water samples collected from the regions like Chametha and Dhanora only trace amount of Cu was recorded. The highest concentration of Cu of 0.036 ppm was recorded in the surface water sample collected from Alwa region. As per the BIS standard the safe limit of Cu in drinking water is 0.05 ppm. In the present study none of the surface water samples exceeded the safe limit of Cu.

The Fe content in the surface water samples ranged from 0.528-9.576 ppm. The highest Fe content was in the sample collected from Alwa region & lowest from Saran region. As per BIS the permissible limit for Fe content in drinking water is 0.3 ppm and therefore all the ten surface water samples exceeded the limit in this study.

Similarly, the Mn concentration in surface water samples ranged from 0.010-0.104 ppm. The highest content was in the samples collected from Koteshwer region (0.104 ppm) and lowest from Chametha (0.018 ppm).

The content of Zn (ppm) in surface water samples ranged from 0.018-0.074 ppm in which the highest Zn content was recorded in the surface water samples collected from Thuvavi and Koteshwer (Vadodara district) and the least content was noted in the surface water sample obtained from Dhanora (Vadodara district); the cotton growing area of middle Gujarat. These results reveal that Zn content was much below the prescribed safe limit of 5.0 ppm for drinking water (Fig: 7).

4.2.2.4 Heavy metal contents in surface water

Ten surface water samples were analysed for heavy metals and data are presented in Table 4.46. The Cd content in surface water samples ranged from Tr-0.022 ppm, wherein the highest Cd content of 0.022 ppm was recorded in the surface water sample collected from Rarod V.P. cotton growing area. Nearly 10 per cent of the samples crossed the prescribed limit of 0.01 ppm Cd, as per BIS standard. Therefore samples which contained > 0.01 ppm Cd are unsafe for drinking purpose.

Similarly the surface water sample content of Co was found to be ranging from 0.018-0.102 ppm, wherein the highest Co content was recorded in the sample collected from Rarod V.P. region and the lowest in ground water sample collected from Dhanora region.

The concentration of Cr ranged from Tr-3.256 ppm in surface water samples. The highest Cr content was recorded in the sample collected from Alwa region (3.256 ppm) and the lowest amount of Cr in the surface water sample collected from Saran region (0.006 ppm). Based on BIS standard, the safe limit of Cr for drinking water is 0.05 ppm. The present survey revealed that 80 % of surface water samples exceeding the safe limit of 0.05 ppm.

The Ni content in the surface water samples ranged from 0.028-0.144 ppm wherein the highest Ni content was recorded in the surface water sample collected from Rarod-V.P. region (0.144 ppm) and the least from Dhanora and Chametha regions (0.028 ppm). Though, the levels of Ni and Co in the surface water are quite low, no conclusion could be drawn as no limits are available for these metals.

Table: 4.46 Micronutrient and heavy metal contents in surface water

Sr. No	Village	Micronutrient (ppm)				Heavy metal (ppm)				
		Cu	**Fe**	**Mn**	**Zn**	**Cd**	**Co**	**Cr**	**Ni**	**Pb**
1	Vav	0.012	1.838	0.042	0.072	0.010	0.042	0.152	0.07	0.052
2	Alwa	0.036	9.576	0.098	0.062	Tr	0.046	3.256	0.116	0.032
3	Thuvavi	0.018	2.028	0.034	0.074	0.004	0.052	0.160	0.070	Tr
4	Kanda pond	0.008	4.720	0.052	0.052	0.002	0.03	0.422	0.066	Tr
5	Koteshwar	0.022	5.002	0.104	0.074	0.008	0.058	1.224	0.102	Tr
6	Chametha	Tr	0.906	0.01	0.042	0.008	0.026	0.052	0.028	Tr
7	Rarod- V.P.	0.024	1.252	0.022	0.070	0.022	0.102	0.248	0.144	0.064
8	Dhanora	Tr	0.898	0.026	0.018	Tr	0.018	0.014	0.028	Tr
9	Chitral	0.008	0.680	0.082	0.038	Tr	0.032	0.06	0.042	Tr
10	Saran	0.002	0.528	0.042	0.042	0.006	0.022	Tr	0.028	0.002

Tr-Traces

The concentration of Pb in the surface water samples ranged from Tr-0.064 ppm, wherein the highest Pb content was recorded in the sample collected from Vadodara region i.e., Rarod V.P. (0.064 ppm).

The surface water samples of certain regions like Thuvavi, Kanda pond, Koteshwar, Chametha, Dhanora and Chitral recorded only traces of Pb. Those surface water samples with Pb content greater than 0.1 ppm as per BIS std, are rated as unsafe (Fig: 8).

Schipper *et al.* (2009) studied the leaching of heavy metal in surface water in agricultural soil of Netherland. They reported that the levels of Co and Zn metal were higher in water due to manures and fertilizers use in agricultural soils.

Kar, D *et al*. (2008) reported that 92 % of the water samples collected from river Ganga in West Bengal during 2004-05 were contaminated by micronutrients and heavy metals viz., Fe, Mn, Zn, Cu, Cr, Pb and Ni. They collected samples in different season and the variations in the results were found in the samples. The maximum concentration of Fe was observed in summer, Mn in monsoon and Cd as well as Cr in winter season.

4.2.3 EC and pH of water

4.2.3.1 EC and pH of ground water

The ground water samples collected were analysed for EC and pH (Table 4.47). The EC and pH ranged from 0.53-17.71 and 8.11-9.10, respectively.

The lowest EC was recorded in the ground water samples from Sahera i.e.: 0.53 dSm^{-1} and highest EC in the sample Handod-1 i.e.17.71 dSm^{-1}.The lowest pH was recorded in ground water samples from Altadara i.e.: 8.11 and highest pH in the sample from Mahli i.e.: 9.10.

4.2.3.2 EC and pH of surface water

The surface water samples were analysed for the EC and pH. The EC and pH of surface water samples ranged from 0.361-4.14 dSm^{-1} and 8.28-9.05, respectively. The data are presented in Table 4.48.

The lowest EC was recorded in the surface water samples from Kanda Pond i.e.: 0.361dSm^{-1} and highest EC in the sample from Saran i.e.4.14 dSm^{-1}. The lowest pH was recorded in surface water samples from Chitral i.e.: 8.28 and highest pH in sample from Vav 1 i.e.: 9.05.

There were differences in EC of ground and surface water samples. The EC of the ground water samples was higher than surface water samples. However, differences in pH among ground water and surface water samples were marginal.

Table: 4.47 EC and pH of ground water

Sr. No.	Village	EC (dSm^{-1})	pH	Sr. No.	Village	EC (dSm^{-1})	pH
1	Umara	3.14	9.05	22	Handod-1	2.86	8.3
2	Hingalla	4.40	8.99	23	Handod-2	17.71	8.55
3	Atladara	8.49	8.11	24	Khandha farm	2.57	8.59
4	Surasamal	2.04	8.78	25	Bhoj	10.06	8.29
5	Rarod T.W.	3.61	8.84	26	Mahli	15.65	9.1
6	Sarbhan	4.14	8.95	27	Dabhasa	13.09	8.61
7	Gola gamadi	2.39	8.43	28	Jadavpura, Chaklashi-1	2.03	8.46
8	Jantral	2.72	8.32	29	Jadavpura, Chaklashi-2	10.35	8.52
9	Gulabpura	0.60	8.85	30	Jadavpura, Chaklashi-3	10.20	8.29
10	Virod	1.27	8.75	31	Jadavpura, Chaklashi-5	4.60	8.36
11	Devaliya	0.66	8.21	32	Agas Station, Anand	12.41	8.29
12	Pipalsat	1.08	8.44	33	Ode-2	9.38	8.92
13	Jahagirpura, Anand	17.55	8.44	34	Ode-4	12.36	8.9
14	Padariya	4.65	8.68	35	Ghora-1	0.75	8.58
15	Sombhoi	13.83	8.35	36	Ghora-2	0.76	8.58
16	Latipura-1	13.29	8.95	37	Ghora-3	0.91	8.51
17	Ranu-1	14.30	8.7	38	Nadiad-1	12.25	8.51
18	Ranu-2	16.26	8.76	39	Nadiad-2	4.56	8.28
19	Rajupura	0.90	8.7	40	Anandi-A	1.30	8.97
20	Sahera	0.53	8.43	41	Anandi-B	17.47	8.57
21	Handod	1.05	8.28				

Table: 4.48 EC and pH of surface water

Sr. No.	Village	EC (dSm^{-1})	pH
1	Saran	4.14	8.95
2	Chitral	1.05	8.28
3	Dhanora	0.41	8.56
4	Rarod W.R.	2.72	8.32
5	Chametha	0.52	8.91
6	Alwa	1.45	8.74
7	Koteshwer	1.08	8.44
8	Vav	3.14	9.05
9	Thuvavi	1.23	8.39
10	Kanda Pond	0.361	8.73

4.2.4 Nitrate-nitrogen content in ground water

Forty one ground water samples collected from the intensive agricultural area of middle Gujarat were analyzed for NO_3-N^- content. The data are given in Table 4.49.

The NO_3-N^- content of the ground water samples ranged from 0.018-1.182 ppm. The lowest NO_3-N content was observed in water collected from Khandha farm i.e.: 0.018 ppm and highest NO_3-N^- content was observed in water samples collected from Jantral i.e.: 1.182 ppm of Vadodara district.

The wide variation in the NO_3-N^- concentration in water samples collected from different locations might be due to use of nitrogenous fertilizers in different doses under different cropping pattern in the region. None of this ground water samples exceeded the prescribed limit of 10 ppm nitrate-nitrogen content for drinking water.

Contrary to the present findings, Ghanem *et al*. (2011) reported that of ground water samples collected from was very high mainly due to agricultural activities and the use fertilizers.

Reddy *et al.* (2009) reported the ground water pollution due to nitrate contamination in north eastern part of Anantapur District (A.P.), India. They analysed ground water samples in two seasons i.e. pre-monsoon and post-monsoon. They found that the ground water contamination by nitrate was higher in pre-monsoon samples than post-monsoon.

Sankararamakrishnan *et al.* (2008) surveyed the nitrate- nitrogen and fluoride concentration in shallow and unconfined ground water aquifers of Kanpur district along the Ganges Alluvial Plain of Northern India. They divided Kanpur district into three zones, namely, Bithore, Kanpur city and beyond Jajmau and sampling was carried out in three seasons (summer, monsoon and winter). They found that in Bithore zone, 19% of the samples exceeded the BIS (Bureau of India Standards) limit as nitrate–N, Kanpur city and beyond Jajmau zone, 10% and 7% samples exceeded the BIS limit, respectively. The Frequency distribution histogram of nitrate–N revealed a skewed (non-normal) distribution. Both point and non-point sources contributed to the ground water contamination. Especially in Bithore zone, the point sources could be attributed to the animal wastes derived from cows and buffaloes and non point sources cou ld be due to the extensive agricultural activity prevalent in that area.

Table: 4.49 Nitrate-nitrogen (NO_3-N^-) content in ground water

Sr. No.	Village	NO_3-N^- in ppm	Sr. No.	Village	NO_3-N^- in ppm
1	Sombhoi	0.042	22	Handod-1	0.122
2	Latipura	0.757	23	Handod-2	0.116
3	Ranu-1	0.620	24	Umara	0.514
4	Ranu-2	0.433	25	Devaliya	0.140
5	Rajupura	0.115	26	Anandi	0.665
6	Sahera	0.545	27	Rajpur	0.982
7	Hingalla	0.149	28	Sarbhan	0.151
8	Khandha farm, AAU	0.018	29	Golagamadi	0.356
9	Bhoj	0.846	30	Jantral	1.182
10	Mahli	0.649	31	Gulabpura	0.152
11	Dabhasa	0.736	32	Virod	0.540
12	Jadavpura, Chaklasi-1	0.862	33	Pipalsat	0.020
13	Jadavpura, Chaklasi-2	0.506	34	Atladara	0.319
14	Jadavpura, chaklasi-3	0.497	35	Nadiad-1	1.001
15	Jadavpura, Chaklasi-5	0.997	36	Nadiad-2	0.954
16	Agas station, Anand	0.910	37	Ghora-1	0.559
17	Padaria	0.096	38	Ghora-2	0.592
18	Surasamal	0.450	39	Ghora-3	0.518
19	Ode-2	1.151	40	Rarod T.W.	0.510
20	Ode-4	1.082	41	Jahagirpura,Anand	1.121
21	Handod	0.497			

Tagma *et al*. (2009) reported ground water pollution due to nitrate by agricultural diffusion in Souss-Massa basin, Morocco and they observed that the western and south western parts of the Souss-Massa were highly contaminated by nitrate due to exceeded application of nitrogenous fertilizers for increasing crop yield and economical purpose.

4.2.5 Phosphorous content in surface water

The surface water samples from 10 regions were analysed for phosphorous content. The phosphorous content of surface water ranged from 0.117-0.737 ppm (Table 4.50). As per Minnesota Pollution control agency study (2007) source and forms of phosphorous have impact on Water Quality and observed that the phosphorous concentration from 0.01-0.03 ppm is normal from water quality view point. However in the present study all the 100 per cent surface water samples recorded phosphorous content beyond the normal level. These results suggest that phosphorous accumulation in surface water could be due to run off from the agricultural fields due to high usage of phosphatic fertilizers in the cotton growing soil.

Table: 4.50 Phosphorous content in surface water

Sr. No.	Village	Phosphorous (ppm)
1	Saran	0.652
2	Chitral	0.594
3	Dhanora	0.241
4	Rarod W.R	0.359
5	Chametha	0.117
6	Alwa	0.646
7	Koteshwer	0.684
8	Vav	0.641
9	Thuvavi	0.737
10	Kandha Pond	0.188

4.3 Soil

4.3.1 Pesticide residues

4.3.1.1 Pesticide residues in cotton growing soils

Total 80 soil samples were collected from cotton growing area. All 80 soil samples were analysed for pesticide residues. The results are shown in Table 4.51.

Out of 80 samples analysed for pesticide residues only 5.0 % samples showed residues above detectable limit (Fig: 9). The pesticides detected were *p,p'*- DDE, *p,p'*-DDT, endosulfan sulfate and pendimethalin. The presence of DDT and *p,p'*-DDT and endosulfan is indeed a matter of concern as both the insecticides from organochlorine groups have been banned for agriculture purpose. Nonetheless, detection of pendimethalin- a weedicide is obvious as many farmers are using the chemical as post-emergence for control of weeds.

4.3.1.2 Pesticide residues in vegetable growing soils

All the 22 soil samples were collected from vegetable growing area among them 3 (13.64 %) soil samples were contaminated with pendimethalin (Table 4.52 and Fig. 10). Reporting levels for the soil sample for OCs and chlropyriphos were 0.01 ppm, OPs 0.05 ppm, SPs 0.05 ppm and HBs 0.1 ppm, respectively.

Adsorption of pesticides is the key process in soil because detoxification mechanisms, such as degradation, metabolism, microbial uptake and mobilization, involve only the nonsorbed fraction of molecules. The extent of adsorption depends on various soil properties, including organic matter content, type and amount of clay, cation exchange capacity and pH. Moreover, various physico-chemical parameters of these compounds, such as water solubility and octanol-water partition coefficient, also

Table: 4.51 Pesticide residues in cotton growing soils

Sr. No.	Village	Pesticide detected	ppm	LOD	LOQ	Sr. No.	Village	Pesticide detected	ppm
1	Choranda	Nil	BRL	-	-	23	Napa-4	Nil	BRL
2	Rajupura	Nil	BRL	-	-	24	Napa-5	Nil	BRL
3	Sahera	Nil	BRL	-	-	25	Kheda-1	Nil	BRL
4	Segva	Nil	BRL	-	-	26	Kheda-2	Nil	BRL
5	Handod-1	Nil	BRL	-	-	27	Kheda-3	Nil	BRL
6	Handod-2	Nil	BRL	-	-	28	Kheda-4	Nil	BRL
7	AAU Farm	Nil	BRL	-	-	29	Kheda-5	Nil	BRL
8	Utaraj	Nil	BRL	-	-	30	Vadodara-1	Nil	BRL
9	Dhavat	Nil	BRL	-	-	31	Vadodara-2	Nil	BRL
10	Kuray	Nil	BRL	-	-	32	Vadodara-3	Nil	BRL
11	Kalitalawadi	Nil	BRL	-	-	33	Vadodara-4	Nil	BRL
12	Kurali	*p, p*'-DDE	0.038	0.001	0.002	34	Vadodara-5	Nil	BRL
13	Totarmata	*p, p*'-DDE	0.190	0.001	0.002	35	Vadodara-6	Nil	BRL
		p, p'-DDT	0.0123	0.002	0.006	36	Vadodara-7	Nil	BRL
14	Vemar	Nil	BRL	-	-	37	Vadodara-8	Nil	BRL
15	Anasta	Nil	BRL	-	-	38	Vadodara-9	Nil	BRL
16	Manjrol	Nil	BRL	-	-	39	Vadodara-10	Nil	BRL
17	Pingalwad	Nil	BRL	-	-	40	Vadodara-11	Nil	BRL
18	Ring road	Nil	BRL	-	-	41	Vadodara-12	Nil	BRL
19	Osaram-1	Nil	BRL	-	-	42	Vadodara-13	Nil	BRL
20	Osram-2	Nil	BRL	-	-	43	Vadodara-14	Nil	BRL
21	Sadhli	Nil	BRL	-	-	44	Vadodara-15	Nil	BRL
22	Napa-1	Nil	BRL	-	-	45	Vadodara-16	Nil	BRL

BRL- Below Reporting Limit

Cont……

Table: 4.51 Pesticide residues in cotton growing soils (cont.)

Sr. No.	Village	Pesticide detected	ppm	Sr. No.	Village	Pesticide detected	ppm	LOD	LOQ
46	Napa-2	Nil	BRL	64	Vadodara-17	Nil	BRL	-	-
47	Napa-3	Nil	BRL	65	Vadodara-18	Nil	BRL	-	-
48	Vadodara-19	Nil	BRL	66	Lilod	Nil	BRL		
49	Vadodara-20	Nil	BRL	67	Gavasat	Nil	BRL	-	-
50	Vadodara-21	Nil	BRL	68	Method	Nil	BRL	-	-
51	Vadodara-22	Nil	BRL	69	Bayad	Nil	BRL	-	-
52	Vadodara-23	Nil	BRL	70	Masar-1	Nil	BRL	-	-
53	Vadodara-24	Nil	BRL	71	Masar-2	Nil	BRL	-	-
54	Vadodara-25	Nil	BRL	72	Jambusar-1	Endo SO_4	0.0180	0.042	0.126
55	Vadodara-26	Nil	BRL	73	Jambusar-2	Nil	BRL	-	-
56	Vadodara-27	Nil	BRL	74	Bhoj	Nil	BRL	-	-
57	Vadodara-28	Nil	BRL	75	Karjan	Nil	BRL	-	-
58	Vadodara-29	Nil	BRL	76	Karjan-1	Nil	BRL	-	-
59	Ghor-1	Nil	BRL	77	Karjan-2	Nil	BRL	-	-
60	Ghor-2	Nil	BRL	78	Karjan-3	Nil	BRL	-	-
61	Ghor-3	Nil	BRL	79	Karjan-4	Nil	BRL		
62	Ghor-4	Nil	BRL	80	Sombhoi	Pendimethalin	0.297	0.010	0.030
63	Sinor	Nil	BRL						

BRL- Below Reporting Level

Table: 4.52 Pesticide residues in vegetable growing soils

Sr. No.	Village	Pesticide detected	ppm	LOD	LOQ
1	Mahli	Nil	BRL	-	-
2	Dabhasa	Nil	BRL	-	-
3	Ranu-1	Nil	BRL	-	-
4	Ranu-2	Pendimethalin	0.197	0.010	0.030
5	Latipura-1	Pendimethalin	3.880	0.010	0.030
6	Latipura-2	Pendimethalin	0.112	0.010	0.030
7	Ode-1	Nil	BRL	-	-
8	Ode-2	Nil	BRL	-	-
9	Ode-3	Nil	BRL	-	-
10	Ode-4	Nil	BRL	-	-
11	Ode-5	Nil	BRL	-	-
12	Anandi-A	Nil	BRL	-	-
13	Anandi-B	Nil	BRL	-	-
14	Jadavpura, Chaklashi-1	Nil	BRL	-	-
15	Jadavpura, Chaklashi-2	Nil	BRL	-	-
16	Jadavpura, Chaklashi-3	Nil	BRL	-	-
17	Jadavpura, Chaklashi-4	Nil	BRL	-	-
18	Jadavpura, Chaklashi-5	Nil	BRL	-	-
19	Nadiad-1	Nil	BRL	-	-
20	Nadiad-2	Nil	BRL	-	-
21	Vegetable Res. Project, AAU, Anand	Nil	BRL	-	-
22	Agas Station, Anand	Nil	BRL	-	-

BRL- Below Reporting Level

play an important role in determining adsorption extent (Singh *et al*., 1990; Barriuso *et al*., 1992). Laird *et al*. (1992) stated that clay minerals can strongly adsorb certain aqueous phase organic compounds containing polar functional groups, suggesting the potential contributions of clay minerals to the retention of organic contaminants and pesticides in soils and subsoils.

4.3.2 Micronutrient and heavy metal content

4.3.2.1 Micronutrient contents in cotton growing soils

Samples were analysed to know micronutrient status (ppm) from cotton growing area. The results are given in Table 4.53. The Cu content in the cotton growing soil samples ranged from 0.84-22.90 ppm. The highest concentration of Cu of 22.90 ppm was recorded in the cotton growing sample collected from Sinor region.

Similarly, the Fe content in the cotton growing soil samples ranged from 0.74-30.44 ppm. The highest Fe content was in the sample collected from Kuray region & lowest from Kalitalawadi region. The Mn concentration in soil samples ranged from 3.34-113.34 ppm. The highest content was in the samples collected from Vemar region (113.34 ppm) and lowest from Vadodara-3 (3.34 ppm).

The content of Zn (ppm) in soil samples was found to be varying from Tr-46.4 ppm in which the highest Zn content was recorded in the soil samples collected from Kalitalawadi, Karjan-2 and Vadodara-8 (Vadodara) and the least content was noted in the soil sample obtained from the region Vadodara-12 (Vadodara district); the cotton growing area of middle Gujarat. As per Ewvers (1991) the standard guideline and legislative regulations, the permissible limits of Zn and Cu are 300 and 100 ppm, respectively. Thus all the samples were below prescribed permissible limit for Zn and Cu.

Table: 4.53 Micronutrient and heavy metal contents in cotton growing soils

Sr. No	Village	Micronutrient (ppm)				Heavy metal (ppm)				
		Cu	**Fe**	**Mn**	**Zn**	**Cd**	**Co**	**Cr**	**Ni**	**Pb**
1	Napa-1	2.76	5.02	8.06	6.82	0.08	0.38	0.38	0.66	0.26
2	Napa-2	2.28	5.78	6.76	1.12	0.08	0.36	Tr	0.48	0.62
3	Napa-3	1.38	11.30	4.06	0.72	0.08	0.28	Tr	0.34	0.82
4	Napa-4	2.24	4.00	5.62	0.98	0.08	0.44	Tr	0.56	0.54
5	Napa-5	1.72	4.42	5.98	0.88	0.08	0.28	0.16	0.38	0.86
6	Kheda-1	2.20	3.36	12.58	1.14	0.06	0.30	Tr	0.40	0.44
7	Kheda-2	2.72	1.92	11.42	1.64	0.08	0.36	Tr	0.52	0.86
8	Kheda-3	0.84	5.22	8.82	1.06	0.10	0.28	Tr	0.44	0.68
9	Kheda-4	2.8	9.74	10.40	0.80	0.14	0.34	0.18	0.50	Tr
10	Kheda-5	4.28	7.26	9.74	1.18	0.06	0.42	0.18	0.70	0.04
11	Karjan	1.16	9.22	18.84	0.78	0.20	0.64	0.56	1.16	1.12
12	Karjan-1	3.00	7.46	31.18	1.14	Tr	0.68	0.46	1.14	0.78
13	Karjan-2	2.30	3.42	5.64	1.04	0.18	0.44	Tr	0.54	0.26
14	Karjan-3	2.96	10.34	25.54	22.18	0.06	0.66	1.06	1.36	1.32
15	Karjan-4	1.44	11.20	18.94	1.88	Tr	0.42	0.24	1.06	0.94
16	Vadodara-1	2.28	3.78	7.70	0.60	0.04	0.44	0.24	0.58	0.94
17	Vadodara-2	2.48	3.92	7.36	0.82	0.26	0.38	0.34	0.58	1.62
18	Vadodara-3	1.52	4.76	3.34	0.68	0.08	0.36	0.3	0.56	0.66
19	Vadodara-4	1.72	2.76	5.94	0.80	Tr	0.34	0.54	0.50	0.44
20	Vadodara-5	2.48	2.62	6.36	1.04	Tr	0.36	Tr	0.56	0.18
21	Vadodara-6	3.56	8.02	13.92	1.56	0.14	0.5	0.32	1.10	0.46
22	Vadodara-7	3.30	5.58	6.06	0.62	Tr	0.42	0.08	0.76	0.24
23	Vadodara-8	1.98	4.26	6.36	17.42	Tr	0.46	0.18	0.06	0.70
24	Vadodara-9	3.64	3.28	4.70	0.90	0.04	0.38	0.30	0.70	0.66
25	Vadodara-10	4.02	5.50	4.20	0.78	0.06	0.44	Tr	0.72	0.74
26	Vadodara-11	2.84	3.10	8.92	0.90	0.06	0.42	0.18	0.58	0.46
27	Vadodara-12	2.82	3.2	7.92	Tr	0.04	0.42	0.06	0.58	0.80
28	Vadodara-13	4.78	4.32	5.64	0.98	Tr	0.40	0.14	0.52	0.82
29	Vadodara-14	2.22	2.58	5.96	0.78	Tr	0.44	Tr	0.54	1.62
30	Vadodara-15	3.02	3.64	21.04	1.06	0.20	0.48	Tr	0.82	1.14
31	Vadodara-16	2.38	2.06	8.98	1.22	0.12	0.36	0.12	0.46	0.72
32	Vadodara-17	2.68	2.62	8.2	0.88	0.10	0.44	Tr	0.54	1.84
33	Vadodara-18	2.32	2.36	25.2	0.92	0.16	0.42	0.28	0.78	0.34
34	Vadodara-19	3.46	4.96	50.74	0.84	0.16	0.68	0.44	1.04	0.14
35	Vadodara-20	3.86	6.14	23.98	1.48	0.12	0.54	0.50	0.70	0.84
36	Vadodara-21	3.46	8.90	31.66	0.82	Tr	0.56	0.20	0.82	0.84
37	Vadodara-22	1.76	3.94	13.02	0.60	0.04	0.46	Tr	0.58	0.60
38	Vadodara-23	2.72	11.92	60.02	0.90	0.10	0.60	0.06	1.04	0.30
39	Vadodara-24	4.24	5.88	46.92	0.80	0.12	0.70	Tr	1.16	0.18
40	Vadodara-25	1.94	5.34	37.16	13.16	0.10	0.68	0.40	0.86	1.90

Tr-Traces (Cont….)

Table: 4.53 Micronutrient and heavy metal contents in cotton growing soils (cont.)

Sr. No.	Village	Micronutrient (ppm)				Heavy metal (ppm)				
		Cu	Fe	Mn	Zn	Cd	Co	Cr	Ni	Pb
41	Vadodara-26	2.16	12.02	27.12	1.00	0.04	0.56	Tr	0.70	0.52
42	Vadodara-27	3.06	2.98	28.44	1.16	0.10	0.50	Tr	0.76	0.24
43	Vadodara-28	4.9	8.94	71.26	1.44	0.14	0.96	Tr	1.64	0.30
44	Vadodara-29	4.68	4.78	92.3	2.58	0.10	0.56	0.24	0.82	0.16
45	Ghora-1	2.76	7.70	11.60	7.04	0.16	0.32	Tr	0.86	0.54
46	Ghora-2	1.98	9.32	18.00	1.76	0.12	0.38	Tr	0.86	0.18
47	Ghora-3	3.20	8.86	14.36	2.30	0.06	0.46	Tr	0.86	0.38
48	Ghora-4	2.34	9.52	20.54	1.74	0.06	0.44	Tr	0.76	0.50
49	Kalitalawadi	1.18	0.74	12.26	46.40	0.20	0.50	0.54	0.84	2.06
50	Bayad	2.16	0.92	35.56	1.90	0.08	0.66	0.30	0.70	0.86
51	Kuray	4.80	30.44	35.90	8.32	Tr	0.60	0.52	1.16	1.02
52	Sombhoi	3.00	9.30	10.86	6.54	0.04	0.46	0.60	1.08	0.70
53	Ring road	5.18	12.90	21.82	1.02	0.12	0.58	0.42	1.64	Tr
54	Segva	2.66	20.36	15.92	3.00	0.04	0.64	0.38	1.28	1.34
55	Handod-1	2.98	9.46	8.90	9.10	0.04	0.58	0.26	1.10	1.36
56	Handod-2	2.30	3.36	55.46	1.06	0.06	0.20	0.40	0.94	1.60
57	Bhoj	2.04	8.40	28.72	18.30	0.06	0.50	0.06	1.08	Tr
58	Khandha farm	4.68	12.14	15.80	5.38	Tr	0.52	0.34	1.26	0.96
59	Anasta	2.90	9.22	24.70	2.86	0.08	0.56	0.52	1.08	1.56
60	Dhavat	2.32	12.02	19.24	1.42	0.12	0.6	0.74	1.48	3.04
61	Vemar	4.60	9.40	113.34	1.58	Tr	1.28	0.22	1.32	0.92
62	Utaraj	2.64	15.60	11.60	2.18	Tr	0.54	0.46	1.08	0.12
63	Kurali	4.80	30.44	35.90	8.32	Tr	0.60	0.52	1.16	1.02
64	Manjrol	2.84	10.24	27.08	0.98	0.14	0.62	0.26	1.54	0.98
65	Rajupra	3.06	11.98	32.80	1.02	0.06	0.50	Tr	1.30	1.48
66	Sahera	2.36	8.98	7.34	0.80	0.08	0.46	0.08	0.92	0.90
67	Osram-1	2.98	12.78	29.98	1.14	Tr	0.66	0.30	0.94	1.50
68	Osram-2	3.64	8.54	13.60	1.84	0.16	0.52	0.74	1.52	1.64
69	Jambusar-1	2.98	7.38	39.60	2.96	0.12	0.46	0.12	3.00	2.32
70	Jambusar-2	4.10	8.92	25.60	0.90	0.12	0.50	0.40	0.88	0.22
71	Pingawada	2.24	23.30	12.76	0.92	Tr	0.60	0.76	1.12	2.34
72	Sadhli	2.56	5.00	17.76	0.92	Tr	0.84	0.48	1.12	0.88
73	Lilod	3.20	9.38	38.66	2.02	0.14	0.48	0.22	1.12	1.48
74	Masar-1	2.92	14.48	23.62	1.08	Tr	0.80	0.52	1.28	2.14
75	Masar-2	2.90	13.22	30.82	1.08	Tr	0.66	1.00	1.56	1.32
76	Totarmata	2.92	13.48	44.00	1.72	Tr	0.46	0.20	1.82	1.82
77	Choranda	4.34	16.08	32.08	0.94	Tr	0.58	0.36	1.12	2.88
78	Method	1.78	6.42	15.32	1.82	Tr	0.36	0.34	1.46	0.78
79	Gavasat	1.44	11.20	18.94	1.88	Tr	0.42	0.24	1.06	0.94
80	Sinor	22.90	10.42	53.22	1.22	Tr	0.74	0.34	1.08	1.68

Tr-Traces

4.3.2.2 Heavy metal content in cotton growing soils

The heavy metal concentrations of 80 cotton growing soil samples were analysed and results are presented in Table 4.53. The range of Cd in soil samples ranged from Tr-0.26 ppm. The highest Cd content of 0.26 ppm was recorded in the soil of Vadodara-2 cotton growing area. As per Ewvers (1991) the std. guideline and legislative regulations, the permissible limit for Ni, Cr, Pb and Cd for soil is 50, 50, 100 and 3 ppm, respectively.

Similarly the soil sample content of Co content ranged from 0.2-1.28 ppm, where the highest Co content was recorded in the sample collected from Vemar region and the lowest in soil sample collected from Handod-2 region.

The concentration of Cr in the analysed soil sample ranged from Tr-1.06 ppm. The highest Cr content was recorded in the sample collected from Karjan-2 region (1.06 ppm) and the lowest amount of Cr in the cotton growing soil sample collected from Vadodara-5, Vadodara-15 (Vadodara district), Napa-2, Napa-3 (Anand district) and Kheda-3 (Kheda district) region (Tr).

The Ni content in the cotton growing soil samples ranged from 0.06-3.00 ppm, wherein the highest Ni content was recorded in the soil sample collected from Jambusar-1 region (3.0 ppm) and the least from Vadodara-8 regions (0.06 ppm).

The concentration of Pb in the soil samples ranged from Tr-2.88 ppm, wherein the highest Pb content was recorded in the sample collected from Vadodara region i.e., Choranda (2.88 ppm). The cotton growing soil samples of certain regions like Ring road and Bhoj recorded only trace amount of Pb.

Above finding reveal that all the heavy metals content in cotton growing soil samples were below prescribed limit (Ewvers, 1991).

4.3.2.3 Micronutrient content in vegetable growing soils

Samples were analysed to find out the status of micronutrient in vegetable growing soils. The results are given in Table 4.54. The Cu content in the soil samples ranged from 1.02 to 15.42 ppm. The highest concentration of Cu of 15.42 ppm was recorded in the soil sample collected from Vegetable Research Project, AAU, Anand region. Similarly, the Fe content in the soil samples ranged from 3.56-28.80 ppm. The highest Fe content was in the sample collected from Jadavpura, Chaklashi-1 region & lowest from Ode-2, Anand region. The Mn concentration in vegetable growing soil samples ranged from 4.44-89.76 ppm. The highest content was in the samples collected from Agas Station, Anand region (89.76 ppm) and lowest from Anandi-A (4.44 ppm).

The content of Zn in vegetable growing soils varied from 0.6-22.06 ppm in which the highest Zn content was recorded in the soil samples collected from Latipur-1 and the least content was noted in the soil sample obtained from the Mahli region (Vadodara district). As per Ewvers (1991) the standard guideline and legislative regulations, the permissible limit of Zn and Cu for soil is 300 and 100 ppm, respectively. This reveal the fact that the above mentioned soil samples recorded to have the Zn and Cu contents below the prescribed permissible levels.

4.3.2.4 Heavy metal content in vegetable growing soils

The heavy metal concentration of 22 vegetable growing soil samples were analysed and the results are depicted in Table 4.54. The Cd content in vegetable

Table: 4.54 Micronutrient and heavy metal contents in vegetable growing soils

Sr. No.	Village	Micronutrient (ppm)				Heavy metal (ppm)				
		Cu	**Fe**	**Mn**	**Zn**	**Cd**	**Co**	**Cr**	**Ni**	**Pb**
1	Agas Station, Anand	2.90	16.06	89.76	3.42	0.04	0.64	Tr	1.06	0.50
2	Veg Res Project, AAU	15.42	3.70	5.74	0.76	Tr	0.26	0.48	0.28	Tr
3	Ode-1	3.16	3.70	23.48	2.36	Tr	0.60	1.26	0.92	1.58
4	Ode-2	4.90	3.56	38.30	3.16	0.18	0.84	1.38	1.40	1.76
5	Ode-3	7.02	4.60	53.24	7.10	Tr	0.46	0.82	0.98	0.80
6	Ode-4	2.56	4.24	16.40	2.08	0.12	0.46	0.40	0.64	1.32
7	Ode-5	2.20	4.12	33.68	1.94	0.06	0.6	0.96	1.04	0.22
8	Anandi-A	2.84	6.14	4.44	0.78	Tr	0.38	0.24	0.56	0.42
9	Anandi-B	1.02	4.34	7.18	0.64	0.16	0.28	0.32	0.38	0.30
10	Ranu-1	1.88	11.90	19.44	6.44	Tr	0.6	0.48	1.18	1.26
11	Ranu-2	1.78	7.14	25.30	3.16	0.04	0.46	0.28	0.52	1.22
12	Latipura-1	2.40	11.28	10.30	22.06	0.1	0.32	0.14	0.96	0.18
13	Latipura-2	2.60	10.08	18.06	2.72	0.18	0.44	0.20	1.18	0.94
14	Jadavpura, Chaklashi-1	1.60	4.42	15.32	0.70	Tr	0.44	0.50	1.14	1.16
15	Jadavpura, Chaklashi-2	5.02	28.80	22.40	3.14	0.06	0.36	Tr	0.88	Tr
16	Jadavpura, Chaklashi-3	1.84	7.24	15.30	1.70	0.10	0.50	0.04	0.92	0.34
17	Jadavpura, Chaklashi-4	1.46	4.26	7.08	1.20	0.04	0.22	0.52	0.38	0.70
18	Jadavpura, Chaklashi-5	1.20	14.80	9.60	1.50	Tr	0.30	0.56	0.46	Tr
19	Mahli	1.58	7.36	5.44	0.60	Tr	0.42	0.14	0.84	0.60
20	Dhabhasa	6.92	9.28	16.44	5.22	Tr	0.46	Tr	1.02	0.88
21	Nadiad-1	1.34	17.68	8.64	0.84	0.12	0.28	Tr	0.44	0.86
22	Nadiad-2	1.76	18.56	11.9	2.6	0.58	0.44	Tr	0.22	0.08

Tr-Traces

growing soil samples varied from Tr-0.18 ppm. The highest Cd content of 0.18 ppm was recorded in the soil sample collected from Latipura-2 vegetable growing area.

Similarly Co content ranged from 0.22-0.84 ppm. The highest Co content was recorded in the sample collected from Ode-2 region and the lowest in soil sample collected from Jadavpura, Chklashi-1, Anand region.

The concentration of Cr ranged from Tr-1.38 ppm. The highest Cr content was recorded in the sample collected from Ode-2 region (1.38 ppm) and the lowest amount of Cr in the vegetable growing soil sample collected from Agas Station,Anand, Jadavpura, Chaklashi-1and Jahangirpura, Anand region (Tr).

The Ni content in the soil samples ranged from 0.28-1.40 ppm. The highest Ni content was recorded in the soil sample collected from Ode-2 region (1.40 ppm) and the least from Vegetable Research Project, AAU, Anand regions (0.28 ppm).

The Pb content in the vegetable growing soil samples varied from Tr-1.76 ppm, where the highest Pb content was recorded in the sample collected from Anand region i.e., Ode-2 (1.76 ppm).The vegetable growing soil samples of certain regions like Jadavpura, Chaklashi-1, Vegetable Research Project and Jadavpura, Chaklashi-5 recorded only trace amount of Pb.

Above finding reveal that Ni, Cr, Pb and Cd contents in the entire soil sample were below prescribed permissible limits (Ewvers, 1991).

4.3.3 Organic carbon content

4.3.3.1 Organic carbon content in cotton growing soils

The soil samples collected were analyzed for the organic carbon content. The organic carbon content of soil samples ranged from 0.03-0.95% (Table 4.55).

Table: 4.55 Organic carbon content (%) in cotton growing soils

Sr. No.	Village	% OC	Sr. No.	Village	% OC
1	Pingalwada	0.57	41	Vadodara-3	0.05
2	Kurali	0.2	42	Vadodara-4	0.12
3	Choranda	0.53	43	Vadodara-5	0.44
4	Kuray	0.9	44	Vadodara-6	0.28
5	Sadhli	0.88	45	Vadodara-7	0.29
6	Totarmata	0.04	46	Vadodara-8	0.18
7	Megod	0.65	47	Vadodara-9	0.2
8	Dhavat	0.54	48	Vadodara-10	0.25
9	Utaraj	0.52	49	Vadodara-11	0.31
10	Karjan-1	0.6	50	Vadodara-12	0.27
11	Osaram-1	0.92	51	Napa-5	0.1
12	Masar-1	0.34	52	Vadodara-13	0.03
13	Ringroad	0.71	53	Karjan-1	0.17
14	Kalitalwadi	0.83	54	Vadodara-14	0.16
15	Lilod	0.95	55	Vadodara-15	0.28
16	Osram-2	0.1	56	Vadodara-16	0.27
17	Gavasad	0.12	57	Vadodara-17	0.27
18	Anasta	0.13	58	Vadodara-18	0.38
19	Masar-2	0.68	59	Vadodara-19	0.17
20	Sinor	0.75	60	Vadodara-20	0.37
21	Jambusar-1	0.71	61	Vadodara-21	0.28
22	Jambusar-2	0.23	62	Vadodara-22	0.18
23	Manjarol	0.93	63	Vadodara-23	0.39
24	Vemar	0.15	64	Vadodara-24	0.40
25	Sahera	0.78	65	Vadodara-25	0.53
26	Khandha Farm AAU	0.4	66	Vadodara-26	0.44
27	Segva	0.79	67	Vadodara-27	0.50
28	Handod-1	0.7	68	Vadodara-28	0.39
29	Handod-2	0.73	69	Ghora-1	0.36
30	Rajupura	0.63	70	Ghora-2	0.32
31	Napa-1	0.12	71	Ghora-3	0.6
32	Napa-2	0.28	72	Ghora-4	0.38
33	Napa-3	0.06	73	Karjan-3	0.58
34	Napa-4	0.22	74	Kurali	0.32
35	Kheda-1	0.12	75	Bhoj	0.52
36	Kheda-2	0.11	76	Bayad-1	0.37
37	Kheda-3	0.15	77	Bayad-2	0.80
38	Kheda-4	0.24	78	Karjan-1	0.40
39	Vadodara-1	0.24	79	Kheda-5	0.55
40	Vadodara-2	0.21	80	Vadodara-29	0.32

The highest percentage organic carbon content was recorded in soil samples from Lilod i.e.: 0.95% and the lowest percentage organic carbon was in the sample from Vadodara-13 i.e.: 0.03%.

The 11.25 per cent soil samples had enough organic carbon content as they exceeded the 0.75 per cent limit (Fig: 11). The variation in the soil organic carbon content could be due to the variation in organic manure application by farmer and also due cropping intensity, crop rotation and farming practices as well as texture of soil.

4.3.3.2 Organic carbon content in vegetable growing soils

The percentage O.C content of soil samples ranged from 0.25-0.97% (Table 4.56). The highest percentage of organic carbon content was recorded in soil samples from Latipura-2 i.e.: 0.97% and lowest percentage of organic carbon in the sample of Anandi-B i.e.: 0.25%. Out of 22 soil samples collected from vegetable growing area 27.27 per cent soil samples had enough organic carbon (Fig: 12). The variation in the soil organic content might be due to nutrient management practices in the region and also due to differences in addition of organic manures like FYM, vermicompost, cakes etc.

In the present investigation, sandy loam soil and clayey soil were rich in organic carbon (0.25-0.97% and 0.03-0.97%) content. The distribution of total organic carbon closely followed the distribution of sediment type i.e., sediment low in clay content was low in total organic carbon content and as the clay content increased, the total organic carbon content also increased as reported by Reddy and Hariharan (1986). It seems that amount of organic C plays a dominant role in adsorption of agrochemicals in different soils under consideration. Hence, variability in the contaminant in the different soils might be due to variation in organic carbon content in soil as soil organic carbon act as a natural chelating agent. The nature of organic matter has little influence on the adsorption processes, however its content has great influence (Bailey and White, 1964; Hayes, 1970; Arienzo and Buondonno, 1993; Jenks *et al*., 1998; Bekbolet *et al*., 1999) and also an increase in clay content results in increasing adsorption of a pesticide (Barriuso *et al*., 1992; Murphy *et al*., 1992; Baskaran *et al*., 1996).

Table: 4.56 Organic carbon content (%) in vegetable growing soils

Sr. No.	Village	OC (%)
1	Ranu-1	0.61
2	Ranu-2	0.62
3	Od-1	0.53
4	Od-2	0.8
5	Od-3	0.8
6	Od-4	0.44
7	Od-5	0.81
8	Mahli	0.64
9	Latipura-1	0.96
10	Latipura-2	0.97
11	Anandi-A	0.54
12	Anandi-B	0.25
13	Jadavpura, Chaklshi-1	0.65
14	Jadavpura, Chaklshi-2	0.82
15	Jadavpura, Chaklshi-3	0.69
16	Jadavpura, Chaklshi-4	0.41
17	Jadavpura, Chaklshi-5	0.59
18	Agas Station, Anand	0.33
19	Nadiad-1	0.58
20	Nadiad-2	0.54
21	Veg Res Project, AAU	0.32
22	Dabhasa	0.72

4.3.4 EC and pH of soil

4. 3. 4. 1 EC and pH of cotton growing soils

The EC and pH of soil samples varied from 0.126-2.260 dSm^{-1} and 7.02-8.67, respectively.

The lowest EC was recorded in soil sample Kheda-3 i.e.: 0.126 dSm^{-1} and highest EC in the soil sample Masar-1 i.e.: 2.260 dSm^{-1} (Table 4.57). The highest pH (8.67) was recorded in soil samples with sample Bayad and lowest pH 7.02 in the soil sample Vadodara-2 (Table -4.57).

4.3.4.2 EC and pH of vegetable growing soils

The EC and pH of soil samples ranged from 0.292-2.680 dSm^{-1} and 7.32-8.67, respectively (Table 4.58).

The highest EC was recorded in soil samples from Jadavpura, Chaklashi-5 i.e., 2.680 dSm^{-1} and lowest EC of 0.292 dSm^{-1} in the sample of Ranu-1. The highest pH was recorded in soil sample of Latipura-2 i.e.: 8.67 and lowest pH in the sample of Ode-1 i.e.: 7.32.

In general, EC increased as the clay content was increased. Soil with clay dominated by high cation-exchange capacity (CEC) clay minerals (e.g., smectite) had higher EC than those with clay dominated kaolinite (USDA Natural Resources Conservation service, 2011).

Soils with coarse texture may acidify easily compared to clay soils, because they have low organic matter content, a low buffering capacity, a low CEC (poor cation retention) and high rates of water percolation and infiltration. Clay and organic matter in mineral soils act as buffers to resist pH variations.

Table: 4.57 EC and pH of cotton growing soils

Sr. No.	Village	EC dSm^{-1}	pH	Sr. No.	Village	EC dSm^{-1}	pH
1	Kuray	0.980	7.66	24	Kheda-1	0.392	8.02
2	Dhavat	0.565	7.45	25	Kheda-2	1.125	7.65
3	kurali	0.471	7.31	26	Kheda-3	0.126	7.43
4	Vemar	0.484	6.58	27	Kheda-4	0.265	7.98
5	Lilod	0.664	7.05	28	Kheda-5	0.918	8.16
6	Dhavat	1.184	7.14	29	Vadodara-1	0.878	7.98
7	Utaraj	0.497	7.47	30	Vadodara-2	0.514	7.02
8	Sadhli	0.492	7.89	31	Vadodara-3	0.426	7.45
9	Sinor	0.836	7.15	32	Vadodara-4	0.630	7.59
10	Segva	0.445	7.76	33	Vadodara-5	0.816	7.71
11	Kalitalwadi	0.498	7.54	34	Vadodara-6	0.936	8.02
12	Manjarol	0.924	7.26	35	Vadodara-7	0.735	7.83
13	Totarmata	1.005	8.14	36	Vadodara-8	0.326	7.45
14	Method	0.572	8.05	37	Vadodara-9	0.219	7.13
15	Pingalwad	0.332	8.23	38	Vadodara-10	0.378	7.62
16	Anasta	0.814	7.78	39	Vadodara-11	0.573	7.32
17	Osram-1	0.965	7.71	40	Vadodara-12	0.136	7.15
18	Osram-2	0.545	7.78	41	Vadodara-13	0.628	8.0
19	Choranda	0.526	7.96	42	Vadodara-14	0.759	7.7
20	Masar-1	2.260	7.54	43	Vadodara-15	0.806	8.1
21	Masar-2	0.635	7.71	44	Vadodara-16	0.325	7.46
22	Jambusar-1	1.106	7.33	45	Vadodara-17	0.869	7.89
23	Jambusar-2	1.163	7.49	46	Vadodara-18	0.233	7.13
47	Vadodara-19	0.465	7.26	64	Napa-3	0.445	7.8
48	Vadodara-20	0.835	8.25	65	Napa-4	0.450	7.47
49	Vadodara-21	0.917	7.69	66	Napa-5	0.289	7.06
50	Vadodara-22	1.126	7.63	67	Bayad	0.635	8.67
51	Vadodara-23	0.246	7.77	68	Bhoj	0.635	7.71
52	Vadodara-24	0.572	7.69	69	Rajupura	1.390	7.38
53	Vadodara-25	0.349	7.53	70	Sahera	0.675	7.52
54	Vadodara-26	0.458	7.45	71	Handod-1	0.680	7.79
55	Vadodara-27	0.923	7.87	72	Handod-2	0.428	7.76
56	Vadodara-28	0.723	8.12	73	Karjan	0.292	8.00
57	Vadodara-29	0.226	7.4	74	Karjan-1	0.359	7.75
58	Ghora-1	0.281	7.73	75	Karjan-2	0.625	7.69
59	Ghora-2	0.863	7.61	76	Karjan-3	0.189	7.56
60	Ghora-3	0.915	8.28	77	Sambhoi	0.714	7.42
61	Ghora-4	0.450	7.78	78	Khandha Farm	0.755	7.89
62	Napa-1	0.303	7.64	79	Gavasat	1.845	8.25
63	Napa-2	0.870	7.57	80	Ring road	0.584	7.06

Table: 4.58 EC and pH of vegetable growing soil

Sr. No.	Village	EC (dSm^{-1})	pH
1	Jadavpura, Chaklashi-1	1.169	7.7
2	Jadavpura, Chaklashi-2	0.313	7.61
3	Jadavpura, Chaklashi-3	0.445	7.72
4	Jadavpura, Chaklashi-4	1.414	8.02
5	Jadavpura, Chaklashi-5	2.680	7.71
6	Anandi-A	0.335	8.1
7	Anandi-B	0.470	8.49
8	Nadiad-1	0.820	7.54
9	Nadiad-2	1.228	7.57
10	Ode-1	0.315	7.32
11	Ode-2	0.299	7.38
12	Ode-3	0.306	7.49
13	Ode-4	0.413	7.56
14	Ode-5	0.356	7.77
15	Latipura-1	1.845	8.25
16	Latipura-2	0.635	8.67
17	Ranu-1	0.292	8.01
18	Ranu-2	1.387	7.69
19	Mahli	0.552	7.63
20	Dabhasa	0.347	7.47
21	Veg Res Project, AAU	0.895	7.86
22	Agas Station, Anand	0.425	7.58

Soil parent material influences soil properties including pH as shown by the contrasting pH of soil formed in calcareous and granitic materials (USDA Natural Resources Conservation service, 2011).

4.4 Vegetables

4.4.1 Farmgate vegetables

4.4.1.1 Pesticide residues in farmgate vegetables

Total 21 vegetable samples (brinjal, (5); tomato, (2); capsicum, (5) and okra (9)) were collected from farmers' field were monitored for pesticide residues by gas chromatography mass spectroscopy techniques. The results are presented in Table 4.59.

None of the brinjal sample was contaminated with pesticide residues In okra, out of 9 samples and in capsicum out of five samples one each contained acephate 0.141 ppm and 0.087 ppm, respectively. Out of 21, two vegetable samples (9.52 %) were found to be contaminated with pesticides (Fig: 13). However, none of the pesticides was above the respective MRL. The reporting levels for different pesticides in vegetables were; OCs -0.01 ppm, OPs- 0.05 ppm and SPs and HBs- 0.1 ppm.

Table: 4.59 Pesticide residues in vegetable fruits

Sr. No	Village / Commodity	Pesticide detected	ppm	LOD	LOQ
1	Ranu-1 (tomato)	Nil	BRL	-	-
2	Ranu-2 (okra)	Nil	BRL	-	-
3	Ode-1 (capsicum)	Nil	BRL	-	-
4	Ode-2 (capsicum)	Nil	BRL	-	-
5	Ode-3 (capsicum)	Nil	BRL	-	-
6	Ode-4 (capsicum)	Nil	BRL	-	-
7	Ode-5 (capsicum)	Acephate	0.087	0.003	0.008
8	Anandi- A(tomato)	Nil	BRL	-	-
9	Anandi-B (okra)	Acephate	0.141	0.003	0.008
10	Jadavpura, Chaklashi-1 (okra)	Nil	BRL	-	-
11	Jadavpura, Chaklashi-2 (okra)	Nil	BRL	-	-
12	Jadavpura, Chaklashi-3 (okra)	Nil	BRL	-	-
13	Jadavpura, Chaklashi-4 (okra)	Nil	BRL	-	-
14	Jadavpura, Chaklashi-5 (okra)	Nil	BRL	-	-
15	Agas Station, Anand (okra)	Nil	BRL	-	-
16	Nadiad-1 (okra)	Nil	BRL	-	-
17	Nadiad-2 (brinjal)	Nil	BRL	-	-
18	Jahagirpura, Anand (brinjal)	Nil	BRL	-	-
19	Mahli (brinjal)	Nil	BRL	-	-
20	Dabhasa (brinjal)	Nil	BRL	-	-
21	Latipura-1(brinjal)	Nil	BRL	-	-

BRL-Below Reporting Level

Swarnam and Velmurugan (2013) studied the presence of pesticide residues in brinjal, okra, green chillies, crucifers and cucurbits collected from farmers' field. They found that 34% samples were contaminated by pesticides. Among them 14.5% samples were contaminated by organochlorine group of pesticides compounds, 32% samples with synthetic pyrethroids compounds and 54% samples with organophosphate group of pesticides. Among them, 15.3% samples were found to contain residues exceeding the prescribed MRL. Multiple residues of more than one compound were detected in 34.1% of samples.

Osman *et al.* (2009) studied pesticide residues in vegetable collected from four major big supermarkets located in Al-Qassim region, Saudi Arabia. They reported that residues were found in 89 of the 160 samples and 53 samples were above the MRLs.

Anwar (2009) conducted survey and reported the status of pesticide residues in vegetable samples in cotton growing areas of Sindh and lower Punjab, Pakistan and found 37 % contamination.

Bhanti and Taneja (2007) studied the residual effect of pesticide in vegetable sample grown in different seasons. They reported that vegetables grown in winter were the most contaminated than the one grown in summer followed by rainy season. The concentration of various pesticides was below the established tolerance level but continuous consumption of such vegetables even with moderate contamination level could accumulate in the human body.

Hassan *et al*. (2007) analysed 124 vegetable samples for pesticide residues of 7 commonly used pesticides. They reported that 89 samples were found contaminated, out of which 47 samples exceeded prescribed MRLs.

4.4.1.2 Micronutrient and heavy metal content

4.4.1.2.1 Micronutrient content in farmgate vegetables

Samples were analysed to know the status of micronutrients in vegetables. The Cu content in the vegetable samples varied from 5.5 to 16.0 ppm (Table 4.60). The highest concentration of Cu of 16.0 ppm was recorded in the vegetable sample collected from Chaklashi, Nadiad-1 (brinjal) region. So it is evident that the above analysed fruit vegetable samples contained Cu within the safe limit of 73.3 ppm prescribed by Codex.

Similarly, the Fe content in the vegetable samples ranged from 89.5-295.5 ppm. The highest iron content was in the sample collected from Ode-3 (capsicum) region & lowest from Latipura-1 (brinjal) region. The Mn concentration in vegetable samples ranged from 11.0-44.0 ppm. The highest content was in the samples collected from Anandi-A (tomato) (Anand district) region (44.0 ppm) and lowest from Latipura-1 (brinjal) (11.0 ppm); (Vadodara District).

The Zn content in vegetable samples varied from 14.0-53.5 ppm in which the highest Zn content was recorded in the vegetable samples collected from Anadi-A (tomato) (Anand district) and the least content was noted in the vegetable sample obtained from the region Latipura-1 (brinjal) (Vadodara district); the vegetable growing area of middle Gujarat. These results show that vegetable samples containing more than 9.4 ppm Zn. The permissible limit of Zn and Cu are 9.4 and 73.3 ppm, respectively (Codex Stan 193, 1995) (Fig: 14).

Table: 4.60 Micronutrient and heavy metal contents in farmgate vegetables

Sr. No.	Village Name/ Commodity	Micronutrient content (ppm)				Heavy metal content(ppm)				
		Cu	Fe	Mn	Zn	Cd	Co	Cr	Ni	Pb
1	Anandi-A (tomato)	13.5	193.0	44.0	53.5	2.0	3.0	6.5	7.5	Tr
2	Anandi-B (okra)	13.5	261.0	38.0	38.5	2.0	3.5	5.5	8.0	Tr
3	Nadiad-1 (okra)	11.5	95.5	19.0	18.5	1.0	0.5	5.0	6.5	3.0
4	Nadiad-2 (brinjal)	10.0	120.0	28.0	38.5	Tr	2.5	4.0	6.0	Tr
5	Jahangirpura, Anand (brinjal)	9.0	235.0	15.5	18.0	Tr	1.5	6.5	4.5	Tr
6	Jadhavpura, Chaklashi-1(okra)	7.5	109.5	24.0	23.5	0.5	1.5	3.5	6.5	4.0
7	Jadhavpura, Chaklashi-2 (okra)	10.5	113.0	25.0	29.5	1.0	2.0	3.5	6.5	Tr
8	Jadhavpura, Chaklashi-3 (okra)	11.5	118.0	25.0	33.0	Tr	2.5	3.5	6.5	14.0
9	Jadhavpura, Chaklashi-4 (okra)	8.5	112.0	25.5	31.0	0.5	3.0	10.5	4.5	Tr
10	Jadhavpura, Chaklashi-5 (okra)	16.0	202.0	22.5	52.5	Tr	2.0	5.0	6.5	5.5
11	Agas station, Anand (okra)	9.5	243.0	24.0	31.0	Tr	3.0	2.5	8.0	Tr
12	Ode-1 (capsicum)	9.5	96.5	21.0	22.0	Tr	1.0	Tr	8.5	4.0
13	Ode-2 (capsicum)	10.0	263.5	14.0	15.0	Tr	2.0	2.0	5.5	Tr
14	Ode-3 (capsicum)	9.0	295.5	14.5	22.5	2.0	1.5	Tr	3.5	Tr
15	Ode-4 (capsicum)	5.5	246.5	13.5	17.5	1.5	2.0	Tr	8.5	5
16	Ode-5 (capsicum)	7.5	240.0	17.0	17.5	0.5	1.0	8.0	0.6	Tr
17	Ranu-1(tomato)	11.0	138.0	18.5	18.0	Tr	1.5	Tr	6.5	18.5
18	Ranu-2 (okra)	12.0	208.0	30.0	18.5	Tr	0.5	6.0	6.0	Tr
19	Mahli (brinjal)	9.5	103.5	15.0	15.5	0.5	Tr	Tr	6.5	Tr
20	Latipura-1 (brinjal)	7.5	89.5	11.0	14.0	Tr	Tr	6.0	3.0	11.0
21	Dabhasa (brinjal)	10.0	112.0	12.5	17.0	1.0	0.5	3.0	4.0	Tr

Tr-Traces

4.4.1.2.2 Heavy metal content in farmgate vegetables

The heavy metal concentration of 21 vegetable samples were analysed and results are given in Table 4.60. The Cd content in fruit vegetable samples ranged from Tr-2.0 ppm. The highest Cd content of 2.0 ppm was recorded in the vegetable sample collected from Anandi-A (tomato), Anandi-B (okra) and Ode-3 (capsicum).

Similarly Co content varied from Tr- 3.5 ppm. The highest Co content was recorded in the samples collected from Anandi-B (okra) region and the lowest in fruit vegetable samples collected from Mahli (brinjal) and Latipura-1 (brinjal) region. The concentration of Cr ranged from Tr-10.5 ppm. The highest Cr content was recorded in samples collected from Jadavpura, Chaklashi-4 (okra) region (10.5 ppm) and the lowest amount of Cr in vegetable samples collected from Mahli (brinjal), Ranu-1 (tomato), Ode-4 (capsicum), Ode-3 (capsicum)and Ode-1 (capsicum) region (Tr).

The Ni content in the fruit vegetable samples ranged from 0.6-8.5 ppm. The highest Ni content was recorded in the vegetable sample collected from Ode-1 (capsicum) and Ode-4 (capsicum) (8.5 ppm) and the least from Ode-5 (capsicum) regions (0.6 ppm).

The concentration of Pb in the vegetable samples ranged from Tr-18.5 ppm. The highest Pb content was recorded in the sample collected from Vadodara region i.e., Ranu-1 (tomato) (18.5 ppm). The vegetable samples of certain regions like Anandi-A (tomato) and B (okra), Ode-2 (capsicum), Ode-3 (capsicum) and Ode-5 (capsicum), Jadavpura, Chaklashi-4 (okra) and Jadavpura, Chaklashi-1 (okra), Agas station Anand (okra), Jahangorpura Anand (brinjal), Nadiad-2 (brinjal), Ranu-2 (okra), Mahli (brinjal) and Dabhasa (brinjal) recorded only trace amount of Pb.

Among different farmgate vegetables 15, 8 and 11 samples (Fig: 15) exceeded prescribed permissible limit (Codex Stan 193, 1995) for Cr, Pb and Cd which are 2.3 ppm, 0.1 ppm and 0.05 ppm, respectively.

Similarly, Inoti *et al*. (2012) also reported that the mean concentrations of Pb, Zn and Cd in all the samples were more than the maximum permitted concentrations, while there was no evidence of Cu contamination.

4.5 Cotton lint

4.5.1 Pesticide residues in cotton lint samples

A total of 29 cotton lint samples were analyzed for the pesticide residues. Among them 10.34 % cotton lint samples were contaminated with pesticide residues. Cotton lint samples were contaminated with chlorpyriphos (0.011 and 0.015 ppm) and quinalphos (0.054 ppm) (Table 4.61 and Fig: 16). The reporting level for cotton lint samples are OCs & chlorpyriphos-0.01 ppm, OPs, SPs & HBs-0.05 ppm, respectively.

Similarly, Diwan *et al*. (2006) also studied that the residues of different insecticides in cotton lint and cotton seed collected from cotton growing areas of middle Gujarat. They reported that 95.83 % cotton lint samples were contaminated with endosulfan sulfate, 16.67 % with λ-cyhalothrin, 33.33 % with chlorpyriphos, 20.83 % with quinalphos and 8.33 % with triazophos, profenophos & cypermethrin, respectively.

Table: 4.61 Pesticide residues in cotton lint samples

Sr. No.	Village	Pesticide detected	ppm	LOD	LOQ
1	Sombhoi	Nil	BRL	-	-
2	Method	Nil	BRL	-	-
3	Rajupura	Nil	BRL	-	
4	Anasta	Nil	BRL	-	-
5	Ghora-1	Nil	BRL	-	-
6	Ghora-2	Nil	BRL	-	-
7	Ghora-4	Chlorpyriphos	0.011	0.01	0.04
8	Segva	Nil	BRL	-	-
9	Pingalwala	Nil	BRL	-	-
10	Near khandha farm	Nil	BRL	-	-
11	Utraj	Nil	BRL	-	-
12	Ringroad	Nil	BRL	-	-
13	Handod-1	Nil	BRL	-	-
14	Handod-2	Nil	BRL	-	-
15	Totarmata	Nil	BRL	-	-
16	Sinor	Nil	BRL	-	-
17	Bhoj	Nil	BRL	-	-
18	Kaditalavadi	Nil	BRL	-	-
19	Lilod	Nil	BRL	-	-
20	Kuray	Nil	BRL	-	-
21	Osaram-1	Nil	BRL	-	-
22	Osaram-2	Quinalphos	0.054	0.01	0.04
23	Sahera-10	Chlorpyriphos	0.015	0.01	0.04
24	Sadhli	Nil	BRL	-	-
25	Verma	Nil	BRL	-	-
26	Majrol	Nil	BRL	-	-
27	Khandha farm,AAU	Nil	BRL	-	-
28	Dhavat	Nil	BRL	-	-
29	Kurali	Nil	BRL	-	-

BRL- Below Reporting Level

4.5.2 Micronutrient and heavy metal content

4.5.2.1 Micronutrient contents in cotton lint

Samples were analysed to know the micronutrient levels in cotton lint (Table 4.62). The Cu content in cotton lint samples ranged from 1.5 to 4.0 ppm. The highest concentration of Cu of 4.0 ppm was recorded in the cotton lint samples collected from Rajupura, Sinor regions.

Similarly, the Fe content in cotton lint samples ranged from 46.0-144.5 ppm. The highest Fe content was in sample collected from Ghora-2 region & lowest from Anasta, Totarmata and Osaram-1 region.

The Mn concentration in cotton lint samples ranged from 5.0-10.5 ppm. The highest content was in the sample collected from Bhoj region (10.5 ppm) and lowest from Kurali (5.0 ppm); (Vadodara District).

The Zn content in cotton lint samples varied from 2.5-7.5 ppm. The highest Zn content was recorded in the cotton lint sample collected from Handod-1 and Sahera (Vadodara discrtict) and the least content was noted in the cotton lint sample obtained from the region Manjarol (Vadodara district) the cotton growing area of middle Gujarat.

4.5.2.2 Heavy metal content in cotton lint sample

The heavy metal concentration of 29 cotton lint samples was analysed and data are given in Table 4.62. The Cd content in cotton lint samples ranged from Tr-2.0 ppm. The highest Cd content of 2.0 ppm was recorded in the cotton lint samples collected from Kalitalawadi and Sadhli cotton growing areas.

Table: 4.62 Micronutrient and heavy metal contents in cotton lint

Sr. No.	Village	Micronutrient content (ppm)				Heavy metal content (ppm)				
		Cu	**Fe**	**Mn**	**Zn**	**Cd**	**Co**	**Cr**	**Ni**	**Pb**
1	Sombhoi	3.0	78.0	6.5	7.0	0.5	0.5	2.0	2.0	Tr
2	Totarmata	1.5	54.5	7.0	4.0	0.5	0.5	5.5	2.0	Tr
3	Method	3.0	70.0	7.0	5.5	Tr	0.5	2.0	3.0	Tr
4	Rajupura	4.0	107.0	8.5	4.0	Tr	1.5	8.5	3.0	Tr
5	Anasta	3.0	54.5	5.5	4.5	Tr	Tr	Tr	2.5	Tr
6	Majrol	2.5	90.5	6.5	2.5	Tr	1.0	5.5	2.5	Tr
7	Ghor-1	2.0	77.0	5.5	4.0	Tr	1.0	2.0	2.5	Tr
8	Ghora-2	3.0	144.5	9.0	6.5	Tr	1.5	1.5	3.5	1.5
9	Ghora-4	2.5	68.5	7.5	4.0	1.5	0.5	Tr	2.5	Tr
10	Segva	3.5	97.5	8.0	4.0	Tr	0.5	9.5	3.0	Tr
11	Pingalwala	3.0	93.0	6.5	6.5	0.5	2.0	8.0	4.5	Tr
12	Near khandha farm	3.0	60.0	6.5	5.5	1.0	1.0	3.5	3.0	Tr
13	Utraj	1.5	90.5	7.5	5.0	Tr	1.5	9.0	4.0	Tr
14	Ringroad	3.5	60.0	6.0	4.5	1.0	0.5	35.0	3.5	Tr
15	Hando-1	3.0	67.0	7.0	7.5	Tr	1.5	Tr	2.5	Tr
16	Handod-2	1.5	79.5	6.0	5.0	Tr	Tr	9.0	2.5	Tr
17	Sinor	4.0	83.5	8.0	5.5	Tr	0.5	2.0	2.5	Tr
18	Bhoj	3.5	112.0	10.5	4.5	1.0	2.0	6.0	3.0	Tr
19	Kaditalavadi	3.5	64.5	8.0	6.0	2.0	1.0	1.0	3.0	Tr
20	Lilod	2.0	67.5	7.0	5.0	1.0	0.5	2.5	1.5	Tr
21	Osaram-1	2.5	54.5	6.0	3.5	Tr	Tr	Tr	2.5	Tr
22	Osaram-2	2.5	75.5	5.5	5.0	Tr	0.5	7.5	3.5	Tr
23	Sahera	3.0	85.5	6.0	7.5	1.0	0.5	5.5	2.5	Tr
24	Sadhli	2.5	75.0	6.0	6.5	2.0	0.5	Tr	3.5	Tr
25	Vermar	3.0	61.5	6.0	5.0	Tr	Tr	1.5	2.5	Tr
26	Kuray	3.5	68.0	6.5	5.0	0.5	Tr	2.5	2.0	Tr
27	Khandha farm, AAU	3.0	77.0	7.0	6.5	0.5	Tr	1.0	3.0	Tr
28	Dhavat	2.5	46.0	5.5	5.0	1.0	Tr	2.0	2.5	Tr
29	Kurali	2.5	55.0	5.0	6.5	Tr	Tr	0.5	3.0	Tr

Tr-Traces

Similarly Co content in cotton lint sample ranged from Tr-2.0 ppm. The highest Co content was recorded in the sample collected from Pingalwala and Bhoj region and the lowest in cotton lint sample collected from Vadodara district region. The Cr content ranged from Tr-9.5 ppm in cotton lint sample. The highest Cr content was recorded in the sample collected from Segva region (9.5 ppm) and the lowest amount of Cr in the cotton lint sample collected from Vadodara district region (Tr).

The Ni content in the cotton lint samples ranged from 1.5-4.5 ppm. The highest Ni content was recorded in the cotton lint sample collected from Pingalwala region (4.5 ppm) and the least from Lilod regions (1.5 ppm).

The concentration of Pb in the cotton lint samples ranged from Tr-1.5 ppm. The highest Pb content was recorded in the sample collected from Anand region i.e., Ghora-2 (1.5 ppm), all other samples contained traces of Pb.

V. SUMMARY AND CONCLUSION

An investigation into the status of pesticide residues and heavy metals in water, soil, vegetables and cotton lint from vegetable and cotton growing area of middle Gujarat was conducted in Pesticide Residue Laboratory during 2012-13**.** The samples were analysed by using facilities of AINP on Pesticide Residues and Micronutrient Project, ICAR Unit -9, AAU, Anand. Approximately 200 samples were collected from Kheda, Anand and Vadodara districts of Middle Gujarat area having intensive cultivation of vegetables and cotton.

Soil samples were collected from the depth (0-15 cm) by auger. For composite sample, small portions of soil were collected from 15-20 well distributed spots, moving in a zigzag manner from each individual sampling site. Soil bulk was reduced to about 500 g by quartering and kept it in a clean cloth bag. Before ground water sample collection tube well was switched on for 15-20 minutes for purging. Water sample filtered through muslin cloth was collected in to a 2.5 L amber colour glass bottle.

The findings of the above research work are summarized as follows:

5.1 Ground and surface water

5.1.1 Pesticide residues

The ground water (41) and surface water (10) samples were monitored for pesticide residues and the results revealed that none of the sample contained pesticides residues above the reporting limit of 0.1 ppb.

5.1.2 Micronutrient status in water

Micronutrient (Cu, Fe, Mn and Zn) and heavy metal (Cd, Co, Cr, Ni and Pb) contents were analysed from water samples.

- ❖ The results of ground water samples indicated that out of 41 samples, 37 samples (90.24%) contained Fe content above prescribed limit of 0.3 ppm for drinking water as per the BIS standard. Similarly all the surface water samples also contained Fe above prescribed BIS limit.

- ❖ The Cu, Mn and Zn content were within the prescribed limit for both ground and surface waters.

5.1.3 Heavy metal status in water

- ❖ The status of heavy metals in ground water showed that 22 out of 41 samples (53.66%) exceed prescribed 0.05 ppm limit for Cr as per the BIS standard, however for Pb, only one sample (Atladara water sample) exceed 0.1 ppm prescribed limit for drinking water by BIS. In case of Cd, 28.83% samples exceeded the prescribed limit of 0.01 ppm for drinking water.
- ❖ In case of surface water only one sample collected from Rarod V.P. contained higher than its prescribed limit of 0.01 ppm. In case of Cr, 80% surface water samples exceeded the limit of 0.05 ppm.

5.1.4 EC and pH of ground and surface water

- ❖ All the ground water samples collected were measured for the EC and pH which ranged from 0.53-17.71 dSm^{-1} and 8.11-9.10, respective.
- ❖ EC in the surface water samples ranged from 0.361-4.14 dSm^{-1} whereas pH from 8.28-9.05.

5.1.5 Nitrate-nitrogen in ground water and phosphorus in surface water

- ❖ The NO_3-N^- content in the ground water samples ranged from 0.018-1.182 ppm. None of the sample exceeded the prescribed critical limit of 10 ppm.
- ❖ The surface water samples from 10 regions were analysed for phosphorous content. The phosphorous content of surface water ranged from 0.117-0.737 ppm. As per Minnesota Pollution control agency study (2007) source and forms of phosphorous have impact on Water Quality and observed that the phosphorous

concentration from 0.01-0.03 ppm which is normal from water quality view point.

5.2 Cotton growing and vegetable growing soil

5.2.1 Pesticide residues

- Total 80 soil samples were collected from cotton growing area. Out of 80 samples analysed for pesticide residues only 5.0 % samples showed residues above detectable limit.
- Out of 22 soil samples collected from vegetable growing area only 3 soil samples were contaminated with pendimethalin. Thus, only 13.64 per cent soil samples were found positive for pesticide residues.

5.2.2 Micronutrient status in soil

- Soil samples were analysed to the know micronutrient status (ppm) from cotton growing area. The Cu content in the cotton growing soil samples ranged from 0.84-22.90 pp, Fe from 0.74-30.44 ppm, Mn from 3.34-113.34 ppm and the Zn from Tr-46.4 ppm. As per Ewvers (1991) the standard guideline and legislative regulations the permissible limits of Zn and Cu are 300 and 100 ppm, respectively.
- Soil samples were analysed to know the status of micronutrients in vegetable growing soils. The Cu content in the soil samples ranged from 1.02 to 15.42 ppm. The Fe content in vegetable growing soil samples ranged from 3.56-28.80, Mn content from 4.44-89.76 and the Zn from 0.6-22.06 ppm.

5.2.3 Heavy metal status in soil

- The heavy metal content of soil samples collected from cotton growing area reveal presence of Cd, Cr, Co, Ni and Pb in the range Tr- 0.26, Tr- 1.06, 0.2-1.28, 0.06- 3.00 and 0.2- 1.28 ppm, respectevly. As per Ewvers (1991) the std.

guideline and legislative regulations the permissible limit of Ni, Cr, Pb and Cd for soil is 50, 50, 100 and 3 ppm, respectively**.**

- ❖ The heavy metal concentration of 22 vegetable growing soil samples were analysed and the Cd content varied from Tr-0.18 ppm. The Co content ranged from 0.22-0.84, Cr Tr-1.38, Ni 0.28-1.40, and Pb Tr-1.76 ppm, respectively. The present finding revealed that Cd, Cr, Ni and Pb contents in the soil samples were below prescribed permissible limits (Ewvers, 1991).

5.2.4 Organic carbon content

- ❖ The percentage O.C content of cotton growing soil samples ranged from 0.03-0.95.
- ❖ The percentage O.C content of vegetable growing soil samples ranged from 0.25-0.97.

5.2.5 EC and pH of cotton growing and vegetable growing soil

- ❖ The EC and pH of soil samples collected from cotton growing area varied from 0.126-2.260 dS m^{-1} and 7.02-8.67, respectively.
- ❖ The EC and pH of soil samples collected from vegetable growing area ranged from 0.292-2.680 dS $^{m-1}$ and 7.32-8.67, respectively.

5.3 Vegetables

5.3.1 Pesticide residue

- ❖ Total 21 vegetable samples comprising brinjal, (5); tomato, (2); capsicum, (5) and okra, (9) collected from farmer’s field were monitored for pesticide residues by gas chromatography techniques. The samples of different vegetables were analyzed to determine the pesticide residues. None of the brinjal sample was contaminated with pesticide residues. Out of 9 okra samples and 5 capsicum samples analysed one each contained acephate at 0.141 and 0.087 ppm, respectively.

5.3.2 Micronutrient status in vegetables

- ❖ Samples were analysed to know the micronutrient status (ppm) in vegetables. The Cu content in the vegetable samples varied from 5.5 to 16.0 ppm. Similarly Fe from 89.5 – 295.5 ppm and Mn from 11.0 - 44.0 ppm. The Zn concentration was found to be ranging from 14.0 -53.5 ppm. As per the Codex Stan 193, (1995); the permissible limit of Zn and Cu are 9.4 and 73.3 ppm, respectively.

5.3.3 Heavy metal status in vegetables

- ❖ The heavy metal concentrations of 21 vegetable samples were analysed. Cd content in vegetable samples ranged from Tr-2.0 ppm, Co from Tr- 3.5 ppm, Ni from 0.6-8.5 ppm, Cr from Tr-10.5 ppm and Pb from Tr-18.5 ppm. The maximum limit prescribed by Codex Stan 193 for Cd, Cr and Pb is 0.05, 2.3 and 0.1 ppm, respectively.

5.4 Cotton lint

5.4.1 Pesticide residues

- ❖ A total of 29 cotton lint samples were analyzed for the pesticide residues. Cotton lint samples were contaminated with chlorpyriphos (0.011 and 0.015 ppm) and quinalphos (0.054 ppm).

5.4.2 Micronutrient status in cotton lint

- ❖ Samples were analysed to know the micronutrient levels in cotton lint. In the case of micronutrient content, Cu concentration ranged from 1.5- 4.0 ppm, Fe from 46.0- 144.5 ppm, Mn from 5.0-10.5 ppm and Zn from 2.5-7.5 ppm.

5.4.2 Heavy metal status in cotton lint

- The heavy metal concentration of 29 cotton lint samples was analysed. Cd content in cotton lint samples ranged from Tr-2.0 ppm, Co from Tr-2.0 ppm, Cr from Tr-9.5 ppm, Pb from Tr-1.5 ppm and Ni from 1.5-4.5 ppm.

Conclusion:

None of the water sample showed pesticides above reporting level of 0.1 ppb. Though surface water is not used for drinking purpose for human being, cattles use the surface water for drinking purpose. As per the BIS standard the safe limit of Cu in drinking water is 0.05 ppm. It is evident from the results that ground water samples contain Cu within the safe limit. This leads to the conclusion that the Zn content in all the ground water samples was below the permissible limit. Twenty two (53.66 %) ground water samples exceeded the prescribe limit of 0.05 ppm for Cr as per the BIS standard. In the present study none of the surface water sample exceeded the safe limit of Cu. The present survey revealed 100 % surface water samples exceeding the critical limit for Fe content and 70 % surface water samples exceeding the critical limit for Cr. The wide variation in the NO_3-N^- concentration in water samples collected from different locations might be due to use of nitrogenous fertilizers in different doses under different cropping pattern in the region. However in the present study 100 per cent surface water samples recorded phosphorous content beyond the normal level. There were differences in EC of ground and surface water samples. The EC of the ground water samples was higher than surface water samples. However, differences in pH among ground and surface water were marginal.

None of the soil sample analyzed found to contain heavy metals concentration that result in toxicity. The 11.25 % soil samples under cotton growing area had enough organic carbon content as they exceeded the 0.75 % limit. Out of 22 soil samples collected from vegetable growing area 27.27 per cent soil samples had enough organic carbon. The variation in the soil organic content might be due to the variation in soil texture, soil microbial activity, OM content, nutrient management practices in the region, differences in addition of organic manures like FYM, vermicompost, cakes and soil pH etc.

Out of 21 vegetable samples only two samples, one each of okra and capsicum showed presence of acephate. The Zn content in tomato samples found to be above the prescribed limit. Low level of pesticides in cotton lint (only 10.34 % samples) could be due to cultivation of *Bt* varieties by most of the farmers, in the area.

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recoveries and % RSD_{WR} were well within the limits prescribed by the SANCO guide lines (2013) for most compounds. According to the SANCO guidelines (2013), any analytical method which recorded mean recovery in the range of 70-120% and % $RSD_{WR} \leq 20$ % is sufficiently accurate and precise. Hence the method employed for the extraction of different group of pesticides for different matrices is accurate and precise.

The data obtained from recovery studies of OCs from water were in the range of 79.75 to 153.67 % at 0.1 ppb level and 74.82 to 132.35 % at 0.5 ppb level. Maximum recovery was observed for endrin ketone (153.67 %), while minimum for a-HCH (79.75 %) at 0.1 ppb level. The corresponding values at 0.5 ppm level showed maximum for methoxychlor (132.35 %), while minimum for heptachlor (74.82 %). The % RSD was found in the range of 1.82 to 5.37 and 4.66 to 10.88 at 0.1 and 0.5 ppm levels, respectively. The LOD for OCs was in the range 0.007 to 0.014 ppb whereas LOQ was in the range of 0.020 to 0.043 ppb at 0.1 ppb level of fortification (Table 4.3).

The data obtained from recovery studies of OPs from water were in the range 89.06 to 102.15 % at 0.1 ppb level and 96.06 to 124.91 % at 0.5 ppb level. Maximum recovery was observed for phosalone (102.15 %), while minimum for ethion (89.06 %) at 0.1 ppb level. The corresponding values at 0.5 ppb level showed maximum for acephate (124.91 %), while minimum for DDVP (96.06 %). The % RSD was found in the range of 7.17 to 11.52 and 0.91 to 6.73 at 0.1 and 0.5 ppb level, respectively. The LOD for OPs compound was 0.034 to 0.057 ppb whereas LOQ was in the range of 0.102 to 0.172 ppb at 0.1 ppb level of fortification (Table 4.5).

Table: 4.3 Recovery of OCs from water at 0.1 ppb

	% Recovery										«g L^{-1}	
Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	81.49	82.54	79.66	82.27	79.31	79.03	73.93	79.75	2.94	3.68	0.009	0.028
β-HCH	84.34	86.71	84.56	86.29	83.51	80.45	77.14	83.29	3.4	4.08	0.011	0.032
γ-HCH	82.74	85.68	81.85	82.47	80.31	78.47	73.73	80.75	3.81	4.72	0.012	0.036
δ-HCH	84.87	84.44	81.09	81.24	79.32	78.15	73.57	80.38	3.88	4.83	0.012	0.037
Heptachlor	92.37	97.3	93.47	95.05	93.14	94.71	88.23	93.47	2.81	3.01	0.009	0.026
Aldrin	88.64	89.59	86.05	87.71	86.72	85.02	80.37	86.3	3.04	3.52	0.010	0.029
Heptachlor epoxide	88.06	89.45	88.93	88.34	86.06	84.09	82.36	86.76	2.68	3.09	0.008	0.025
γ-Chlordane	86.01	90.73	86.68	86.86	84.45	83.95	81.86	85.79	2.8	3.27	0.009	0.026
α-Endosulfan	91.92	94.56	91.04	92.65	89.51	88.86	85.75	90.61	2.87	3.17	0.009	0.027
***p,p*'-DDE**	91.08	93.25	89.36	90.25	89.28	87.36	83.01	89.08	3.23	3.63	0.010	0.030
Dieldrin	92.06	94.01	88.89	90.35	86.21	90.78	84.65	89.57	3.27	3.65	0.010	0.031
Endrin	106.75	115.35	106.42	114.98	112.47	114.38	109.81	111.45	3.82	3.42	0.012	0.036
β-Endosulfan	95.9	98.51	97.42	100.54	94.3	95.21	91.64	96.22	2.92	3.03	0.009	0.028
***p,p*'-DDD**	90.61	89.9	85.92	87.02	82.19	79.71	79.63	85.00	4.57	5.37	0.014	0.043
Endosulfan Sulphate	95.56	101.12	96.06	101.96	101.95	100.07	101.38	99.73	2.76	2.76	0.009	0.026
***p,p*'-DDT**	121.83	127.96	123.71	128.86	125.63	124.57	126.27	125.55	2.43	1.94	0.008	0.023
Methoxychlor	115.14	117.55	116.93	117.06	114.44	111.42	115.42	115.42	2.10	1.82	0.007	0.020
Endrin ketone	151.28	160.23	148.58	157.86	152.44	152.59	152.75	153.67	3.99	2.60	0.013	0.038

Table: 4.4 Recovery of OCs from water at 0.5 ppb

% Recovery											«g L^{-1}	
Compound	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	71.62	80.16	77.32	75.64	71.58	76.40	71.05	74.82	3.49	4.66	0.055	0.164
γ-HCH	82.46	85.61	84.36	81.09	63.42	76.72	75.85	78.50	7.58	9.66	0.119	0.357
Heptachlor	76.64	85.39	81.33	73.86	62.91	72.58	75.26	75.42	7.11	9.42	0.112	0.335
Aldrin	85.82	91.12	87.73	87.69	73.81	80.57	81.64	84.05	5.81	6.91	0.091	0.274
β -HCH	115.58	108.46	118.33	105.92	105.26	107.93	94.48	107.99	7.74	7.16	0.121	0.364
δ-HCH	116.03	112.11	115.22	109.77	98.54	111.65	103.28	109.51	6.40	5.84	0.100	0.301
Heptachlor epoxide	103.84	106.13	108.52	105.41	93.78	96.24	92.76	100.95	6.49	6.43	0.102	0.306
α- Endosulfan	107.32	109.34	112.53	107.68	96.55	100.32	96.22	104.28	6.52	6.25	0.102	0.307
γ-Chlordane	117.85	117.38	115.38	107.94	100.53	110.19	104.19	110.50	6.72	6.08	0.106	0.317
***p,p*' - DDE**	126.72	120.65	120.79	112.31	104.05	118.50	110.77	116.25	7.63	6.56	0.120	0.359
Dieldrin	95.25	99.51	98.08	91.89	86.39	88.60	90.16	92.84	4.92	5.30	0.077	0.232
Endrin	101.60	104.83	106.40	102.38	91.52	96.92	93.01	99.52	5.79	5.82	0.091	0.273
***p,p*' - DDD**	118.70	116.80	119.29	112.71	99.16	109.41	100.31	110.91	8.38	7.56	0.132	0.395
β- Endosulfan	114.71	109.85	113.37	113.49	100.26	103.12	95.42	107.17	7.58	7.07	0.119	0.357
***p,p*' - DDT**	133.86	133.85	135.23	124.77	108.42	124.44	115.95	125.22	10.15	8.11	0.159	0.478
Endosulfan sulphate	119.76	119.96	125.02	120.46	106.80	108.66	110.80	115.92	7.03	6.06	0.110	0.331
Methoxychlor	130.72	152.73	145.75	130.85	110.35	135.85	120.17	132.35	14.40	10.88	0.226	0.678
Endrin Ketone	119.76	119.29	120.41	115.18	102.30	111.21	106.65	113.54	7.08	6.24	0.111	0.334

Table: 4.5 Recovery of OPs from water at 0.1 ppb

% Recovery											«g L^{-1}:	
Compound	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	103.36	116.54	90.59	96.38	107.47	92.78	99.55	100.95	9.02	0.089	0.028	0.085
Acephate	108.57	97.06	83.03	101.46	113.65	86.18	106.39	99.48	11.46	0.115	0.036	0.108
Monocrotophos	103.62	96.59	81.97	99.23	105.45	92.71	95.93	96.50	7.79	0.081	0.024	0.073
Dimethoate	103.69	96.61	80.39	97.58	113.46	92.1	100.33	97.74	10.19	0.104	0.032	0.096
Phorate	106.61	97.71	82.37	99.81	111.93	96.99	106.39	100.26	9.59	0.096	0.030	0.090
Me-Parathion	106.09	95.54	77.56	92.18	105.65	91.64	98.41	95.30	9.76	0.102	0.031	0.092
Phorate sulphone	114.84	104.56	90.27	102.73	104.21	98.11	99.44	102.02	7.48	0.073	0.024	0.071
Malathion	110.32	90.84	81.22	98.65	106.27	90.54	98.47	96.62	9.97	0.103	0.031	0.094
Chlorpyriphos	102.14	97.57	81.29	95.54	98.50	91.98	98.46	95.07	6.82	0.072	0.021	0.064
Quinalphos	104.38	96.50	83.95	92.10	105.42	89.71	96.77	95.55	7.73	0.081	0.024	0.073
Profenophos	94.94	93.03	76.88	94.93	95.74	80.40	94.29	90.03	7.89	0.088	0.025	0.074
Ethion	97.16	89.05	79.22	81.34	98.75	84.11	93.82	89.06	7.78	0.087	0.024	0.073
Triazophos	103.42	93.52	81.35	92.80	106.08	91.60	105.39	96.31	9.08	0.094	0.029	0.086
Phosalone	112.13	106.99	93.16	89.12	116.56	93.48	103.64	102.15	10.48	0.103	0.033	0.099

Table: 4.6 Recovery of OPs from water at 0.5 ppb

% Recovery											«g L^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	95.28	98.10	97.16	95.74	95.10	95.26	95.80	96.06	1.14	1.18	0.018	0.053
Acephate	124.02	125.01	125.05	126.96	124.90	123.26	125.20	124.91	1.14	0.91	0.018	0.054
Phorate	100.3	97.27	98.79	96.14	99.63	100.20	98.35	98.67	1.55	1.57	0.024	0.073
Monocrotophos	93.75	99.60	97.02	97.57	93.86	93.26	99.28	96.33	2.69	2.80	0.042	0.127
Dimethoate	97.9	100.56	97.89	98.98	97.77	97.85	97.65	98.37	1.06	1.08	0.017	0.050
Chlorpyriphos	99.02	100.74	96.97	100.85	98.18	100.85	100.28	99.56	1.53	1.54	0.024	0.072
Parathion - Me	97.26	100.70	100.99	100.68	95.89	100.75	97.58	99.12	2.13	2.15	0.034	0.101
Malathion	101.8	101.70	102.54	103.04	96.14	102.35	102.01	101.37	2.35	2.32	0.037	0.111
Quinalphos	100.15	98.08	99.03	100.23	95.36	99.76	99.27	98.84	1.70	1.72	0.027	0.080
Ph- Sulphone	97.12	97.81	97.71	97.09	94.30	97.53	97.75	97.05	1.25	1.28	0.020	0.059
Profenophos	99.4	103.26	99.30	100.62	96.88	99.68	99.80	99.85	1.90	1.90	0.030	0.089
Ethion	100.48	100.99	101.45	100.57	94.11	100.56	100.58	99.82	2.54	2.55	0.040	0.120
Triazophos	99.86	99.87	103.66	100.20	92.91	99.69	99.23	99.35	3.20	3.22	0.050	0.151
Phosalone	111.12	109.01	102.37	114.13	97.51	98.52	114.43	106.73	7.19	6.73	0.113	0.339

The data obtained from recovery studies of SPs+HBs from water were in the range of 75.28 to 109.69 % at 0.1 ppb level and 63.28 to 110.99 % at 0.5 ppb level. Maximum recovery was observed for fenvarelate-I (109.69 %), while minimum for fluchloralin (75.28 %) at 0.1 ppb level. The corresponding values at 0.5 ppb level showed maximum for deltamethrin (110.99 %), while minimum for trifluralin (63.82 %). The % RSD was found in the range of 2.71 to 11.38 and 1.97 to 4.66 at 0.1 and 0.5 levels, respectively. The LOD for SPs+HBs compound was in the range 0.007 to 0.031 ppb whereas LOQ in the range of and 0.021 to 0.092 at 0.1 ppb level of fortification (Table 4.7).

The data obtained from recovery studies of OCs from soil were in the range 78.25 to 140.61 % at 0.01 ppm level, 78.25 to 108.33 % at 0.05 ppm level and 67.10 to 86.06 % at 0.1 ppm level. Maximum recovery was observed for endrin ketone (140.61 %), while minimum for α-HCH (78.25 %) at 0.01 ppm level. The corresponding values at 0.05 ppm level showed maximum for endrin ketone (108.33 %), while minimum for α-HCH (71.36 %) and at 0.1 ppm level showed maximum for δ-HCH (86.06 %) while minimum for methoxychlor (67.10 %). The % RSD was found in the range of 1.74 to 7.49, 2.06 to 3.96 and 5.82 to 25.08 at 0.01, 0.05 and 0.1 ppm level, respectively. The LOD for OCs compound was in the range 0.001 to 0.003 at 0.01 ppm level whereas LOQ in the range 0.015 to 0.008 at 0.01 ppm level of fortification (Table 4.9).

Table: 4.7 Recovery of SPs+HBs from water at 0.1 ppb

% Recovery											«g L^{-1}	
Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	82.90	87.02	84.03	86.61	87.15	90.22	85.85	86.25	2.37	2.74	0.007	0.022
Fluchloralin	71.13	75.23	72.00	75.96	76.89	80.16	75.57	75.28	3.03	4.02	0.010	0.029
Alachlor	77.79	81.53	80.09	82.65	84.22	83.59	82.59	81.78	2.22	2.71	0.007	0.021
Metalochlor	101.13	100.09	104.29	106.11	105.43	108.35	104.69	104.30	2.86	2.74	0.009	0.027
Pendimethalin	85.43	87.07	64.12	89.34	91.49	92.16	89.19	85.54	9.73	11.38	0.031	0.092
Butachlor	75.31	79.09	77.84	80.92	83.61	80.00	79.11	79.41	2.57	3.24	0.008	0.024
Bifenthrin	84.94	84.96	88.68	89.57	91.25	89.86	87.20	88.07	2.45	2.78	0.008	0.023
Fenpropethrin	85.16	85.18	87.75	90.85	91.64	88.12	87.60	88.04	2.50	2.84	0.008	0.024
λ- Cyhalothrin	93.11	102.11	99.68	103.03	109.04	105.51	108.83	103.04	5.58	5.41	0.018	0.053
β-Cyfluthrin	99.41	90.32	109.40	84.79	84.81	87.30	87.88	91.99	9.16	9.96	0.029	0.086
α-Cypermethrin	100.35	104.01	77.81	82.33	87.69	84.47	84.15	88.69	9.74	10.98	0.031	0.092
Fenvalerate-I	100.89	101.58	108.34	112.57	116.49	118.02	109.92	109.69	6.70	6.11	0.021	0.063
Fenvalerate-II	90.40	87.65	99.88	107.90	106.95	103.29	94.93	98.71	7.96	8.07	0.025	0.075
Deltamethrin	99.60	80.36	85.36	86.07	81.73	84.20	83.24	85.79	6.40	7.46	0.020	0.060

Table: 4.8 Recovery of SPs+HBs from water at 0.5 ppb

% Recovery											«g L^{-1}	
Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	61.72	62.56	66.23	64.62	62.02	62.79	63.03	63.28	1.60	2.53	0.025	0.075
Fluchloralin	74.45	77.55	81.54	77.95	75.54	79.72	78.77	77.93	2.41	3.10	0.038	0.114
Alachlor	95.30	97.30	102.82	99.58	96.79	98.77	99.74	98.61	2.45	2.48	0.038	0.115
Metalochlor	102.89	103.10	112.81	105.81	107.29	110.17	107.12	107.02	3.59	3.36	0.056	0.169
Pendimethalin	86.88	89.13	93.28	89.69	89.26	89.49	89.77	89.64	1.89	2.10	0.030	0.089
Butachlor	91.03	90.27	94.48	93.70	91.66	90.16	93.89	92.17	1.82	1.97	0.029	0.086
Bifenthrin	96.99	103.67	108.21	102.34	98.57	101.44	101.03	101.75	3.63	3.57	0.057	0.171
Fenpropathrin	96.98	99.43	105.21	98.86	100.42	99.71	101.18	100.26	2.55	2.55	0.040	0.120
λ- Cyhalothrin	100.10	102.26	109.95	106.12	98.77	99.59	101.14	102.56	4.05	3.95	0.064	0.191
β-Cyfluthrin	101.72	105.88	115.30	107.90	100.84	102.84	106.96	105.92	4.93	4.66	0.077	0.232
α-Cypermethrin	101.15	102.31	106.38	103.15	95.50	98.74	104.23	101.64	3.61	3.55	0.057	0.170
Fenvalerate-I	103.22	102.80	109.67	104.56	101.48	102.31	102.59	103.80	2.75	2.65	0.043	0.130
Fenvalerate-II	101.96	109.25	118.42	106.69	106.05	108.25	107.92	108.36	5.02	4.63	0.079	0.236
Deltamethrin	112.31	107.39	119.56	109.67	108.09	109.85	110.10	110.99	4.09	3.69	0.064	0.193

Table: 4.9 Recovery of OCs from soil at 0.01 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	82.64	78.74	70.59	74.00	87.03	81.33	73.43	78.25	5.86	7.49	0.002	0.0055
β-HCH	84.10	90.23	86.18	91.64	89.04	91.47	86.85	88.50	2.87	3.24	0.001	0.0027
γ-HCH	89.25	87.66	88.82	86.70	94.03	90.63	91.48	89.79	2.48	2.76	0.001	0.0023
δ-HCH	89.15	88.87	84.50	86.74	94.30	93.07	85.87	88.93	3.65	4.10	0.001	0.0034
Heptachlor	88.15	89.11	88.07	83.94	91.36	91.45	91.29	89.05	2.71	3.04	0.001	0.0026
Aldrin	86.12	85.75	83.57	82.19	87.79	88.23	83.77	85.34	2.26	2.65	0.001	0.0021
Heptachlor epoxide	88.34	89.50	88.35	87.12	90.72	90.68	92.21	89.56	1.76	1.96	0.001	0.0017
γ-Chlordane	88.64	92.26	91.54	88.69	91.41	92.30	92.39	91.03	1.66	1.83	0.001	0.0016
α-Endosulfan	86.80	85.31	82.43	83.59	87.10	88.16	83.71	85.30	2.14	2.51	0.001	0.0020
p,p'-DDE	90.89	91.89	94.23	92.50	94.74	95.31	92.60	93.16	1.62	1.74	0.001	0.0015
Dieldrin	92.97	92.44	89.16	93.51	93.66	94.50	93.13	92.77	1.71	1.85	0.001	0.0016
Endrin	91.84	101.86	100.19	101.15	105.68	103.38	107.55	101.66	5.04	4.96	0.002	0.0048
β-Endosulfan	88.36	86.83	82.64	84.80	91.91	90.16	85.86	87.22	3.18	3.65	0.001	0.0030
p,p'-DDD	85.12	84.21	84.94	87.99	90.58	87.58	88.12	86.94	2.27	2.61	0.001	0.0021
Endosulfan Sulphate	90.25	96.34	96.72	100.57	103.97	103.27	99.84	98.71	4.73	4.80	0.001	0.0045
p,p'-DDT	94.44	94.98	83.97	95.96	96.95	97.31	84.70	92.62	5.75	6.21	0.002	0.0054
Methoxychlor	97.62	99.29	97.19	101.52	109.70	105.46	100.40	101.60	4.52	4.45	0.001	0.0043
Endrin ketone	143.59	133.59	128.81	154.24	146.64	138.12	139.27	140.61	8.44	6.00	0.003	0.0080

Table: 4.10 Recovery of OCs from soil at 0.05 ppm

% Recovery										«g g^{-1}	
Compound	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	70.55	68.89	71.18	72.58	76.07	68.66	71.55	71.36	2.51	3.51	0.004	0.012
β-HCH	77.17	82.25	84.86	86.04	80.44	80.70	85.64	82.44	3.26	3.96	0.005	0.015
γ-HCH	74.82	77.11	82.12	81.92	80.30	78.21	82.37	79.55	2.91	3.66	0.005	0.014
δ-HCH	77.92	75.41	80.97	79.97	82.74	79.59	81.67	79.75	2.46	3.09	0.004	0.012
Heptachlor	81.63	83.19	86.33	83.91	81.79	84.93	84.85	83.80	1.73	2.06	0.003	0.008
Aldrin	75.18	77.19	78.35	80.53	75.24	78.20	78.42	77.59	1.91	2.46	0.003	0.009
Heptachlor epoxide	80.08	79.26	84.37	86.44	78.70	79.75	82.53	81.59	2.93	3.59	0.005	0.014
γ-Chlordane	81.98	81.15	87.22	87.30	82.08	83.49	87.68	84.42	2.88	3.41	0.005	0.014
α-Endosulfan	82.08	81.28	85.20	86.73	82.63	83.63	83.88	83.64	1.87	2.24	0.003	0.009
***p,p*'-DDE**	79.31	83.47	87.31	85.41	79.47	85.84	85.96	83.83	3.24	3.86	0.005	0.015
Dieldrin	86.54	86.61	90.14	92.65	88.47	89.33	89.18	88.99	2.11	2.37	0.003	0.010
Endrin	98.18	97.79	104.23	106.26	99.03	104.56	105.42	102.21	3.70	3.62	0.006	0.017
β-Endosulfan	83.28	79.71	85.18	82.03	83.88	83.33	84.98	83.20	1.88	2.25	0.003	0.009
***p,p*'-DDD**	85.39	84.58	90.79	89.66	84.11	88.39	89.39	87.47	2.72	3.11	0.004	0.013
Endosulfan Sulphate	92.85	91.34	100.74	98.42	95.93	95.59	95.35	95.74	3.16	3.30	0.005	0.015
***p,p*'-DDT**	89.68	88.00	94.11	89.01	89.91	92.47	92.11	90.76	2.18	2.40	0.003	0.010
Methoxychlor	94.72	91.91	98.70	99.41	100.75	97.91	101.07	97.78	3.34	3.42	0.005	0.016
Endrin ketone	105.12	104.91	112.67	110.67	106.16	107.89	110.90	108.33	3.10	2.87	0.005	0.015

Table: 4.11 Recovery of OCs from soil at 0.1 ppm

% Recovery											«g g^{-1}	
Compound	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	97.01	76.51	81.45	72.69	75.30	78.96	67.45	78.48	9.32	11.88	0.029	0.088
β-HCH	95.43	73.57	75.19	72.98	70.42	72.02	78.84	76.92	8.59	11.16	0.027	0.081
γ-HCH	97.46	82.28	84.75	80.54	80.82	83.03	75.72	83.52	6.77	8.10	0.021	0.064
δ-HCH	124.30	80.62	83.55	79.53	78.85	80.53	75.02	86.06	17.05	19.82	0.054	0.161
Heptachlor	99.29	81.50	83.48	78.54	79.30	80.86	74.83	82.54	7.87	9.53	0.025	0.074
Aldrin	92.52	73.72	76.50	71.72	71.87	73.81	68.09	75.46	7.95	10.53	0.025	0.075
Heptachlor epoxide	94.43	73.76	77.66	73.40	72.43	74.99	69.99	76.67	8.18	10.66	0.026	0.077
γ-Chlordane	92.96	75.50	77.89	74.99	74.09	72.73	71.34	77.07	7.31	9.48	0.023	0.069
α-Endosulfan	96.59	85.07	87.02	84.51	83.60	84.15	81.03	85.99	5.00	5.82	0.016	0.047
***p,p*'-DDE**	96.89	73.54	75.87	74.31	71.66	71.90	70.60	76.40	9.21	12.06	0.029	0.087
Dieldrin	97.23	74.11	77.49	74.43	71.49	73.26	69.84	76.84	9.31	12.11	0.029	0.088
Endrin	117.67	69.34	71.82	69.50	66.42	68.17	66.04	75.57	18.67	24.71	0.059	0.176
β-Endosulfan	96.78	67.01	71.02	67.40	67.35	65.84	64.60	71.43	11.35	15.89	0.036	0.107
***p,p*'-DDD**	90.58	66.07	66.68	67.17	63.38	61.98	62.88	68.39	9.99	14.61	0.031	0.094
Endosulfan Sulphate	110.14	76.48	77.72	77.00	74.42	73.87	72.20	80.26	13.32	16.59	0.042	0.126
***p,p*'-DDT**	98.22	77.87	80.57	79.73	76.11	75.00	71.56	79.87	8.64	10.82	0.027	0.081
Methoxychlor	97.91	63.92	64.05	64.25	60.53	59.28	59.75	67.10	13.76	20.50	0.043	0.130
Endrin ketone	117.15	68.83	68.89	71.21	67.08	65.45	65.30	74.85	18.77	25.08	0.059	0.177

The data obtained from recovery studies of OPs from soil were in the range 88.43 to 113.34 %, 86.47 to 122.39 % and 92.45 to 106.94 % at 0.01, 0.05 and 0.1 ppm level, respectively. Maximum recovery was observed for malathion (113.34 %), while minimum for phorate (88.43 %) at 0.01 ppm level. The corresponding values at 0.05 Phorate sulphoxide (122.39 %), while minimum for phorate (86.47 %) and at 0.1 ppm level maximum recovery was observed for Phorate sulphoxide (106.94 %) while minimum for phosalone (92.45 %). The % RSD was found in the range of 3.77 to 18.92, 9.34 to 19.08 and 5.01 to 15.34 at 0.01, 0.05 and 0.1 ppm level. The LOD for OPs compound was in the range 0.001 to 0.006 at 0.01 where as LOQ in the range 0.004 to 0.018 at 0.01 ppm level of fortification (Table 4.12).

The data obtained from recovery studies of SPs from soil were in the range 85.10 to 133.33 % at 0.01 ppm level, 73.82 to 100.09 % at 0.05 ppm level and 65.17 to 96.14 % at 0.1 ppm level. Maximum recovery was observed for bifenthrin (133.33 %), while minimum for λ-cyhalothrin (85.10 %) at 0.01 ppm level. The corresponding values at 0.05 ppm level showed maximum for deltamethrin (100.09 %), while minimum for fenpropathrin (73.82 %) and at 0.1ppm level showed maximum for deltamethrin (96.14 %) while minimum for fenpropathrin (65.17 %). The % RSD was found in the range of 10.99 to 14.20 at 0.05, 6.43 to 18.88 at 0.1 and 2.57 to 5.19 at 0.5 ppm level. The LOD for SPs compound was in the range 0.003 to 0.005 at 0.01ppm whereas LOQ in the range 0.010 to 0.014 at 0.01 ppm level of fortification (Table 4.15).

The data obtained from recovery studies of HBs from soil were in the range 94.67 to 118.70 % at 0.01 ppm level, 82.57 to 94.86 % at 0.05 ppm level and 84.63

Table: 4.12 Recovery of OPs from soil at 0.01 ppm

% Recovery											«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Phorate	79.23	105.14	70.83	90.92	77.24	109.02	86.62	88.43	14.33	16.20	0.005	0.014
Dimethoate	83.50	123.12	71.14	103.74	87.58	113.29	111.39	99.11	18.75	18.92	0.006	0.018
Chlorpyriphos	99.37	86.94	96.56	113.69	94.62	89.03	95.29	96.50	8.72	9.04	0.003	0.008
Me-parathion	102.22	92.60	105.13	116.2	121.17	94.75	98.06	104.30	10.79	10.35	0.003	0.010
Malathion	123.84	99.57	109.51	119.41	120.32	104.24	116.48	113.34	9.06	7.99	0.003	0.009
Quinalphos	112.54	104.80	115.54	109.18	111.14	105.20	113.91	110.33	4.16	3.77	0.001	0.004
Phorate sulphone	111.97	99.56	119.27	115.69	108.77	103.42	104.82	109.07	7.03	6.45	0.002	0.007
Profenophos	109.45	100.13	107.67	84.47	116.31	119.96	117.67	107.95	12.4	11.49	0.004	0.012
Ethion	109.50	104.88	112.16	123.14	105.31	102.26	106.46	109.10	6.98	6.40	0.002	0.007
Triazophos	111.22	105.13	122.45	80.52	126.76	107.04	128.81	111.70	16.69	14.94	0.005	0.016

Table: 4.13 Recovery of OPs from soil at 0.05 ppm

% Recovery										«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Phorate	70.43	81.34	77.96	72.38	100.51	90.89	111.79	86.47	15.34	17.74	0.024	0.072
Dimethoate	102.31	125.28	91.70	97.71	104.61	93.2	92.74	101.08	11.76	11.63	0.018	0.055
Chlorpyriphos	92.83	98.65	79.63	76.92	90.75	107.31	114.26	94.34	13.65	14.47	0.021	0.064
Me-parathion	80.49	96.69	103.54	71.38	82.15	114.57	119.51	95.48	18.22	19.08	0.029	0.086
Malathion	93.79	105.95	82.10	83.27	93.63	123.96	119.55	100.32	16.68	16.63	0.026	0.079
Phorate sulphoxide	117.26	112.51	115.03	115.22	117.07	159.06	120.59	122.39	16.36	13.37	0.026	0.077
Quinalphos	99.17	99.57	91.43	85.72	98.17	111.5	115.63	100.17	10.47	10.45	0.016	0.049
Phorate sulphone	92.97	101.66	86.72	85.34	92.09	117.39	116.79	98.99	13.43	13.57	0.021	0.063
Profenophos	83.72	110.44	74.34	76.28	84.35	111.18	102.00	91.76	15.78	17.20	0.025	0.074
Ethion	99.64	99.14	93.24	89.47	99.15	112.71	115.04	101.20	9.45	9.34	0.015	0.045
Triazophos	79.18	94.65	74.17	73.07	82.74	107.25	104.97	88.00	14.27	16.22	0.022	0.067
Phosalone	86.09	84.36	98.19	97.69	70.66	92.45	113.66	91.87	13.47	14.66	0.021	0.063

Table: 4.14 Recovery of OPs from soil at 0. 1 ppm

% Recovery										«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Phorate	120.54	80.43	114.58	97.15	118.97	87.52	111.43	104.37	16.01	15.34	0.05	0.151
Dimethoate	103.10	93.56	102.04	106.35	106.2	109.90	101.25	103.20	5.19	5.03	0.016	0.049
Chlorpyriphos	97.34	88.40	82.57	101.81	100.74	94.89	104.78	95.79	7.90	8.25	0.025	0.074
Me-parathion	110.67	85.68	112.39	103.82	114.52	94.92	112.69	104.95	10.9	10.39	0.034	0.103
Malathion	107.63	95.42	103.85	105.08	110.67	107.89	110.88	105.92	5.31	5.01	0.017	0.05
Phorate sulphoxide	91.88	97.16	80.22	101.72	81.11	105.84	89.25	92.45	9.80	10.60	0.031	0.092
Quinalphos	93.13	95.78	77.24	99.70	97.62	105.15	102.59	95.89	9.16	9.55	0.029	0.086
Phorate sulphone	95.90	100.4	83.34	103.23	100.36	110.82	101.09	99.31	8.36	8.42	0.026	0.079
Profenophos	97.72	97.12	113.13	106.73	100.55	112.28	106.64	104.88	6.58	6.27	0.021	0.062
Ethion	94.13	100.53	78.12	101.78	98.98	109.52	104.46	98.22	10.05	10.23	0.032	0.095
Triazophos	94.78	105.1	90.30	107.21	101.61	115.56	104.08	102.66	8.27	8.06	0.026	0.078
Phosalone	108.23	126.83	102.00	88.87	128.38	97.6	96.68	106.94	15.28	14.29	0.048	0.144

Table: 4.15 Recovery of SPs from soil at 0.01 ppm

% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	117.24	127.50	131.23	151.58	132.49	118.81	154.48	133.33	14.66	10.99	0.005	0.014
λ-Cyhalothrin	77.75	71.55	87.95	100.26	86.78	77.65	93.80	85.10	10.08	11.84	0.003	0.010
β-Cyfluthrin	74.73	76.20	91.37	102.30	90.64	83.15	98.35	88.11	10.56	11.98	0.003	0.010
α-Cypermethrin	77.36	76.77	93.25	108.76	91.58	85.91	100.98	90.66	11.79	13.00	0.004	0.011
Fenvalerate-I	76.41	73.78	90.13	105.83	85.74	79.09	99.04	87.15	11.97	13.74	0.004	0.011
Fenvalerate-II	89.77	78.66	103.58	119.05	84.68	92.88	102.87	95.93	13.62	14.20	0.004	0.013
Deltamethrin	84.46	77.85	93.30	110.56	91.25	87.15	99.52	92.01	10.68	11.60	0.003	0.010

Table: 4.16 Recovery of SPs from soil at 0.05 ppm

% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	79.33	75.73	90.89	83.76	95.26	86.22	109.20	88.63	11.21	12.65	0.02	0.05
Fenpropathrin	72.90	70.47	78.75	78.59	65.57	73.78	76.67	73.82	4.75	6.43	0.01	0.02
λ-Cyhalothrin	74.65	72.40	84.02	84.25	92.52	81.93	103.99	84.82	10.75	12.68	0.02	0.05
β-Cyfluthrin	83.40	81.01	93.37	96.13	105.53	93.85	121.10	96.34	13.63	14.15	0.02	0.06
α-Cypermethrin	78.58	75.80	88.03	86.57	96.83	87.97	114.21	89.71	12.81	14.28	0.02	0.06
Fenvalerate-I	77.32	74.49	86.67	86.78	91.71	81.96	107.67	86.66	10.99	12.68	0.02	0.05
Fenvalerate-II	77.16	69.48	84.09	80.97	86.74	78.09	103.96	82.93	10.80	13.02	0.02	0.05
Deltamethrin	84.58	81.13	100.29	100.57	108.88	96.39	128.79	100.09	15.89	15.88	0.02	0.07

to 97.32 % at 0.1 ppm level, respectively. Maximum recovery was observed for metalochlor (167.31 %), while minimum for pendimethalin (94.67 %) at 0.01 ppm level. The corresponding values at 0.05 and 0.1 ppm level showed maximum for metalochlor (94.86 %) and metalochlor (97.32 %), while minimum for fluchloralin (82.57 %) and pendimethalin (84.63 %), respectively. The % RSD was found in the range of 4.31 to 9.06, 5.46 to 7.93 and 5.54 to 8.80 at 0.01, 0.05 and 0.1 ppm level. The LOD for HBs compound was in the range 0.001 to 0.05 at 0.01 ppm whereas LOQ 0.004 to 0.013 at 0.01 ppm level of fortification (Table 4.18).

The data obtained from recovery studies of OCs from brinjal were in the range of 74.57 to 148.34 %, 74.49 to 237.33 % and 72.39 to 184.87 % at 0.01, 0.05 and 0.1 ppm level, respectively. Maximum recovery was observed for methoxychlor (148.34 %), while minimum for aldrin (74.57 %) at 0.01 ppm level. The corresponding values at 0.05 and at 0.1 ppm level maximum for endrin ketone (237.33 %) and endrin ketone (184.87 %), while minimum for dieldrin (74.49 %) and dieldrine (72.39 %) respectively. The % RSD was found in the range of 1.78 to 15.85, 0.97 to 2.52 and 2.43 to 6.64 at 0.01, 0.05 and 0.1 ppm level, respectively. The LOD for OCs compound was in the range of 0.0005 to 0.004 at 0.01 ppm whereas LOQ in the range 0.001 to 0.013 at 0.01ppm level of fortification (Table 4.21).

The data obtained from recovery studies of OPs from brinjal were in the range of 63.24 to 128.80 %, 100.86 to 111.98 % and 83.98 to 103.73 % at0.05, 0.25 and 0.5 ppm level, respectively. Maximum recovery was observed for phosalone (128.80 %) while minimum for dimethoate (63.24 %) at 0.05 ppm level. The

Table: 4.17 Recovery of SPs from soil at 0.1 ppm

% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	88.24	86.59	83.88	88.39	87.50	83.46	83.79	85.98	2.21	2.57	0.007	0.021
Fenpropathrin	64.32	65.49	60.81	68.92	67.08	68.72	60.88	65.17	3.38	5.19	0.011	0.032
λ-Cyhalothrin	82.46	80.93	75.15	86.25	82.73	82.98	77.76	81.18	3.68	4.53	0.012	0.035
β-Cyfluthrin	92.27	93.94	88.62	97.16	97.32	95.04	87.77	93.16	3.83	4.11	0.012	0.036
α-Cypermethrin	85.75	87.03	81.88	90.29	88.09	89.74	81.18	86.28	3.60	4.17	0.011	0.034
Fenvalerate-I	84.72	83.77	79.78	86.40	87.56	86.03	79.52	83.97	3.19	3.79	0.010	0.030
Fenvalerate-II	82.95	84.86	79.19	86.26	85.44	84.36	76.78	82.83	3.53	4.26	0.011	0.033
Deltamethrin	92.15	96.65	91.30	100.71	100.58	98.19	93.42	96.14	3.91	4.07	0.014	0.037

Table: 4.18 Recovery of HBs from soil at 0.01 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	124.68	116.35	126.82	120.47	109.52	112.83	120.18	118.70	6.21	5.23	0.002	0.006
Fluchloralin	97.69	94.96	100.27	100.27	91.21	86.84	98.62	95.69	5.06	5.28	0.002	0.005
Alachlor	101.79	99.56	106.96	105.61	92.61	85.31	86.80	96.95	8.78	9.06	0.003	0.008
Metalochlor	181.85	175.03	163.55	178.27	159.00	172.78	140.68	167.31	14.22	8.50	0.005	0.013
Pendimethalin	96.49	93.37	102.07	94.13	90.38	89.46	96.78	94.67	4.28	4.52	0.001	0.004
Butachlor	115.28	121.69	112.49	120.86	110.98	109.55	110.92	114.54	4.94	4.31	0.002	0.005

Table: 4.19 Recovery of HBs from soil at 0.05 ppm

% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	92.81	90.89	79.17	77.28	89.61	89.51	78.29	85.37	6.77	7.93	0.011	0.032
Fluchloralin	86.51	86.21	82.15	70.69	85.17	86.33	80.90	82.57	5.69	6.89	0.009	0.027
Alachlor	90.79	91.62	84.82	78.55	90.02	91.32	86.29	87.63	4.79	5.46	0.008	0.022
Metalochlor	89.82	98.61	98.34	92.37	96.62	101.90	86.34	94.86	5.52	5.82	0.009	0.026
Pendimethalin	86.79	87.72	84.08	69.25	83.44	85.99	81.01	82.61	6.31	7.64	0.010	0.030
Butachlor	88.58	94.79	90.15	78.59	86.71	93.46	89.85	88.88	5.31	5.97	0.008	0.025

Table: 4.20 Recovery of HBs from soil at 0.1 ppm

% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	100.05	85.82	85.38	94.51	83.26	77.29	83.32	87.09	7.67	8.80	0.024	0.072
Fluchloralin	95.29	85.20	84.64	93.46	81.51	77.10	81.05	85.47	6.66	7.79	0.021	0.063
Alachlor	101.05	91.11	88.92	97.03	90.01	81.33	89.27	91.25	6.31	6.91	0.020	0.060
Metalochlor	105.44	96.57	97.01	101.91	93.03	89.11	98.15	97.32	5.39	5.54	0.017	0.051
Pendimethalin	93.68	83.42	83.42	92.56	80.67	76.44	82.25	84.63	6.27	7.41	0.020	0.059
Butachlor	104.89	92.87	93.06	97.12	88.68	83.93	94.35	93.56	6.57	7.02	0.020	0.062

Table: 4.21 Recovery of OCs from brinjal at 0.01 ppm

% Recovery											«g g^{-1}	
Compound	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	77.36	76.27	85.06	75.73	76.42	84.40	83.96	79.89	4.33	5.42	0.001	0.004
β-HCH	84.38	77.52	72.62	88.37	75.92	75.19	75.91	78.56	5.65	7.19	0.002	0.005
γ-HCH	84.31	89.04	96.12	82.09	89.25	92.21	87.09	88.59	4.71	5.32	0.001	0.004
δ-HCH	92.71	80.24	97.13	95.85	82.66	73.44	77.10	85.59	9.54	11.15	0.003	0.009
Heptachlor	101.83	84.74	110.48	102.71	90.98	90.91	89.40	95.86	9.22	9.62	0.003	0.009
Aldrin	77.56	72.72	71.37	79.25	79.22	70.88	70.96	74.57	3.93	5.27	0.001	0.004
Heptachlor Epoxide	76.04	81.45	73.03	90.07	71.67	86.92	71.61	78.69	7.56	9.61	0.002	0.007
γ-Chlordane	88.30	71.81	77.82	80.21	86.25	88.66	64.71	79.68	9.02	11.32	0.003	0.008
α-Endosulfan	104.33	106.55	113.48	121.08	107.14	81.68	101.43	105.10	12.21	11.62	0.004	0.012
***p,p*'-DDE**	88.77	92.92	83.32	116.81	80.52	83.82	97.78	91.99	12.48	13.57	0.004	0.012
Dieldrin	82.99	88.09	78.61	103.30	77.81	79.77	79.92	84.36	9.05	10.73	0.003	0.009
Endrin	108.72	93.01	90.71	72.77	90.89	72.25	74.13	86.07	13.64	15.85	0.004	0.013
β-Endosulfan	84.98	89.65	82.63	79.51	88.17	80.93	81.01	83.84	3.88	4.63	0.001	0.004
***p,p*'-DDD**	86.60	75.09	82.94	95.69	72.90	72.77	79.42	80.77	8.38	10.38	0.003	0.008
Endosulfan sulphate	104.78	86.51	86.86	103.47	94.28	88.38	83.23	92.50	8.61	9.31	0.003	0.008
***p,p*-DDT**	87.00	82.84	85.11	86.15	84.03	85.02	83.24	84.77	1.51	1.78	0.0005	0.001
Endrin Ketone	71.63	76.13	74.84	92.93	103.01	100.56	99.73	88.40	13.70	15.50	0.004	0.013
Methoxy chlor	143.22	138.80	157.03	146.19	152.91	151.78	148.42	148.34	6.19	4.17	0.002	0.006

Table: 4.22 Recovery of OCs from brinjal at 0.05 ppm

% Recovery											«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	86.21	86.34	87.48	87.16	87.25	86.20	89.69	87.19	1.23	1.41	0.002	0.006
β-HCH	90.15	89.45	92.24	92.59	89.82	88.63	90.69	90.51	1.45	1.60	0.002	0.007
γ-HCH	86.69	87.92	89.87	88.31	88.69	89.92	90.85	88.89	1.42	1.59	0.002	0.007
δ-HCH	92.85	92.56	94.87	92.05	93.59	93.49	96.63	93.72	1.57	1.67	0.002	0.007
Heptachlor	76.74	75.82	78.11	77.79	78.82	77.74	79.65	77.81	1.26	1.62	0.002	0.006
Aldrin	74.45	75.09	77.44	75.60	76.40	76.34	76.25	75.94	0.98	1.29	0.002	0.005
Heptachlor epoxide	76.86	80.11	77.80	77.75	78.64	78.63	78.55	78.33	1.01	1.29	0.002	0.005
γ-Chlordane	80.84	80.91	81.77	81.76	82.14	83.44	83.50	82.05	1.08	1.31	0.002	0.005
α-Endosulfan	83.76	86.19	83.48	85.04	85.69	83.91	87.07	85.02	1.37	1.61	0.002	0.006
***p,p*'-DDE**	102.64	103.62	105.36	104.86	102.97	103.59	106.97	104.29	1.53	1.47	0.002	0.007
Dieldrin	74.65	74.12	74.23	75.39	74.52	75.26	73.27	74.49	0.72	0.97	0.001	0.003
Endrin	95.54	94.73	97.11	96.20	95.42	96.92	98.75	96.38	1.34	1.39	0.002	0.006
β-Endosulfan	81.12	80.20	81.33	80.83	81.25	78.25	76.68	79.95	1.80	2.25	0.003	0.008
***p,p*'-DDD**	118.68	124.91	122.74	125.29	124.74	129.00	125.48	124.40	3.13	2.52	0.005	0.015
Endosulfan Sulphate	87.56	86.66	87.99	88.70	87.26	87.26	88.25	87.67	0.69	0.79	0.001	0.003
***p,p*'-DDT**	148.82	147.03	146.57	150.62	148.34	141.82	149.55	147.53	2.88	1.95	0.005	0.014
Methoxychlor	76.81	78.36	77.33	77.17	76.20	76.65	78.01	77.22	0.76	0.98	0.001	0.004
Endrin ketone	234.45	243.48	239.94	242.58	234.31	235.22	231.31	237.33	4.66	1.96	0.007	0.022

Table: 4.23 Recovery of OCs from brinjal at 0.1 ppm

% Recovery											«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	84.51	88.53	83.15	86.96	84.28	87.97	81.66	85.29	2.58	3.02	0.008	0.024
β-HCH	87.33	89.19	85.41	88.99	84.37	88.99	84.21	86.93	2.24	2.57	0.007	0.021
γ-HCH	85.28	88.47	84.68	89.06	84.91	88.42	83.48	86.33	2.25	2.61	0.007	0.021
δ-HCH	90.00	94.07	88.68	92.48	90.79	95.54	89.44	91.57	2.54	2.78	0.008	0.024
Heptachlor	76.51	80.80	75.39	82.13	76.05	78.99	75.45	77.90	2.74	3.52	0.009	0.026
Aldrin	74.02	76.90	71.29	76.06	71.77	74.89	70.65	73.65	2.45	3.33	0.008	0.023
Heptachlor epoxide	79.67	82.89	76.73	81.80	78.82	83.05	76.24	79.89	2.80	3.51	0.009	0.026
γ-Chlordane	79.36	81.56	75.32	80.43	74.52	80.44	76.67	78.33	2.79	3.56	0.009	0.026
α-Endosulfan	76.60	74.96	80.76	77.69	82.21	75.63	81.62	78.49	2.99	3.81	0.009	0.028
***p,p*'-DDE**	93.36	95.91	89.00	95.99	90.68	92.99	89.81	92.53	2.82	3.04	0.009	0.027
Dieldrin	73.29	75.24	70.51	74.57	69.41	75.10	68.61	72.39	2.82	3.90	0.009	0.027
Endrin	93.89	93.88	91.62	100.84	91.92	97.56	90.42	94.30	3.69	3.91	0.012	0.035
β-Endosulfan	80.90	81.61	76.31	80.73	75.87	82.60	76.99	79.29	2.80	3.53	0.009	0.026
***p,p*'-DDD**	109.11	116.70	106.82	119.04	107.53	117.27	110.76	112.46	5.07	4.51	0.016	0.048
Endosulfan Sulphate	87.36	90.80	84.79	89.02	86.05	88.23	85.52	87.40	2.12	2.43	0.007	0.020
***p,p*'-DDT**	149.03	143.98	131.67	137.55	127.03	132.98	124.00	135.18	8.98	6.64	0.028	0.085
Methoxychlor	78.07	80.64	74.65	80.80	76.82	79.24	72.83	77.58	3.01	3.88	0.009	0.028
Endrin ketone	188.90	201.41	176.36	191.31	174.14	189.06	172.96	184.87	10.63	5.75	0.033	0.100

corresponding values at 0.25 and 0.5 ppm level showed maximum for malathion (111.98 %) and quinalphos (103.73 %), while minimum for DDVP (100.86 %) and Phorate sulphoxide (83.98 %), respectively. The % RSD was found in the range of 1.90 to 10.54, 0.58 to 4.06 and 10.74 to 11.74 at 0.05, 0.25 and 0.5 ppm level. The LOD for OPs compound was in the range 0.003 to 0.020 at 0.05 ppm whereas LOQ in the range 0.008 to 0.060, at 0.05 of fortification (Table 4.24).

The data obtained from study of SPs+HBs from brinjal were variation the range of 69.85 to 117.42 %, 75.60 to 116.41 % and 87.60 to 119.31 % at 0.1, 0.5 and 1.0 ppm level respectively. Maximum recovery was observed for metalochlor (117.42 %), while minimum for trifluralin (69.85 %) at 0.1 ppm level. The corresponding values at 0.5 and 1.0 ppm level showed maximum for deltamethrin (116.81 %) and fenvalerate-II (119.31%), while minimum for metalochlor (75.60 %) and metalochlor (87.60 %), respectively. The % RSD was found in the range of 9.63 to 19.07, 3.92 to 6.93 and 4.39 to 6.84 at 0.1, 0.5 and 1.0 ppm level, respectively. The LOD for SPs+HBs compound was in the range of 0.007 to 0.0160 at 0.1 ppm whereas LOQ in the range 0.021 to 0.047 at 0.1 ppm level of fortification (Table 4.27).

Table: 4.24 Recovery of OPs from brinjal at 0.05 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	94.45	98.07	97.74	96.2	97.51	97.13	91.25	96.05	2.44	2.55	0.004	0.012
Acephate	93.69	94.90	93.53	93.13	97.79	92.24	94.75	94.29	1.80	1.90	0.003	0.008
Monocrotophos	84.73	118.83	107.33	108.16	101.77	102.29	99.48	103.23	10.36	10.04	0.016	0.049
Dimethoate	57.63	67.31	55.06	72.89	61.34	63.70	64.77	63.24	5.98	9.45	0.009	0.028
Phorate	94.93	105.89	98.20	107.97	100.84	97.90	102.62	101.19	4.64	4.58	0.007	0.022
Me-Parathion	99.64	106.15	104.51	93.00	101.61	105.68	88.03	99.80	6.90	6.91	0.011	0.032
Phorate sulphone	108.41	146.15	114.75	118.80	124.96	119.98	110.17	120.46	12.70	10.54	0.020	0.060
Phorate sulphoxide	97.59	98.73	101.61	103.98	107.78	106.94	119.91	105.22	7.53	7.16	0.012	0.035
Malathion	109.18	108.60	109.18	104.84	105.05	110.84	106.60	107.76	2.29	2.12	0.004	0.011
Chlorpyriphos	99.00	94.67	83.39	102.76	104.96	86.63	89.71	94.45	8.23	8.71	0.013	0.039
Quinalphos	110.31	109.97	117.68	105.44	111.23	121.49	112.78	112.70	5.32	4.72	0.008	0.025
Profenophos	125.82	116.08	126.52	112.07	127.29	121.84	119.69	121.33	5.76	4.75	0.009	0.027
Ethion	98.89	109.72	100.83	100.68	112.01	99.41	110.95	104.64	5.92	5.66	0.009	0.028
Phosalone	137.66	125.59	108.12	121.76	138.17	126.31	143.98	128.80	12.19	9.46	0.019	0.057

Table: 4.25 Recovery of OPs from brinjal at 0.25 ppm

% Recovery											«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	100.21	100.21	100.58	99.86	100.97	102.33	101.83	100.86	0.92	0.91	0.007	0.022
Acephate	100.55	100.55	102.58	100.92	103.15	102.80	103.11	101.95	1.22	1.19	0.010	0.029
Phorate	112.33	112.33	112.28	111.55	112.47	110.64	112.12	111.96	0.65	0.58	0.005	0.015
Monocrotophos	100.99	100.99	101.42	101.59	103.40	100.86	102.07	101.62	0.89	0.88	0.007	0.021
Dimethoate	108.16	108.16	106.35	106.25	110.46	110.58	111.93	108.84	2.20	2.02	0.017	0.052
Chlorpyriphos	108.40	108.40	107.38	106.21	110.56	108.26	106.37	107.94	1.48	1.37	0.012	0.035
Parathion - Me	107.51	107.51	108.46	105.83	110.37	107.34	107.37	107.77	1.38	1.28	0.011	0.033
Malathion	113.17	113.17	110.25	111.41	111.88	111.20	112.78	111.98	1.11	0.99	0.009	0.026
Quinalphos	107.32	107.32	111.52	110.97	108.16	110.76	111.70	109.68	1.99	1.81	0.016	0.047
Ph- Sulphone	109.54	109.54	103.84	108.51	108.33	107.43	107.92	107.87	1.94	1.80	0.015	0.046
Profenophos	108.12	108.12	104.54	104.28	107.65	106.98	104.36	106.29	1.82	1.71	0.014	0.043
Ethion	108.29	108.29	108.86	107.20	109.03	108.19	110.70	108.65	1.08	0.99	0.008	0.025
Triazophos	106.29	106.29	104.08	102.02	105.33	105.20	105.53	104.96	1.50	1.43	0.012	0.035
Phosalone	101.48	101.48	102.78	109.40	112.20	109.05	107.02	106.20	4.31	4.06	0.034	0.101

Table: 4.26 Recovery of OPs from brinjal at 0. 5 ppm

% Recovery										«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	107.66	83.27	100.85	93.76	114.21	92.85	89.20	97.40	10.81	11.10	0.170	0.509
Acephate	108.09	83.43	101.84	89.07	111.00	91.34	87.32	96.01	10.86	11.32	0.171	0.512
Phorate	113.59	87.26	105.43	94.82	113.99	94.48	91.16	100.10	10.86	10.85	0.171	0.512
Monocrotophos	110.51	83.80	102.56	88.31	110.71	93.15	90.05	97.01	10.91	11.25	0.171	0.514
Dimethoate	111.25	86.20	108.48	94.25	116.29	94.62	93.19	100.61	11.26	11.19	0.177	0.530
Chlorpyriphos	114.45	87.56	107.51	94.71	119.67	96.84	93.36	102.01	11.98	11.74	0.188	0.564
Parathion - Me	111.82	82.52	102.04	91.71	112.44	93.92	92.08	98.08	11.15	11.37	0.175	0.525
Malathion	113.76	87.20	106.00	96.09	114.90	93.81	92.97	100.68	10.89	10.81	0.171	0.513
Ph - sulphoxide	93.55	76.92	91.77	75.29	96.53	77.44	76.34	83.98	9.45	11.26	0.148	0.445
Quinalphos	116.48	89.52	113.11	95.53	117.93	96.72	96.84	103.73	11.68	11.26	0.183	0.550
Ph- Sulphone	112.13	84.72	105.83	95.07	114.27	94.39	93.50	99.99	10.93	10.93	0.172	0.515
Profenophos	113.18	86.39	106.18	92.22	111.55	92.01	94.37	99.41	10.68	10.74	0.168	0.503
Ethion	112.03	85.94	108.35	93.92	118.25	94.75	96.59	101.41	11.60	11.44	0.182	0.546
Triazophos	114.49	87.44	110.52	97.26	117.06	94.18	98.24	102.74	11.26	10.96	0.177	0.531
Phosalone	113.44	83.95	109.75	95.70	117.42	97.63	100.90	102.69	11.63	11.32	0.183	0.548

Table: 4.27 Recovery of SPs+HBs from brinjal at 0.1 ppm

	% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluchloralin	64.05	83.5	66.62	70.56	56.7	56.92	90.6	69.85	12.92	18.5	0.013	0.039
Fluchloralin	59.50	66.69	60.04	74.47	82.07	76.00	77.07	70.83	8.82	12.46	0.009	0.026
Alachlor	64.56	82.13	66.62	72.42	75.72	72.15	108.2	77.4	14.76	19.07	0.015	0.044
Metolachlor	102.21	112.77	106.25	120.21	134.79	118.44	127.30	117.42	11.45	9.75	0.011	0.034
Butachlor	65.06	71.06	64.84	76.35	81.73	74.63	82.04	73.67	7.10	9.63	0.007	0.021
Fenpropathrin	62.54	83.38	89.44	88.98	82.71	76.34	104.72	84.02	12.94	15.41	0.013	0.039
λ-Cyhalothrin	74.10	76.29	79.04	85.74	100.02	89.81	102.26	86.75	11.22	12.94	0.011	0.034
β-Cyfluthrin	80.85	76.07	71.32	72.91	71.5	69.65	90.78	76.15	7.46	9.8	0.008	0.022
α-Cypermethrin	92.14	75.29	70.92	70.43	69.84	69.6	90.01	76.89	9.9	12.87	0.010	0.030
Fenvalerate-I	69.88	76.11	74.99	90.81	104.64	97.35	104.86	88.38	14.69	16.62	0.015	0.044
Fenvalerate-II	68.87	76.24	72.53	90.63	105.93	90.68	107.69	87.51	15.63	17.86	0.016	0.047
Deltamethrin	65.71	73.86	72.18	85.19	102.28	92.95	104.37	85.22	15.26	17.91	0.015	0.046

Table: 4.28 Recovery of SPs+HBs from brinjal at 0.5 ppm

% Recovery											«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	88.03	78.73	84.44	77.34	86.65	77.81	88.20	83.03	4.92	5.92	0.077	0.232
Fluchloralin	87.04	75.23	83.48	74.98	84.51	77.29	84.92	81.07	5.06	6.24	0.079	0.238
Alachlor	90.45	78.50	88.99	79.80	88.14	79.10	89.39	84.91	5.46	6.43	0.086	0.257
Metalochlor	80.81	71.49	77.97	70.32	78.07	71.56	79.01	75.60	4.31	5.70	0.068	0.203
Pendimethalin	91.76	80.37	89.54	80.25	89.39	81.20	91.42	86.28	5.38	6.24	0.085	0.254
Butachlor	120.24	112.08	116.19	108.34	113.13	107.37	114.12	113.07	4.44	3.92	0.070	0.209
Bifenthrin	98.44	85.67	96.26	84.26	94.92	86.19	95.23	91.57	5.93	6.48	0.093	0.279
Fenpropethrin	94.33	86.10	92.60	82.60	93.58	84.25	94.35	89.69	5.15	5.75	0.081	0.243
λ-Cyhalothrin	106.21	90.60	102.33	92.17	102.07	92.80	105.03	98.74	6.63	6.72	0.104	0.312
β-Cyfluthrin	109.36	97.79	109.11	98.46	112.51	98.37	108.23	104.83	6.34	6.05	0.100	0.299
α-Cypermethrin	111.37	98.86	109.28	97.46	111.45	97.95	114.08	105.78	7.34	6.93	0.115	0.346
Fenvalerate-I	111.75	101.13	115.04	105.35	114.46	105.90	116.39	110.00	5.86	5.33	0.092	0.276
Fenvalerate-II	116.69	105.71	117.52	107.76	119.56	109.01	119.62	113.70	5.97	5.25	0.094	0.281
Deltamethrin	120.21	108.13	119.08	108.73	123.62	110.33	124.78	116.41	7.17	6.16	0.112	0.337

Table: 4.29 Recovery of SPs+HBs from brinjal at 1.0 ppm

% Recovery											«g g^{-1}	
Compound Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	91.26	87.81	94.86	84.38	94.30	83.30	93.58	89.93	4.79	5.33	0.150	0.451
Fluchloralin	92.15	87.92	95.43	87.27	93.67	85.90	95.29	91.09	4.00	4.39	0.126	0.377
Alachlor	97.88	88.69	101.68	89.26	98.12	90.34	97.33	94.76	5.20	5.48	0.163	0.490
Metalochlor	89.18	81.77	90.17	82.78	91.96	85.01	92.34	87.60	4.37	4.99	0.137	0.411
Pendimethalin	99.66	90.36	101.05	89.18	101.40	89.41	101.61	96.10	6.07	6.32	0.191	0.572
Butachlor	98.16	92.85	103.89	92.39	102.59	94.86	101.10	97.98	4.71	4.81	0.148	0.444
Bifenthrin	98.84	90.98	102.94	89.91	99.95	92.47	103.57	96.95	5.74	5.92	0.180	0.540
Fenpropathrin	100.13	91.42	102.60	88.79	101.05	91.15	101.23	96.63	5.88	6.08	0.185	0.554
λ-Cyhalothrin	105.14	94.54	106.86	93.51	105.98	93.92	105.00	100.71	6.32	6.27	0.198	0.595
β-Cyfluthrin	112.63	100.71	119.91	103.08	112.41	102.86	116.50	109.73	7.50	6.84	0.236	0.707
α-Cypermethrin	112.49	103.25	112.56	104.12	113.97	102.68	114.82	109.13	5.48	5.02	0.172	0.516
Fenvalerate-I	116.44	107.67	117.82	104.18	116.95	104.30	117.18	112.07	6.38	5.69	0.200	0.601
Fenvalerate-II	122.24	113.98	125.51	111.63	126.08	114.05	121.68	119.31	5.97	5.00	0.187	0.562
Deltamethrin	119.66	108.14	119.44	108.92	120.92	110.75	119.74	115.37	5.78	5.01	0.181	0.544

The data obtained from recovery studies of OCs from cotton lint were in the range of 75.25 to 339.11 %, 83.58 to 200.63 % and 76.99 to 242.98 % at 0.01, 0.05 and 0.1 ppm level, respectively. Maximum recovery was observed for *p,p*'-DDT (339.11 %), while minimum for α-endosulfan (75.25 %) at 0.01 ppm level. The corresponding values at 0.05 and at 0.1 ppm level maximum for *p,p'*-DDT (200.63 %) and *p,p'*-DDT (242.98 %), while minimum for α-endosulfan (83.58 %) and *p,p'*-DDE (72.39 %) respectively. The % RSD was found in the range of 3.28 to 18.11, 8.12 to 10.82 and 5.06 to 10.05 at 0.01, 0.05 and 0.1 ppm level, respectively. The LOD for OCs compound was in the range of 0.001 to 0.017 at 0.01 ppm whereas LOQ in the range 0.001 to 0.013 at 0.01ppm level of fortification (Table 4.30).

The data obtained from recovery studies of OPs from cotton lint sample were in the range of 95.56 to 142.98%, 79.57 to 105.97 % and 75.76 to 108.13% at 0.05, 0.1 and 0.5 ppm level, respectively. Maximum recovery was observed for profenophos (142.98 %), while minimum for quinalphos (95.56 %) at 0.05 ppm level. The corresponding values at 0.1 and 0.5 ppm level showed maximum for profenophos (105.97 %) and DDVP (108.13 %), while minimum for DDVP (79.57 %) and phosalone (75.76 %), respectively. The % RSD was found in the range of 7.49 to 11.12, 10.48 to 14.40 and 7.04 to 13.79 at 0.05, 0.1 and 0.5 ppm level. The LOD for OPs compound were in the range 0.01 to 0.02 at 0.05 ppm whereas LOQ in the range 0.03 to 0.07 at 0.05 ppm level fortification (Table 4.33).

The data obtained from recovery studies of SPs from cotton lint were in the range of 71.30 to 100.99 %, 81.50 to 108.03 % and 79.34 to 90.68 % at 0.05, 0.1 and 0.5 ppm level, respectively. Maximum recovery was observed for β-cyfluthrin

Table: 4.30 Recovery of OCs from cotton lint at 0.01 ppm

	% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	91.07	87.25	98.26	97.34	97.45	102.68	92.76	95.26	5.18	5.44	0.002	0.005
β-HCH	77.49	78.61	89.79	88.62	89.81	89.70	84.67	85.53	5.43	6.34	0.002	0.005
γ-HCH	97.82	89.63	103.02	107.02	101.02	106.13	98.37	100.43	5.93	5.90	0.002	0.006
δ-HCH	95.83	97.61	105.05	105.77	106.33	103.68	101.90	102.31	4.12	4.02	0.001	0.004
Heptachlor	120.21	113.31	125.01	131.37	127.17	128.39	125.02	124.36	5.96	4.80	0.002	0.006
Aldrin	111.45	106.45	121.90	122.99	123.89	128.57	114.55	118.54	7.88	6.65	0.002	0.007
Heptachlor epoxide	83.82	87.32	89.66	93.79	90.90	83.42	85.07	87.71	3.91	4.45	0.001	0.004
γ-Chlordane	117.56	117.43	168.28	180.19	184.22	172.23	172.30	158.89	28.78	18.11	0.009	0.027
α-Endosulfan	69.62	74.63	76.57	78.19	79.80	72.90	75.05	75.25	3.38	4.50	0.001	0.003
***p,p*'-DDE**	101.08	95.12	101.65	106.83	108.21	109.40	98.05	102.91	5.40	5.25	0.002	0.005
Dieldrin	87.25	89.42	92.86	95.93	92.19	87.08	86.62	90.19	3.55	3.94	0.001	0.003
Endrin	134.10	127.19	144.99	143.04	144.96	154.91	137.02	140.89	8.98	6.37	0.003	0.008
β-Endosulfan	87.36	88.50	89.18	92.03	94.98	90.86	86.44	89.91	2.95	3.28	0.001	0.003
***p,p*'-DDD**	78.88	83.52	76.00	83.60	87.18	74.85	83.72	81.11	4.58	5.65	0.001	0.004
Endosulfan Sulphate	81.56	81.21	88.21	86.61	89.07	91.12	84.73	86.07	3.77	4.38	0.001	0.004
***p,p*'-DDT**	367.47	307.15	329.70	360.08	331.70	363.35	314.30	339.11	24.54	7.24	0.008	0.023
Methoxychlor	97.86	93.53	97.81	104.04	104.27	102.57	98.60	99.81	3.96	3.97	0.001	0.004
Endrin ketone	322.23	222.46	260.72	308.93	319.59	390.68	293.76	302.62	52.80	17.45	0.017	0.050

Table: 4.31 Recovery of OCs from cotton lint at 0.05 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	91.25	93.51	103.15	116.06	112.90	91.52	97.99	100.91	10.19	10.10	0.016	0.048
β-HCH	87.41	87.99	99.47	106.50	104.38	86.59	91.43	94.82	8.47	8.93	0.013	0.040
γ-HCH	97.77	98.14	111.74	122.70	116.65	96.85	104.70	106.93	10.28	9.61	0.016	0.048
δ-HCH	92.45	95.04	106.43	120.43	114.63	94.25	101.79	103.57	10.81	10.44	0.017	0.051
Heptachlor	121.89	120.01	137.61	152.94	143.18	115.75	126.39	131.11	13.73	10.47	0.022	0.065
Aldrin	92.24	91.60	108.18	114.64	109.12	90.48	95.65	100.27	10.03	10.01	0.016	0.047
Heptachlor epoxide	88.70	86.64	101.10	107.26	104.99	85.27	93.25	95.32	9.07	9.52	0.014	0.043
γ-Chlordane	103.56	103.30	113.40	123.83	118.15	97.86	106.42	109.50	9.26	8.45	0.015	0.044
α-Endosulfan	76.94	76.35	89.68	97.32	91.04	73.51	80.20	83.58	9.04	10.82	0.014	0.043
***p,p*'-DDE**	92.40	90.44	97.80	105.74	103.00	83.65	91.39	94.92	7.71	8.12	0.012	0.036
Dieldrin	87.44	88.58	97.52	105.57	102.06	82.15	88.92	93.18	8.61	9.24	0.014	0.041
Endrin	124.16	120.88	135.63	148.66	144.03	114.69	123.51	130.22	12.71	9.76	0.020	0.060
β-Endosulfan	87.80	87.26	97.61	108.71	95.95	82.64	85.53	92.21	9.09	9.85	0.014	0.043
***p,p*'-DDD**	84.21	80.54	96.00	99.57	96.60	79.33	83.29	88.50	8.54	9.65	0.013	0.040
Endosulfan Sulphate	89.80	87.29	100.61	108.44	101.96	82.65	89.67	94.35	9.35	9.92	0.015	0.044
***p,p*'-DDT**	199.05	196.81	209.83	235.19	210.28	171.33	181.90	200.63	20.81	10.37	0.033	0.098
Methoxychlor	89.85	86.65	98.00	107.36	102.97	83.58	86.58	93.57	9.21	9.84	0.014	0.043
Endrin ketone	193.23	183.00	193.97	222.10	201.35	166.43	178.15	191.18	17.89	9.36	0.028	0.084

Table: 4.32 Recovery of OCs from cotton lint at 0.1 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
α-HCH	88.10	93.81	97.20	87.36	87.71	81.05	81.05	88.04	5.99	6.80	0.019	0.056
β-HCH	90.37	93.01	94.11	85.55	87.33	82.01	82.01	87.77	4.93	5.62	0.015	0.046
γ-HCH	99.60	104.30	105.17	95.43	96.50	89.03	89.03	97.01	6.54	6.74	0.021	0.062
δ-HCH	95.59	102.48	106.76	92.89	95.04	89.93	89.93	96.09	6.35	6.61	0.020	0.060
Heptachlor	126.99	134.52	138.30	122.28	124.58	111.29	111.29	124.18	10.41	8.38	0.033	0.098
Aldrin	95.94	99.48	99.87	87.38	91.65	86.80	86.80	92.56	5.87	6.34	0.018	0.055
Heptachlor epoxide	93.72	99.55	97.19	85.19	89.35	83.30	83.30	90.23	6.70	7.43	0.021	0.063
γ-Chlordane	104.38	108.55	111.53	96.29	97.50	93.59	93.59	100.77	7.34	7.28	0.023	0.069
α-Endosulfan	86.43	88.95	85.55	74.68	79.87	75.28	75.28	80.86	6.06	7.49	0.019	0.057
***p,p*'-DDE**	98.19	101.80	98.38	84.88	89.31	86.13	86.13	92.12	7.09	7.70	0.022	0.067
Dieldrin	95.05	98.86	96.92	82.51	89.87	82.19	82.19	89.65	7.41	8.26	0.023	0.070
Endrin	135.67	135.94	132.97	117.72	123.36	118.86	118.86	126.20	8.35	6.61	0.026	0.079
β-Endosulfan	102.50	101.89	102.31	86.52	92.23	90.52	90.52	95.21	6.79	7.13	0.021	0.064
***p,p*'-DDD**	82.34	81.47	78.76	72.90	76.00	73.72	73.72	76.99	3.89	5.06	0.012	0.037
Endosulfan Sulphate	97.10	97.50	95.62	84.01	91.14	85.37	85.37	90.87	5.96	6.56	0.019	0.056
***p,p*'-DDT**	261.41	269.40	268.91	226.20	245.27	214.84	214.84	242.98	24.43	10.05	0.077	0.230
Methoxychlor	97.57	97.93	94.16	85.52	87.14	84.33	84.33	90.14	6.19	6.87	0.019	0.058
Endrin ketone	234.16	244.26	235.67	200.11	219.30	194.05	194.05	217.37	21.33	9.81	0.067	0.201

Table: 4.33 Recovery of OPs from cotton lint at 0.05 ppm

% Recovery											«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	87.00	95.53	99.41	110.14	91.56	94.93	96.22	96.40	7.22	7.49	0.01	0.03
Phorate	86.67	89.94	100.78	111.56	88.04	94.30	98.18	95.64	8.75	9.14	0.01	0.04
Dimethoate	114.14	121.61	142.93	157.68	122.92	132.05	135.83	132.45	14.73	11.12	0.02	0.07
Chlorpyriphos	87.65	91.80	99.88	113.48	89.74	92.06	99.89	96.36	8.92	9.25	0.01	0.04
Me-parathion	94.77	98.48	109.63	121.44	98.82	101.00	106.38	104.36	9.05	8.68	0.01	0.04
Malathion	103.28	107.39	118.13	129.48	101.96	106.35	110.19	110.97	9.74	8.78	0.02	0.05
Quinalphos	85.37	94.45	101.71	111.99	84.89	93.79	96.73	95.56	9.41	9.85	0.01	0.04
Phorate sulphone	95.61	98.03	114.17	120.65	100.26	107.63	110.15	106.64	9.15	8.58	0.01	0.04
Profenophos	132.94	134.20	152.94	166.48	127.56	141.62	145.14	142.98	13.38	9.35	0.02	0.06
Ethion	88.47	89.17	104.15	113.90	91.64	98.05	99.80	97.88	9.16	9.36	0.01	0.04
Triazophos	114.64	115.50	137.23	148.09	118.80	121.80	131.39	126.78	12.57	9.91	0.02	0.06
Phosalone	118.58	111.62	127.42	140.76	103.92	117.79	127.05	121.02	12.00	9.92	0.02	0.06

Table: 4.34 Recovery of OPs from cotton lint at 0.1 ppm

% Recovery										«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	85.97	78.32	78.98	73.78	95.44	76.29	68.21	79.57	8.83	11.10	0.028	0.08
Phorate	88.05	79.75	81.80	76.04	99.50	77.14	72.60	82.12	9.09	11.06	0.029	0.09
Dimethoate	111.93	101.42	105.08	97.38	124.11	99.18	89.58	104.10	11.18	10.74	0.035	0.11
Chlorpyriphos	93.69	83.75	87.09	80.77	103.83	84.31	76.25	87.10	9.13	10.48	0.029	0.09
Me-parathion	97.01	87.12	92.13	84.24	109.28	86.58	81.16	91.07	9.56	10.50	0.030	0.09
Malathion	104.38	93.82	98.71	88.62	117.30	91.09	82.38	96.61	11.53	11.94	0.036	0.11
Quinalphos	92.31	82.58	86.73	76.20	101.02	78.76	72.51	84.30	9.90	11.74	0.031	0.09
Phorate sulphone	103.63	92.07	93.75	86.32	116.15	87.76	79.06	94.11	12.30	13.07	0.039	0.12
Profenophos	117.20	100.51	110.95	96.17	128.87	98.80	89.28	105.97	13.73	12.96	0.043	0.13
Ethion	90.70	84.63	84.53	77.31	103.25	79.35	73.10	84.70	9.98	11.78	0.031	0.09
Triazophos	112.04	101.52	104.28	97.01	123.89	99.16	89.00	103.84	11.28	10.86	0.035	0.11
Phosalone	106.02	90.89	93.49	84.26	113.13	86.57	73.74	92.59	13.33	14.40	0.042	0.13

Table: 4.35 Recovery of OPs from cotton lint at 0.5 ppm

% Recovery											«g g^{-1}	
Comp. Name	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
DDVP	110.63	94.99	112.83	108.80	100.31	114.53	114.80	108.13	7.61	7.04	0.12	0.36
Phorate	72.43	60.96	76.08	77.65	74.32	87.72	90.68	77.12	9.90	12.84	0.16	0.47
Dimethoate	70.00	78.11	71.97	80.48	77.86	93.99	98.59	81.57	10.78	13.21	0.17	0.51
Chlorpyriphos	65.56	74.14	70.14	78.81	76.65	93.19	93.86	78.91	10.88	13.79	0.17	0.51
Me-parathion	67.06	76.26	70.82	78.04	74.63	90.53	92.47	78.55	9.57	12.19	0.15	0.45
Malathion	74.33	74.52	74.40	82.75	80.06	94.12	97.04	82.46	9.56	11.59	0.15	0.45
Quinalphos	72.13	76.48	71.80	79.28	76.03	91.74	93.81	80.18	9.00	11.23	0.14	0.42
Phorate sulphone	74.42	79.79	74.90	82.61	79.02	94.46	98.29	83.35	9.39	11.27	0.15	0.44
Profenophos	84.32	67.03	83.20	88.70	85.87	96.86	95.85	85.98	9.94	11.56	0.16	0.47
Ethion	72.80	72.05	72.24	79.52	77.47	92.33	94.96	80.20	9.64	12.02	0.15	0.45
Triazophos	69.95	78.63	67.87	75.88	73.66	87.34	89.85	77.60	8.34	10.75	0.13	0.39
Phosalone	68.19	71.13	66.03	74.76	74.03	86.88	89.32	75.76	8.99	11.87	0.14	0.42

Table: 4.36 Recovery of SPs from cotton lint at 0.05 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-IV**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	90.04	80.40	56.11	58.57	70.09	70.71	73.17	71.30	11.78	16.52	0.019	0.056
Fenpropathrin	91.84	82.61	49.84	57.39	72.93	77.59	75.89	72.58	14.47	19.93	0.023	0.068
λ-Cyhalothrin	96.64	89.54	63.78	79.99	78.40	79.04	79.13	80.93	10.24	12.65	0.016	0.048
β-Cyfluthrin	112.10	108.98	85.38	116.40	94.03	94.32	95.74	100.99	11.47	11.35	0.018	0.054
α-Cypermethrin	90.74	81.86	64.11	69.37	75.81	74.31	77.06	76.18	8.57	11.25	0.013	0.040
Fenvalerate-I	115.58	83.48	78.45	89.62	69.46	76.22	76.83	84.23	15.18	18.03	0.024	0.072
Fenvalerate-II	121.44	103.92	71.10	78.22	87.23	89.93	85.91	91.11	16.80	18.44	0.026	0.079
Deltamethrin	89.71	86.41	65.45	61.84	78.79	84.27	79.31	77.97	10.55	13.53	0.017	0.050

Table: 4.37 Recovery of SPs from cotton lint at 0.1 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	89.49	86.68	90.87	87.71	93.27	91.28	94.41	90.53	2.80	3.10	0.009	0.026
Fenpropathrin	79.00	76.97	82.38	78.14	85.64	85.28	83.06	81.50	3.48	4.27	0.011	0.033
λ-Cyhalothrin	100.89	93.90	96.76	93.91	97.22	100.50	99.65	97.55	2.93	3.00	0.009	0.028
β-Cyfluthrin	102.06	107.12	107.95	108.00	109.15	111.71	110.23	108.03	3.06	2.83	0.010	0.029
α-Cypermethrin	100.87	92.36	96.25	94.60	95.03	102.77	97.11	97.00	3.65	3.76	0.011	0.034
Fenvalerate-I	97.43	79.44	84.42	85.70	86.92	84.97	86.26	86.45	5.43	6.28	0.017	0.051
Fenvalerate-II	98.06	101.67	103.11	99.06	100.84	110.30	107.15	102.89	4.41	4.29	0.014	0.042
Deltamethrin	104.26	106.13	104.58	106.69	108.57	115.08	109.43	107.82	3.72	3.45	0.012	0.035

(100.99 %), while minimum for bifenthrin (71.30 %) at 0.05 ppm level. The corresponding values at 0.1 and 0.5 ppm, showed maximum for β-cyfluthrin (108.03 %) and fenvalerate-II (90.68 %), while minimum for fenpropathrin (81.50 %) and α-cypermethrin (79.34 %), respectively. The % RSD was found in the range of 11.25 to 19.93, 2.83 to 6.28 and 8.59 to 13.30 at 0.05, 0.1 and 0.5 ppm level, respectively. The LOD for SPs compound was in the range 0.013 to 0.026 at 0.05 ppm LOQ in the range 0.040 to 0.079 at 0.05 ppm level of fortification. (Table 4.36).

The data obtained from recovery studies of HBs from cotton lint sample were in the range of 78.07 to 146.43 %, 72.83 to 104.50 % and 72.91 to 89.85 % at 0.05, 0.1 and 0.5 ppm level, respectively. Maximum recovery was observed for butachlor (140.43 %), while minimum for fluchloralin (87.60 %) at 0.05 ppm level. The corresponding values at 0.1 and 0.5 ppm level showed maximum for butachlor (104.50 %) and butachlor (89.85 %), while minimum for fluchloralin (72.83 %) and fluchloralin (72.91 %). The % RSD was found in the range of 2.51 to 5.29, 2.51 to 5.29 and 4.32 to 7.13 at 0.05, 0.1 and 0.5 ppm level, respectively. The LOD for HBs compound was in the range 0.003 to 0.009 at 0.05 ppm whereas LOQ in the range of and 0.010 to 0.026 at 0.05 ppm level of fortification (Table 4.39).

Table: 4.38 Recovery of SPs from cotton lint at 0.5 ppm

	% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Bifenthrin	84.59	80.10	70.89	75.26	70.88	92.31	93.15	81.03	9.37	11.56	0.147	0.442
Fenpropathrin	88.81	83.28	75.93	79.94	74.25	94.55	96.17	84.71	8.72	10.30	0.137	0.411
λ-Cyhalothrin	97.76	80.39	73.70	75.36	69.49	90.90	93.87	83.07	11.05	13.30	0.174	0.521
β-Cyfluthrin	85.59	81.19	75.01	77.77	74.52	88.96	92.77	82.26	7.07	8.60	0.111	0.333
α-Cypermethrin	84.32	76.74	72.28	72.70	70.13	91.01	88.20	79.34	8.41	10.61	0.132	0.397
Fenvalerate-I	100.20	82.92	79.70	77.03	74.37	93.06	92.52	85.69	9.65	11.26	0.152	0.455
Fenvalerate-II	99.03	89.50	82.76	84.20	82.57	96.88	99.83	90.68	7.79	8.59	0.122	0.367
Deltamethrin	81.74	80.10	72.06	76.41	71.09	93.67	89.38	80.63	8.47	10.50	0.133	0.399

Table: 4.39 Recovery of HBs from cotton lint at 0.05 ppm

	% Recovery										«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	88.86	76.33	79.42	77.57	80.29	87.14	86.83	82.35	2.60	3.30	0.004	0.012
Fluchloralin	83.76	72.45	74.37	74.50	77.07	80.25	84.13	78.07	2.91	4.00	0.005	0.014
Alachlor	89.15	81.71	83.69	81.44	87.25	84.37	92.29	85.70	2.17	2.51	0.003	0.010
Metalochlor	112.54	83.37	101.90	87.14	97.85	102.87	104.99	98.67	2.75	2.96	0.004	0.013
Pendimethalin	91.18	82.81	85.22	83.27	89.10	86.41	88.58	86.65	4.12	5.02	0.006	0.019
Butachlor	131.05	132.92	143.07	137.85	147.46	143.83	146.80	140.43	5.53	5.29	0.009	0.026

Table: 4.40 Recovery of HBs from cotton lint at 0.1 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	78.37	73.80	82.02	79.37	78.27	80.74	77.94	78.64	2.60	3.30	0.008	0.024
Fluchloralin	73.11	68.26	74.45	73.39	73.11	77.30	70.19	72.83	2.91	4.00	0.009	0.027
Alachlor	86.46	81.78	86.95	87.55	85.27	87.90	87.76	86.24	2.17	2.51	0.007	0.020
Metalochlor	91.31	90.63	91.01	90.64	92.80	96.62	96.80	92.83	2.75	2.96	0.009	0.026
Pendimethalin	84.27	73.13	83.23	85.37	82.03	83.94	83.31	82.18	4.12	5.02	0.013	0.039
Butachlor	102.38	94.79	104.93	109.09	103.55	104.36	112.39	104.50	5.53	5.29	0.017	0.052

Table: 4.41 Recovery of HBs from cotton lint at 0.5 ppm

% Recovery											«g g^{-1}	
Compounds	**R-I**	**R-II**	**R-III**	**R-VI**	**R-V**	**R-VI**	**R-VII**	**Mean**	**SD**	**RSD**	**LOD**	**LOQ**
Trifluralin	85.39	79.97	84.28	77.81	79.36	73.76	73.60	79.17	4.61	5.83	0.072	0.217
Fluchloralin	78.92	73.94	80.31	70.47	72.19	67.02	67.54	72.91	5.20	7.13	0.082	0.245
Alachlor	93.61	90.03	96.03	86.20	89.71	81.47	84.92	88.85	5.05	5.69	0.079	0.238
Metalochlor	91.04	89.75	92.33	87.39	90.25	83.60	82.41	88.11	3.81	4.32	0.060	0.180
Pendimethalin	89.93	82.31	88.58	83.08	83.66	78.06	80.21	83.69	4.26	5.09	0.067	0.201
Butachlor	96.43	88.37	94.98	88.85	89.61	85.03	85.71	89.85	4.34	4.83	0.068	0.205

Table: 4.42 Limit Of Quantitation (LOQ)

(A) Organochlorines

Compound	**Water**		**Soil**			**Brinjal**			**Cotton lint**		
	Level of fortification ($\mu g\ L^{-1}$)		Level of fortification ($\mu g\ g^{-1}$)			Level of fortification ($\mu g\ g^{-1}$)			Level of fortification ($\mu g\ g^{-1}$)		
	0.1	0.5	0.01	0.05	0.1	0.01	0.05	0.1	0.01	0.05	0.1
α-HCH	0.028	0.164	0.0055	0.012	0.088	0.004	0.006	0.024	0.005	0.048	0.056
β-HCH	0.032	0.357	0.0027	0.015	0.081	0.005	0.007	0.021	0.005	0.040	0.046
γ-HCH	0.036	0.335	0.0023	0.014	0.064	0.004	0.007	0.021	0.006	0.048	0.062
δ-HCH	0.037	0.274	0.0034	0.012	0.161	0.009	0.007	0.024	0.004	0.051	0.060
Heptachlor	0.026	0.364	0.0026	0.008	0.074	0.009	0.006	0.026	0.006	0.065	0.098
Aldrin	0.029	0.301	0.0021	0.009	0.075	0.004	0.005	0.023	0.007	0.047	0.055
Heptachlor epoxide	0.025	0.306	0.0017	0.014	0.077	0.007	0.005	0.026	0.004	0.043	0.063
γ-Chlordane	0.026	0.307	0.0016	0.014	0.069	0.008	0.005	0.026	0.027	0.044	0.069
α-Endosulfan	0.027	0.317	0.0020	0.009	0.047	0.012	0.006	0.028	0.003	0.043	0.057
p,p'-DDE	0.030	0.359	0.0015	0.015	0.087	0.012	0.007	0.027	0.005	0.036	0.067
Dieldrin	0.031	0.232	0.0016	0.010	0.088	0.009	0.003	0.027	0.003	0.041	0.070
Endrin	0.036	0.273	0.0048	0.017	0.176	0.013	0.006	0.035	0.008	0.060	0.079
β-Endosulfan	0.028	0.395	0.0030	0.009	0.107	0.004	0.008	0.026	0.003	0.043	0.064
p,p'-DDD	0.043	0.357	0.0021	0.013	0.094	0.008	0.015	0.048	0.004	0.040	0.037
Endosulfan Sulfate	0.026	0.478	0.0045	0.015	0.126	0.008	0.003	0.020	0.004	0.044	0.056
p,p'-DDT	0.023	0.331	0.0054	0.010	0.081	0.001	0.014	0.085	0.023	0.098	0.230
Methoxychlor	0.020	0.678	0.0043	0.016	0.130	0.013	0.004	0.028	0.004	0.043	0.058
Endrin ketone	0.038	0.334	0.0080	0.015	0.177	0.006	0.022	0.100	0.050	0.084	0.201

Table: 4.42 (cont.)

(B) Organophosphates

	Water		**Soil**			**Brinjal**			**Cotton lint**		
	Level of fortification ($\mu g\ L^{-1}$)		Level of fortification ($\mu g\ g^{-1}$)			Level of fortification ($\mu g\ g^{-1}$)			Level of fortification ($\mu g\ g^{-1}$)		
	0.1	0.5	0.01	0.05	0.1	0.01	0.25	0.5	0.05	0.1	0.5
DDVP	0.135	0.053	-	-	-	0.012	0.22	0.509	0.03	0.08	0.36
Acephate	0.172	0.054	-	-	-	0.008	0.029	0.512	-	-	-
Monocrotophos	0.117	0.073	-	-	-	0.049	0.015	0.514	-	-	-
Dimethoate	0.153	0.127	0.018	0.055	0.049	0.028	0.021	0.530	0.07	0.11	0.51
Phorate	0.144	0.050	0.014	0.720	0.151	0.022	0.052	0.512	0.04	0.09	0.47
Me-parathion	0.146	0.072	0.01	0.086	0.103	0.032	0.033	0.525	0.04	0.09	0.45
Phorate sulphone	0.112	0.101	0.007	0.063	0.079	0.060	0.046	0.515	0.04	0.12	0.44
Phorate sulphoxie	-	-	-	-	-	0.035	-	0.445	-	-	-
Malathion	0.149	0.111	0.009	0.079	0.050	0.011	0.026	0.513	0.05	0.11	0.45
Chlorpyriphos	0.102	0.080	0.008	0.064	0.074	0.039	0.035	0.564	0.04	0.09	0.51
Quinalphos	0.116	0.059	0.004	0.049	0.086	0.025	0.047	0.550	0.04	0.09	0.42
Profenophos	0.118	0.089	0.012	0.074	0.062	0.027	0.043	0.503	0.06	0.13	0.47
Ethion	0.117	0.120	0.007	0.045	0.095	0.028	0.025	0.546	0.04	0.09	0.45
Triazophos	0.136	0.151	0.016	0.067	0.078	-	0.035	0.531	0.06	0.11	0.39
Phosalone	0.157	0.339	-	0.063	0.144	0.057	0.101	0.548	0.06	0.13	0.42

Table: 4.42 (cont.)

(C) Synthetics and Herbicides

Compound	Water		Soil			Brinjal			Cotton lint		
	Level of fortification (μg L^{-1})		Level of fortification (μg g^{-1})			Level of fortification (μg g^{-1})			Level of fortification (μg g^{-1})		
	0.1	0.5	0.01	0.05	0.1	0.1	0.5	1.0	0.05	0.1	0.5
Herbicides											
Trifluralin	0.053	0.191	0.006	0.032	0.072	0.039	0.232	0.451	0.012	0.024	0.217
Fluchloralin	0.086	0.232	0.005	0.027	0.063	0.026	0.238	0.377	0.014	0.027	0.245
Alachlor	0.092	0.170	0.008	0.023	0.060	0.044	0.257	0.490	0.010	0.020	0.238
Metalochlor	0.063	0.130	0.013	0.026	0.050	0.034	0.203	0.411	0.013	0.026	0.180
Pendimethalin	0.075	0.236	0.004	0.030	0.059	-	0.254	0.572	0.019	0.039	0.201
Butachlor	0.060	0.193	0.005	0.025	0.062	0.021	0.209	0.444	0.026	0.052	0.205
Bifenthrin	0.022	0.075	0.014	0.050	0.021	-	0.279	0.540	0.056	0.026	0.442
Synthetic pyrethroids											
Fenpropathrin	0.029	0.114	-	0.020	0.032	0.039	0.243	0.554	0.068	0.033	0.411
λ-Cyhalothrin	0.021	0.115	0.010	0.050	0.035	0.034	0.312	0.595	0.048	0.028	0.521
β-Cyfluthrin	0.027	0.169	0.010	0.060	0.036	0.022	0.299	0.707	0.054	0.029	0.333
α-Cypermethrin	0.092	0.089	0.011	0.060	0.034	0.030	0.346	0.516	0.040	0.034	0.397
Fenvalerate-I	0.024	0.086	0.011	0.050	0.030	0.044	0.276	0.601	0.072	0.051	0.455
Fenvalerate-II	0.023	0.171	0.013	0.050	0.033	0.047	0.281	0.562	0.079	0.042	0.367
Deltamethrin	0.024	0.120	0.010	0.070	0.037	0.046	0.337	0.5440	0.050	0.035	0.399

IV. RESULTS AND DISCUSSION

Use of agrochemical has no doubt increased the agricultural production in general but persistent residues of these chemicals have not only jeopardized the human health but raised serious issues on the adverse impact on the environment. Considerable attention has been focused on the threat to human life due to the presence of agricultural chemicals in the dietary food, drinking water and the residential risk. Pesticides and their contamination in food products are regulated through some concept like Maximum Residue Limits (MRLs), Average Daily Intake (ADIs) and Good Agricultural Practices (GAPs). MRL is the maximum concentration of pesticide residue resulting from the studies conducted on the use of pesticides according to GAP. It is the limit that is legally permitted to recognize as acceptable in or on food, agricultural commodities, or animal food. An attempt was made in the present investigation to know the status of pesticide residues and heavy metals in plant, soil and water. The study was undertaken with the following objectives.

OBJECTIVES:

(1) Estimation of nitrate-nitrogen, heavy metals and pesticide residues in ground water collected from cotton and vegetable growing areas of Padra, Karjan, Nadiad and Kheda of middle Gujarat.

(2) Status of phosphorus, heavy metals and pesticide residues in surface water.

(3) Estimation of heavy metals and pesticide residues in soil.

(4) Estimation of heavy metals and pesticide residues from vegetables and cotton lint.

Table: 4.1 Scope of the agrochemicals and other contaminants for water, soil, vegetable and cotton lint samples

Sr. No.	Contaminant	Scope
1	Heavy metals	Nickel (Ni), cobalt (Co), lead (Pb), cadmium (Cd), chromium (Cr)
2	Nitrate-nitrogen	NO_3-N from ground water collected from cotton and vegetable growing areas of middle Gujarat
3	Phosphorus	from surface water collected from cotton and vegetable growing areas of middle Gujarat.
4	Pesticides	**(i)** OCs: α-HCH, γ-HCH, β-HCH, heptachlor, δ-HCH, aldrin, heptachlor epoxide, α-chlordane, α-endosulfan, *p,p'*-DDE, dieldrin, endrin, *p,p'*-DDD, β-endosulfan, *p,p'*-DDT, endosulfan sulfate, endrin ketone, methoxychlor **(ii)** OPs: DDVP, phorate, dimethoate, parathion methyl, fenitrothion, phorate sulphone, malathion, phorate sulphoxide, chlorpyrifos, chlorfenvinphos, profenophos, ethion, quinalphos, triazophos, phosalone **(iii)** SPs:Bifenthrin, fenpropathrin, λ-cyhalothrin, β-cyfluthrin, α-cypermethrin, fenvalerate, deltamethrin **(iv)** HBs: Trifluralin, fluchloralin, alachlor, metolachlor, butachlor, pendimethylene

The detail results obtained from the present investigation are reported in this chapter along with statistical inferences. The results are presented and discussed under the respective sub-heads.

4.1 Validation of multi-residue methods for pesticides for different matrices

Method validation is the process used to confirm that the analytical procedure employed for a specific test is suitable for its intended use. Results from method validation can be used to judge the quality, reliability, and consistency of analytical results; it is an integral part of any good analytical practice (Huber, 2007**)**. Prior to analyzing unknown samples for the presence of pesticide residues, several validation parameters were determined viz., linearity, accuracy (mean % recovery), precision as % Relative Standard Deviation (%RSD_{wR}), limit of detection - LOD and limit of quantitation - LOQ.

A linearity study was performed to determine the performance of ECD and FPD detectors of GLC. For the linearity study, a graph of detector's response v/s concentration of pesticide mixture was plotted and correlation coefficient and equation were determined.

Accuracy of the methods employed for the analysis of pesticides from different matrices was determined by recovery studies. Mean recovery obtained from such studies reflects the accuracy of the methods. Precision of the analytical method was reflected by % RSD_{wR}.

Before quantitation of pesticide from unknown samples *viz*., soil, water and vegetable the LOD and LOQ were worked out. The limits of detection (LODs) and quantitation (LOQs) were calculated in accordance with Taylor (1987). For this purpose, 7 independent analyses of different samples spiked with different group of pesticide at different levels were performed. The LOD and LOQ were calculated from the standard deviation of these determinations. The limit of detection (LOD) and limit of quantitation (LOQ) was worked out in order to detect and quantify the minimum amount of pesticide residues with optimum confidence level. The LOD & LOQ were calculated as below:

$$\textbf{LOD}\ (\mu g\ g^{-1}) = \frac{(\text{SD x Fortification level, } \mu g\ g^{-1})}{100} \times t_{(99.5)\ (n-1)}$$

$$\textbf{LOQ}\ (\mu g\ g^{-1}) = \text{LOD} \times 3.0$$

4.1.1 Linearity study

To establish the linearity of different group of pesticides on electron capture detector and flame photometric detector, equal volume of seven different concentrations of the pesticide *viz.*, 0.01, 0.025, 0.05, 0.1, 0.25, 0.5 and 1.0 ppm were injected and their corresponding responses (μV) were recorded. As per linearity study organochlorines (OCs), organophosphates (OPs) and synthetic pyrethroids (SPs) along with herbicides (HBs) were found linear in the range of 0.01 to 0.1, 0.01 to 1.0 and 0.1 to 1.0 ppm. The regression equation and R^2 values for different pesticide presented in Table 4.2.

4.1.2 Recovery studies

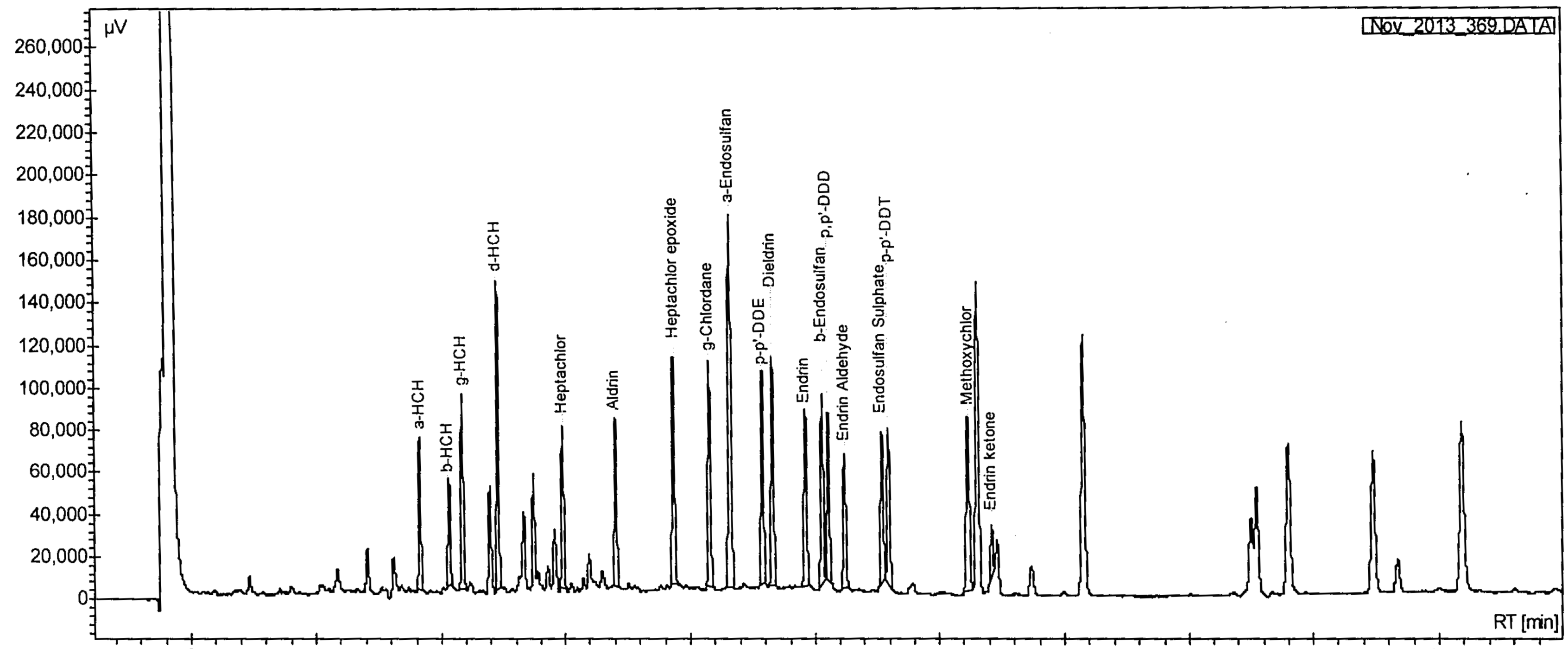
4.1.2.1 Recovery study of pesticides from water, soil, vegetable and cotton lint following multiresidue methods

Prior to assessment of pesticide residues different matrices *viz.*, water, soil, vegetable and cotton lint were subjected to recovery study. Water was spiked at 2 different levels i.e. 0.1 and 0.5 ppb and other commodity were spiked at 3 different levels i.e. 0.01, 0.05 and 0.1 ppm and further analysed with method mentioned in section 3.6 of materials and methods chapter. The data obtained from the study are mentioned in Tables-4.3 to 4.41. The recovery experiment was performed with 7 replications along with a control and reagent blank.

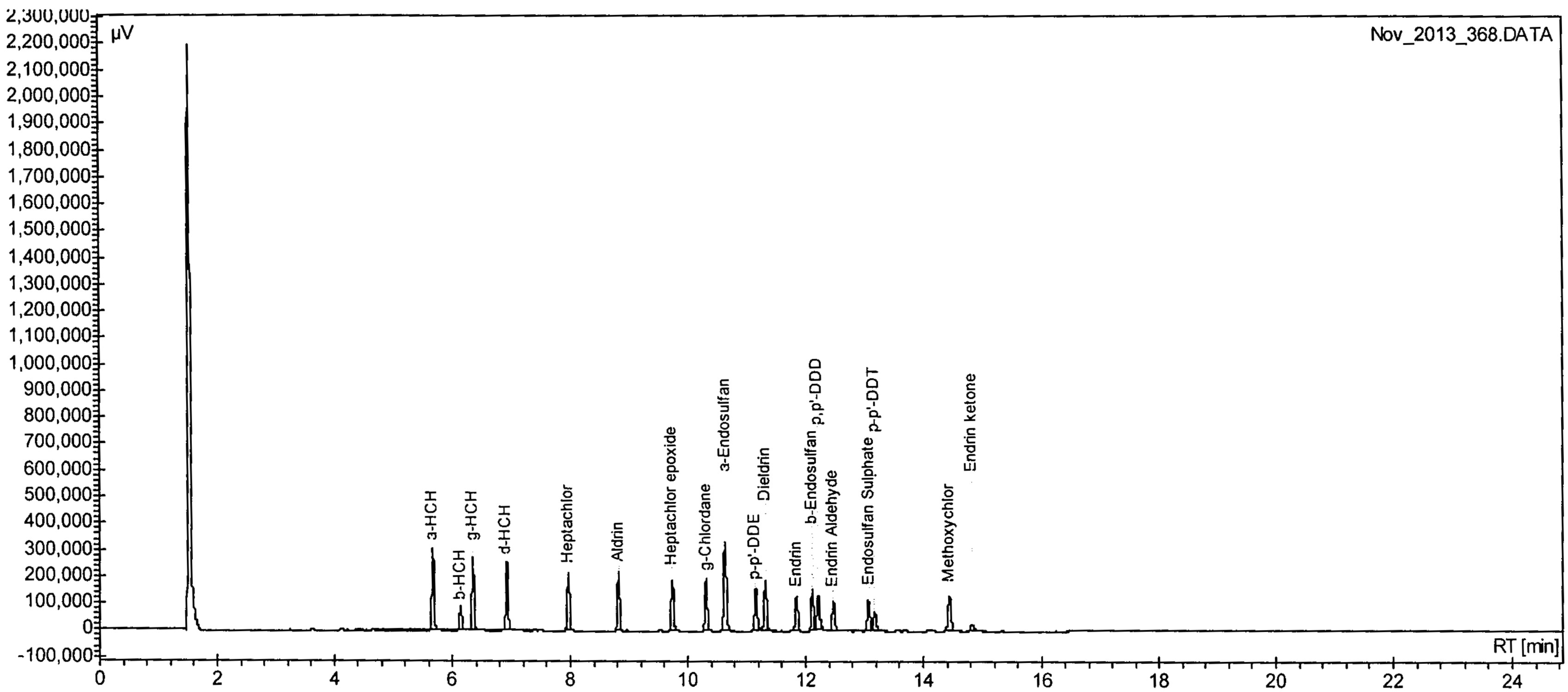
The mean recovery of OCs, OPs, SPs, and HBs from water, soil, vegetable fruits and cotton lint samples has been mentioned in the respective table at each spiking level. Per cent RSD_{WR} has been recorded for each spiking level. LOD and LOQ were also determined for different compounds viz., OCs, OPs, SPs and HBs and are presented in the tables 4.3 to 4.41. The analytical methods employed for the extraction of water, soil, vegetable fruits and cotton lint samples were found accurate and precise as mean recoveries and % RSD_{WR} were well within the limits prescribed by the SANCO guide

Table: 4.2 Regression equation and R^2 values of pesticides

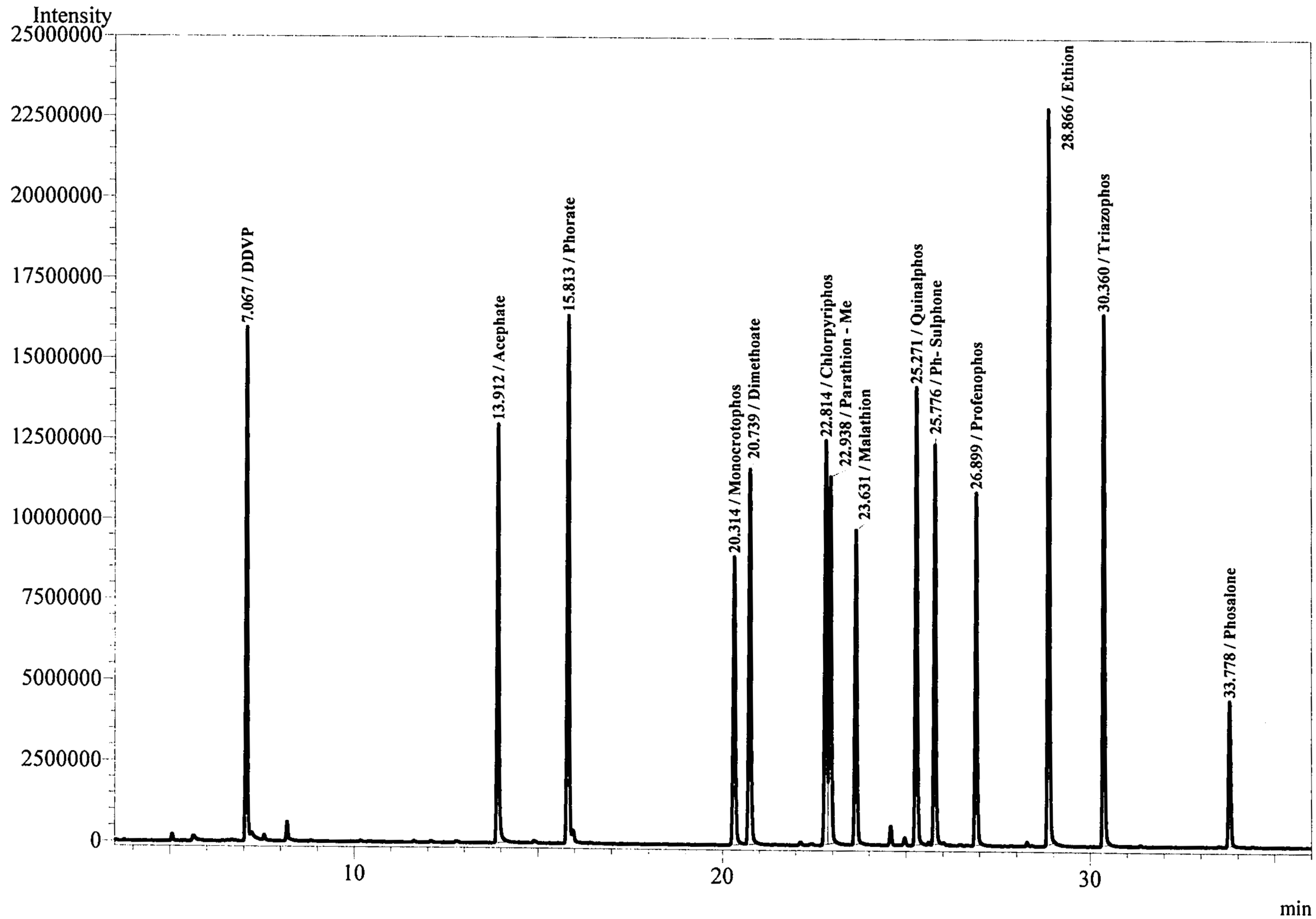
	Pesticides	Regression equation	R^2
(A)	**Organochlorines**		
	α-HCH	y = 5E+06x - 24397	0.995
	β-HCH	y = 1E+06x - 1697	0.999
	γ-HCH	y = 4E+06x - 18439	0.995
	δ-HCH	y = 4E+06x - 19364	0.994
	Heptachlor	y = 4E+06x - 15516	0.994
	Aldrin	y = 3E+06x - 12653	0.998
	Heptachlor epoxide	y = 3E+06x - 10469	0.997
	γ-Chlordane	y = 3E+06x - 13899	0.993
	α-Endosulfan	y = 2E+06x - 8152	0.997
	α-Chlordane	y = 3E+06x - 10097	0.997
	p,p′-DDE	y = 3E+06x - 11523	0.995
	Dieldrin	y = 3E+06x - 11426	0.996
	Endrin	y = 2E+06x - 5886	0.997
	β-Endosulfan	y = 2E+06x - 6196	0.998
	p,p′-DDD	y = 2E+06x - 5251	0.999
	Endosulfan sulfate	y = 2E+06x - 5737	0.995
	p,p′-DDT	y = 2E+06x - 8868	0.994
	Methoxychlor	y = 2E+06x - 3809	0.997
	Endrin ketone	y = 72793x - 1628	0.998
(B)	**Organophosphate**		
	DDVP	y = 13193x + 145.5	0.998
	Acephate	y = 50552x - 36458	0.990
	Phorate	y = 1E+06x - 8314	0.999
	Monocrotophos	y = 99119x - 3422	0.995
	Chlorpyriphos	y = 92892x - 4531	0.999
	Me-parathion	y = 1E+06x - 6958	0.999
	Malathion	y = 85835x - 4128	0.999
	Phorate sulphoxide	y = 61535x - 885.2	0.998
	Quinalphos	y = 1E+06x - 7700	0.999
	Phorate sulphone	y = 99792x - 6441	0.999
	Profenophos	y = 75570x - 8037	0.999
	Ethion	y = 1E+06x + 23862	0.991
	Triazophos	y = 1E+06x - 15020	0.999
	Phosalone	y = 42039x - 5830	0.998
(C)	**Synthetic Pyrethroids**		
	Bifenthrin	y = 34721x - 7182	0.991
	Fenpropathrin	y = 33922x - 5167	0.993
	λ-Cyhalothrin	y = 75954x - 23370	0.998
	β-Cyfluthrin	y = 27957x - 2270	0.991
	α-Cypermethrin	y = 39936x - 3434	0.990
	Fenvalerate-I	y = 28685x - 2221	0.999
	Fenvalerate-II	y = 79718x + 2251	0.999
	Deltamethrin	y = 33580x - 13643	0.998
(D)	**Herbicide**		
	Trifluralin	y = 1E+06x - 33836	0.997
	Fluchloralin	y = 1E+06x - 39648	0.998
	Alachlor	y = 27148x + 2561	0.997
	Metalachlor	y = 11370x + 3789	0.996
	Pendimethalin	y = 55738x - 14166	0.998
	Butachlor	y = 21992x + 2039	0.995



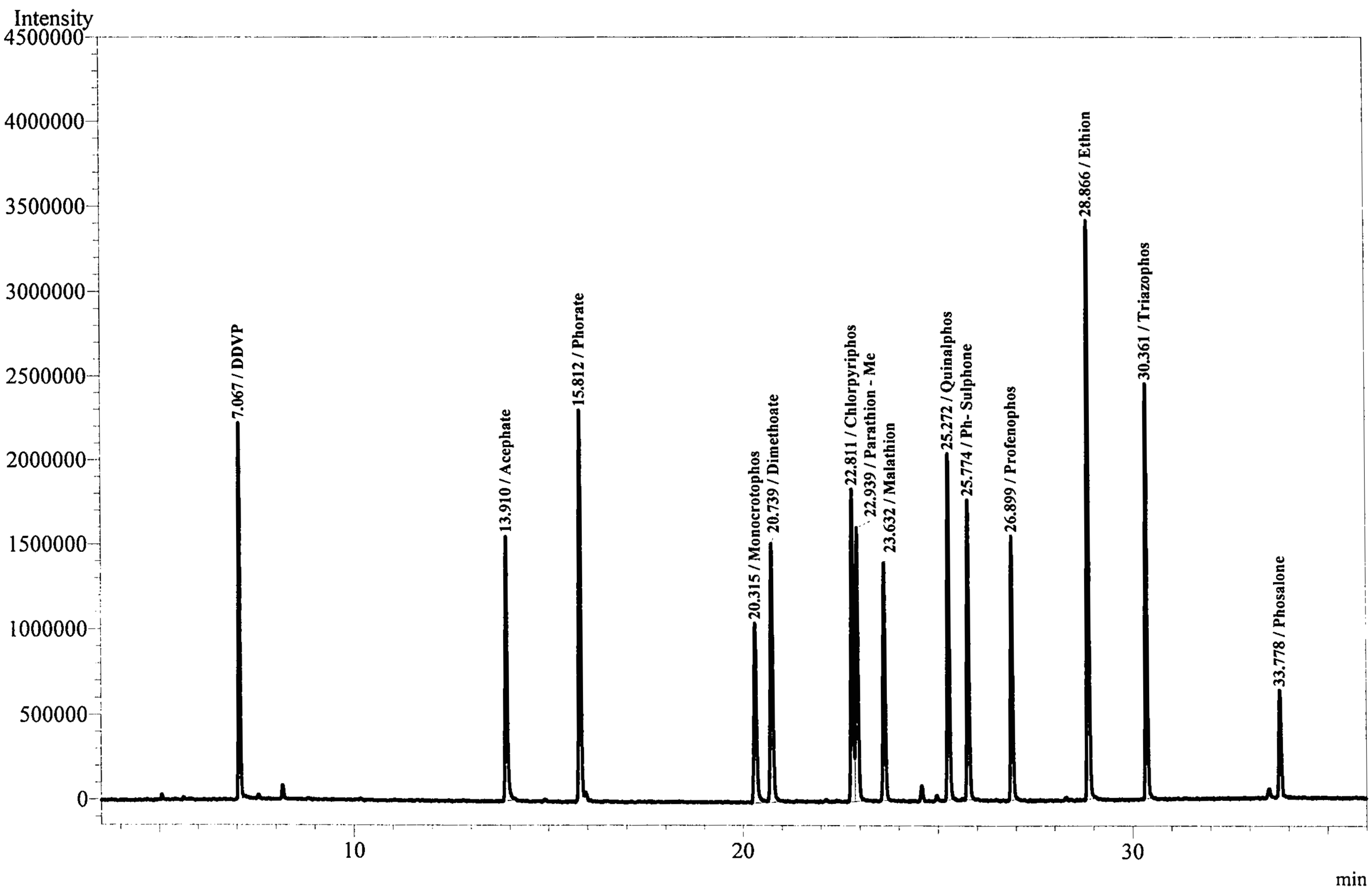
Chromatogram: Water Rec OCs 0.1 ppb R-I



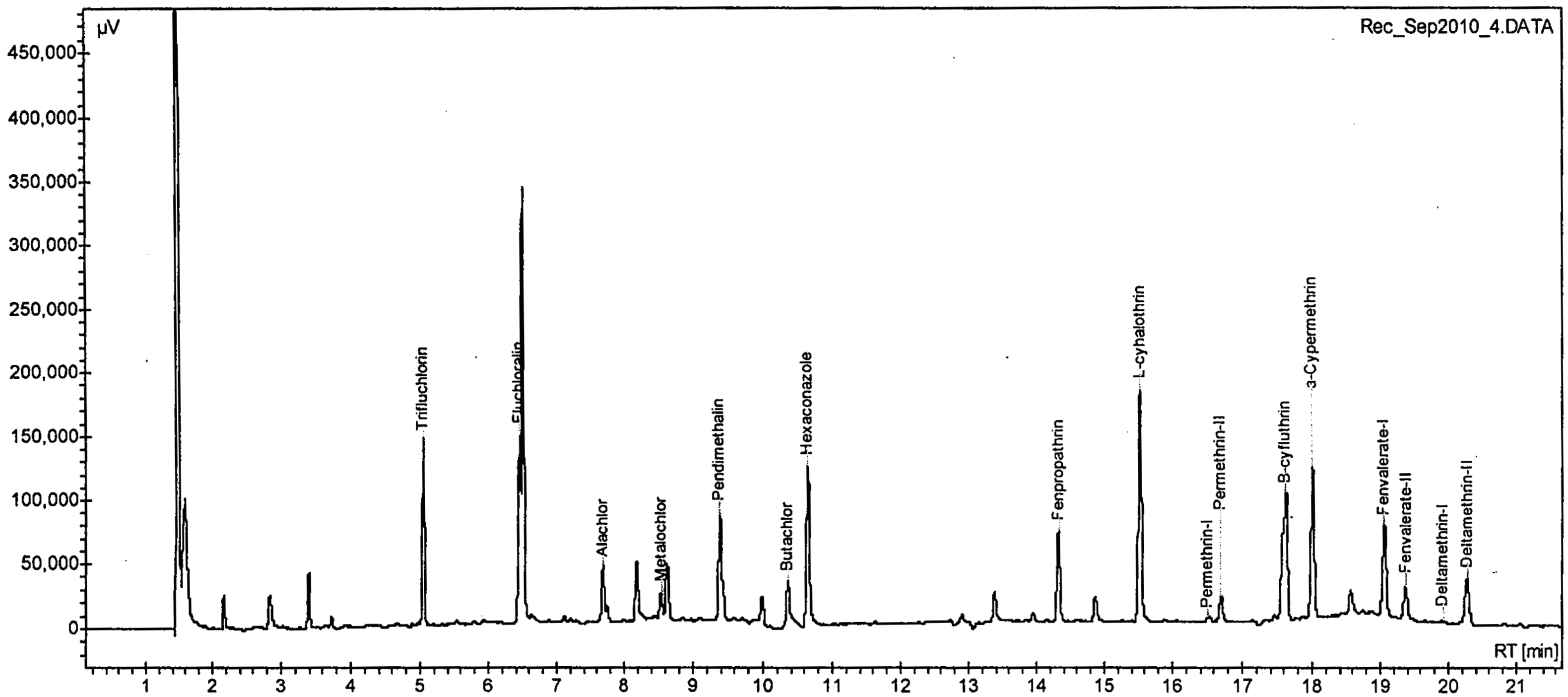
Chromatogram: Std OCs 0.1 ppm



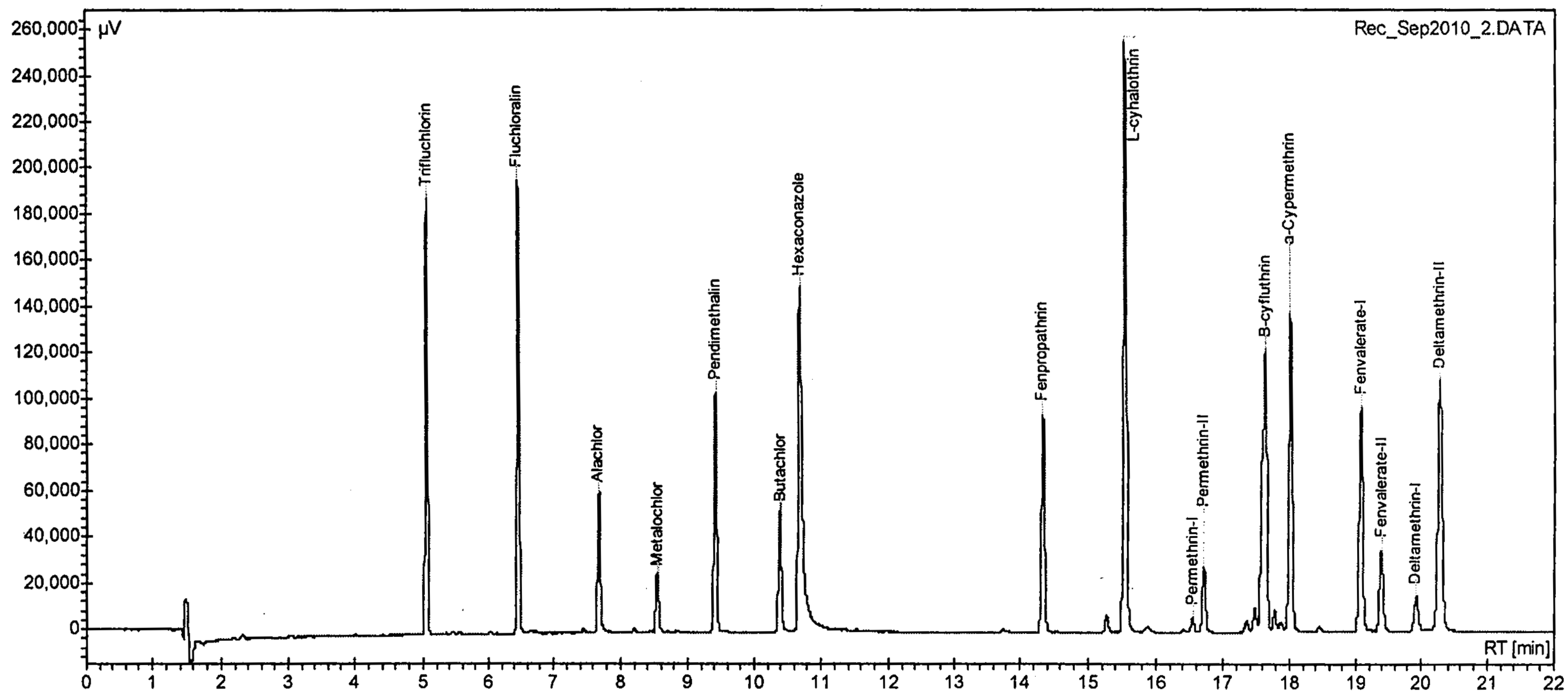
Chromatogram : Water Rec 0.1 ppb R-I



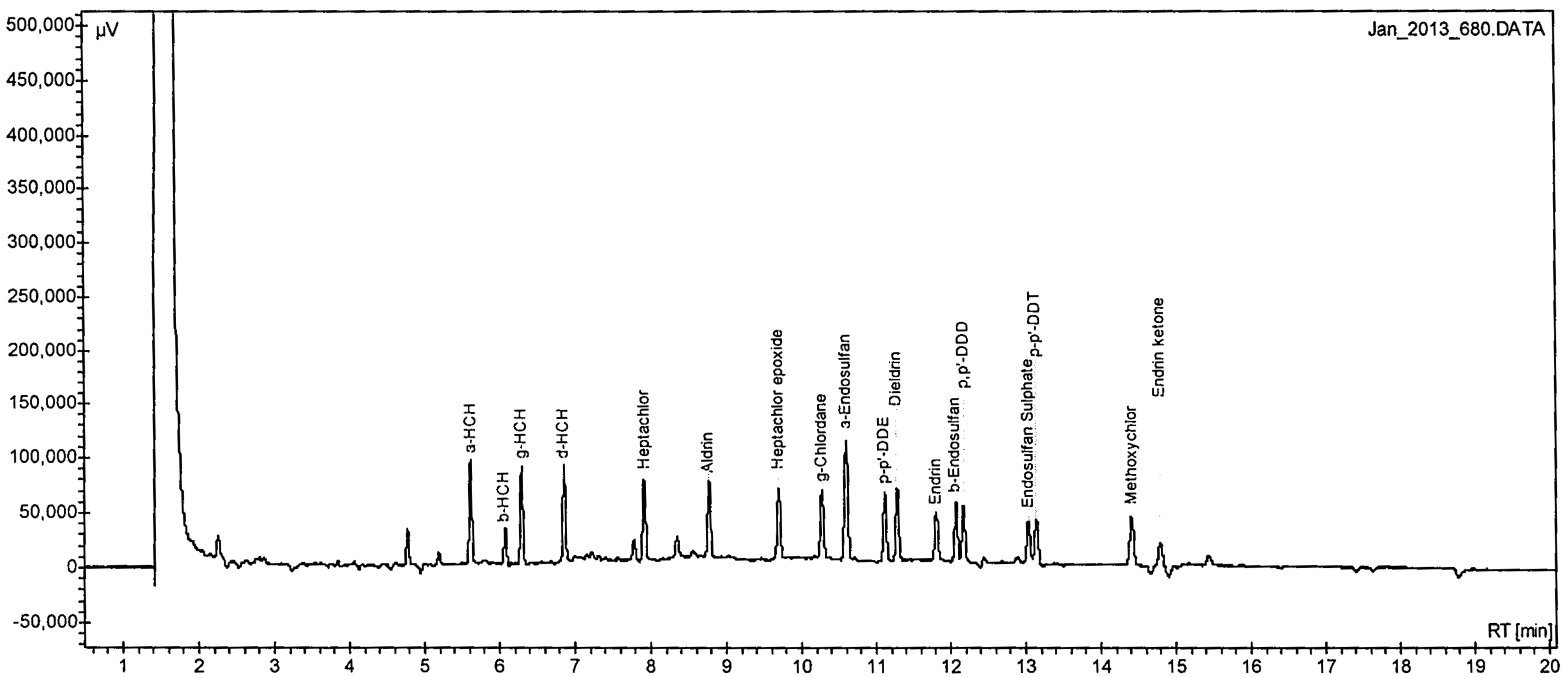
Chromatogram : Std OPs-15 0.1 ppm



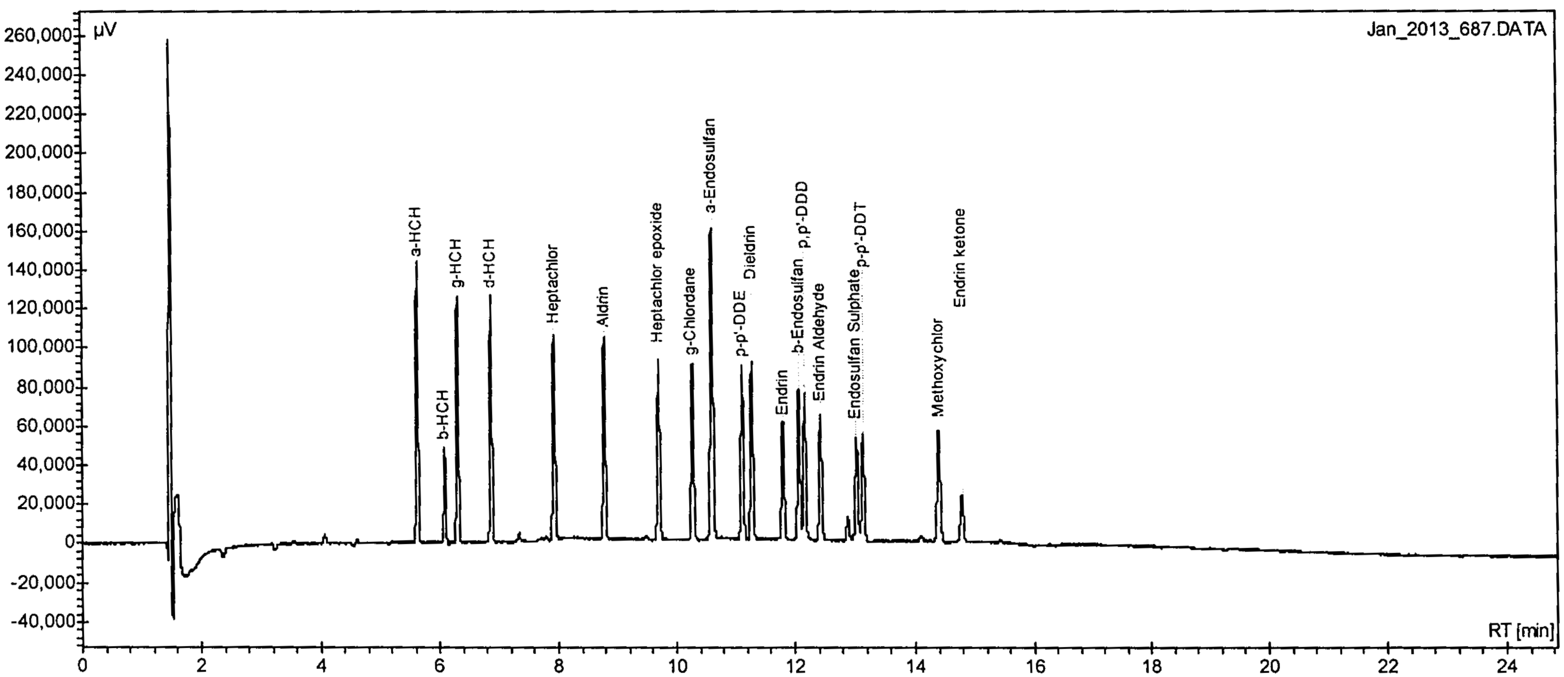
Chromatogram: Water Rec SPs+HBs 0.1 ppb R-I



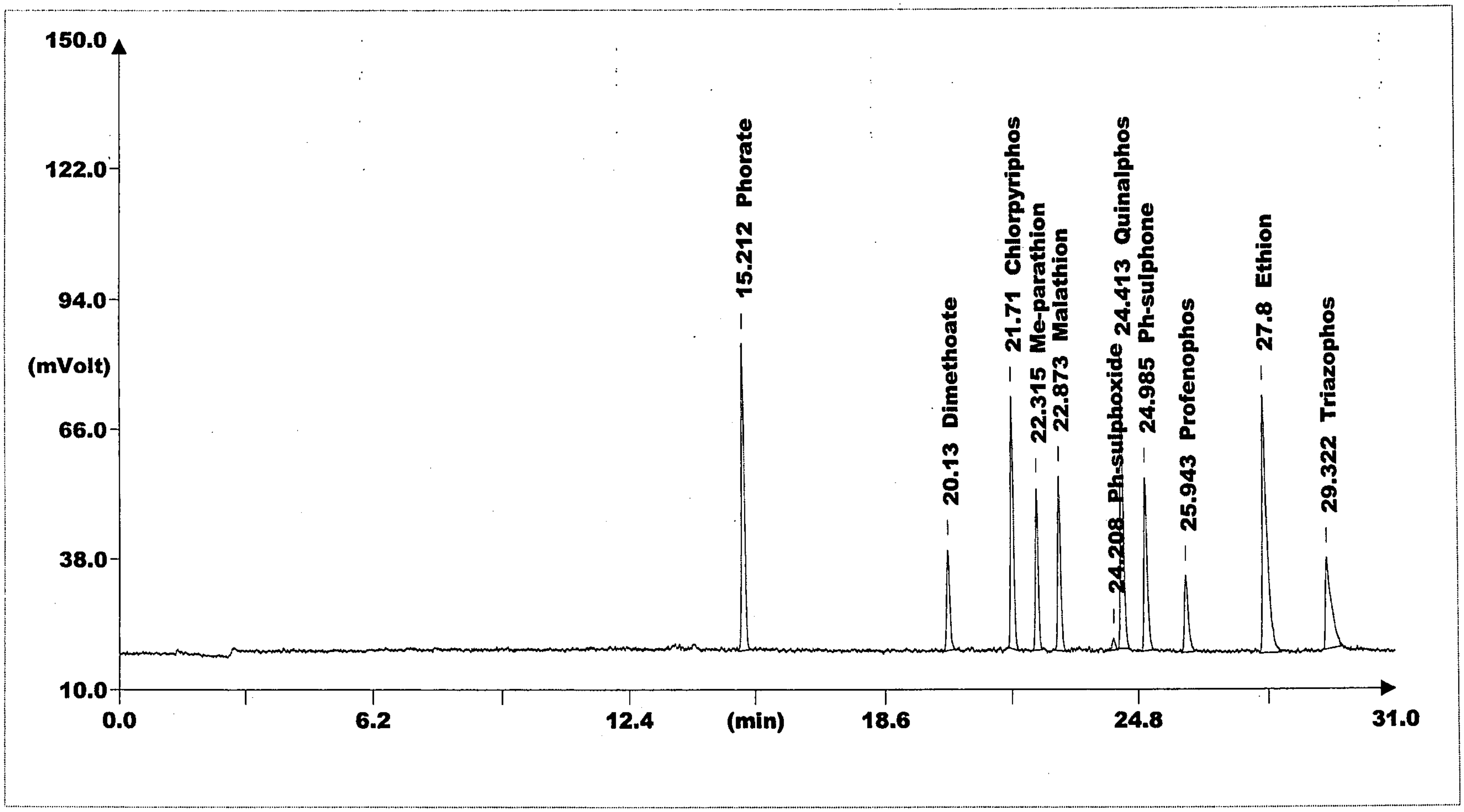
Chromatogram: Std SPs+HBs 0.1 ppm



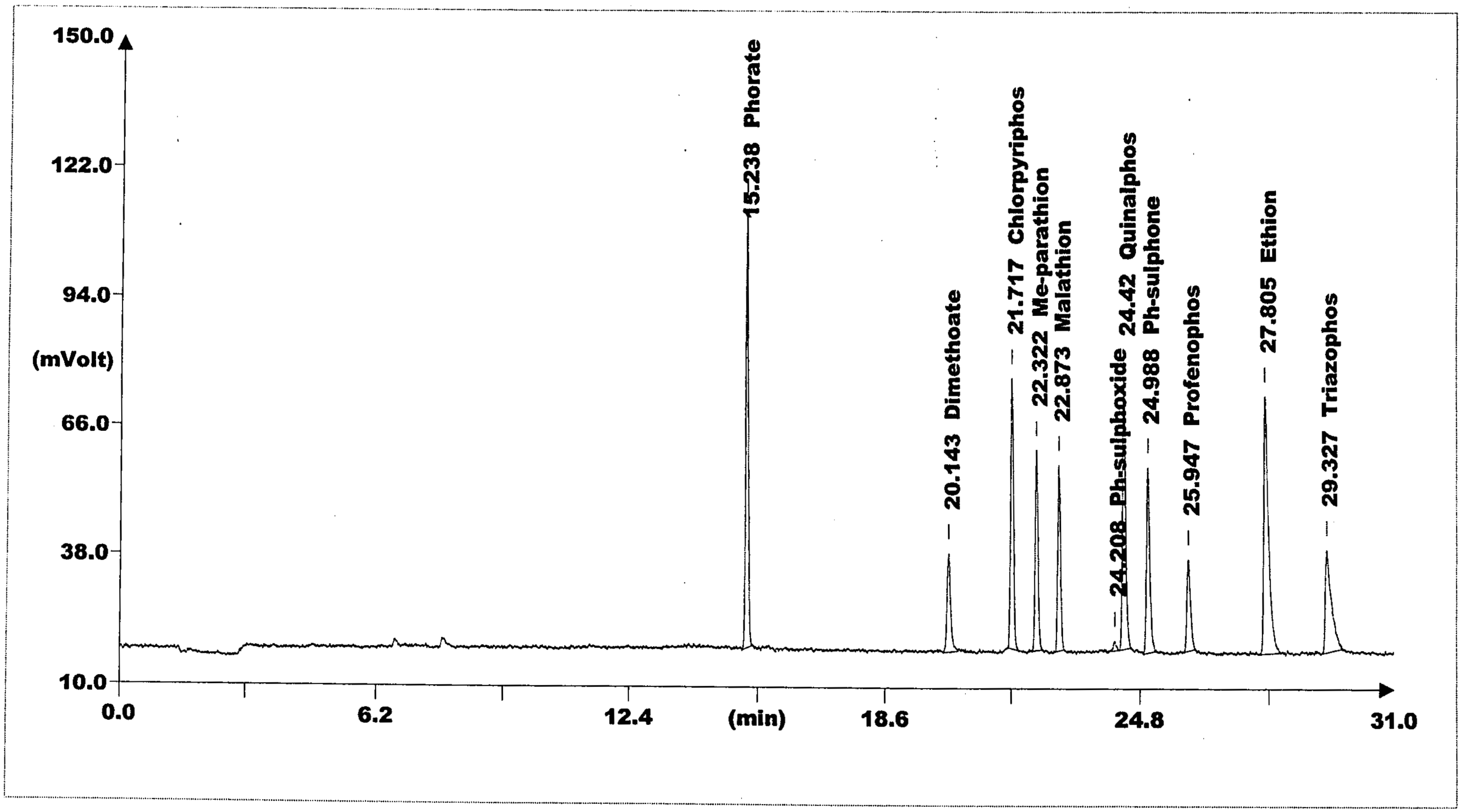
Chromatogram: Soil OCs Rec 0.01 ppm R-I



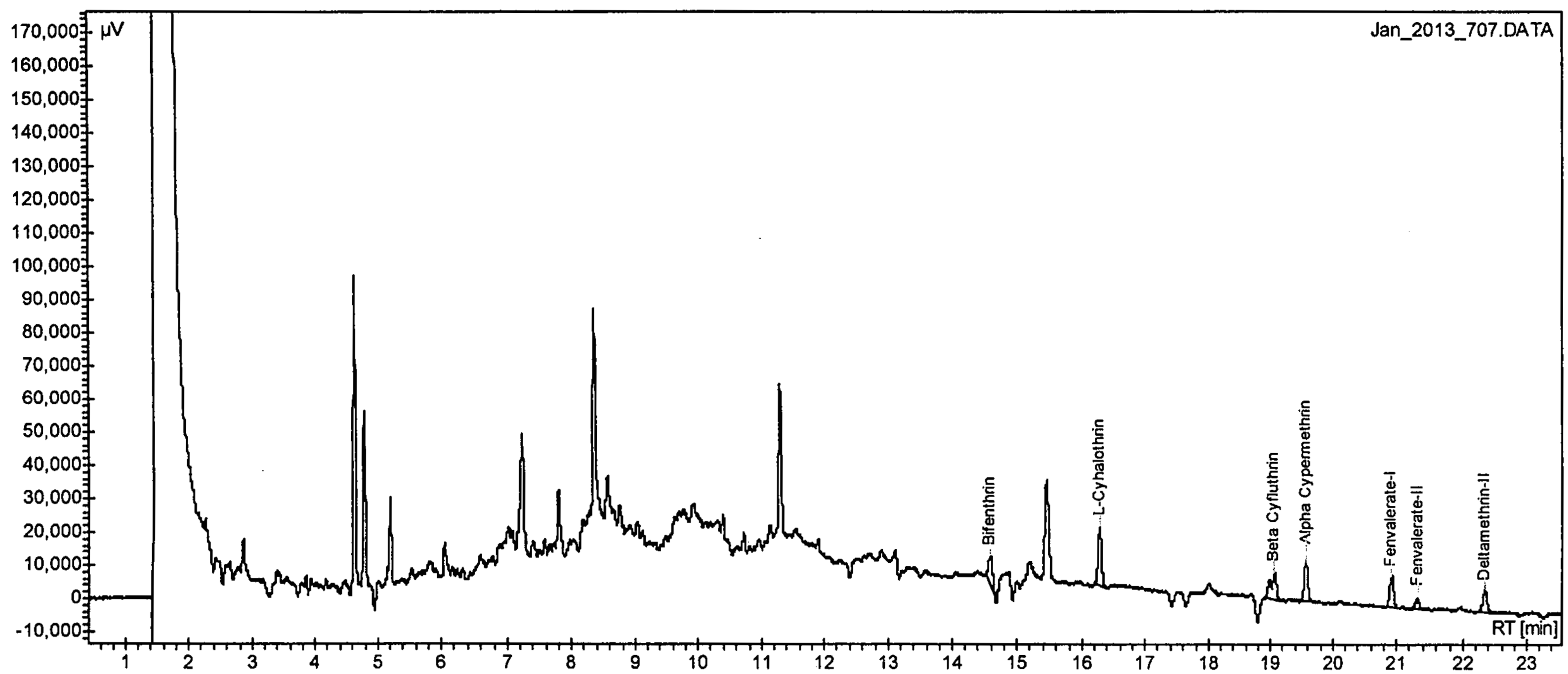
Chromatogram: Std OCs 0.025 ppm



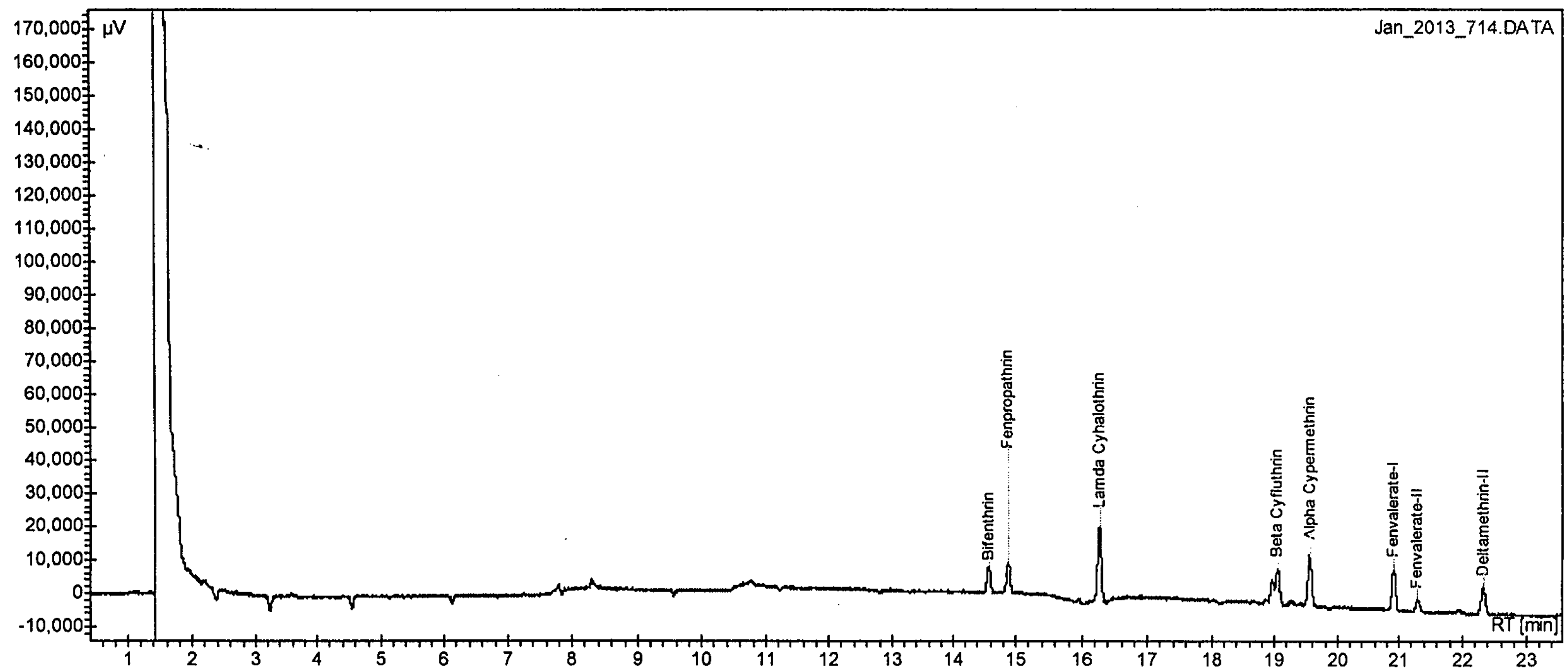
Soil Rec OPs 0.05 ppm R-I



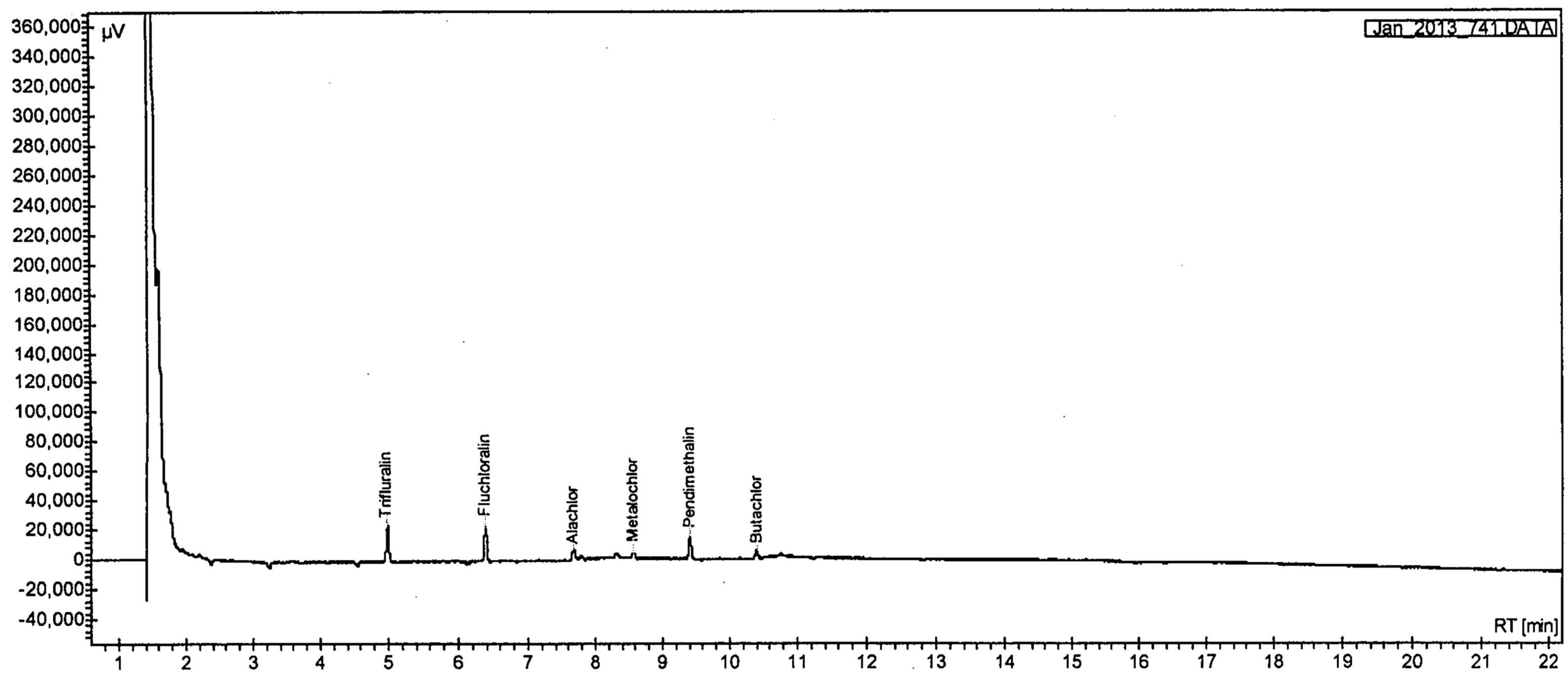
Std OPs 0.1 ppm



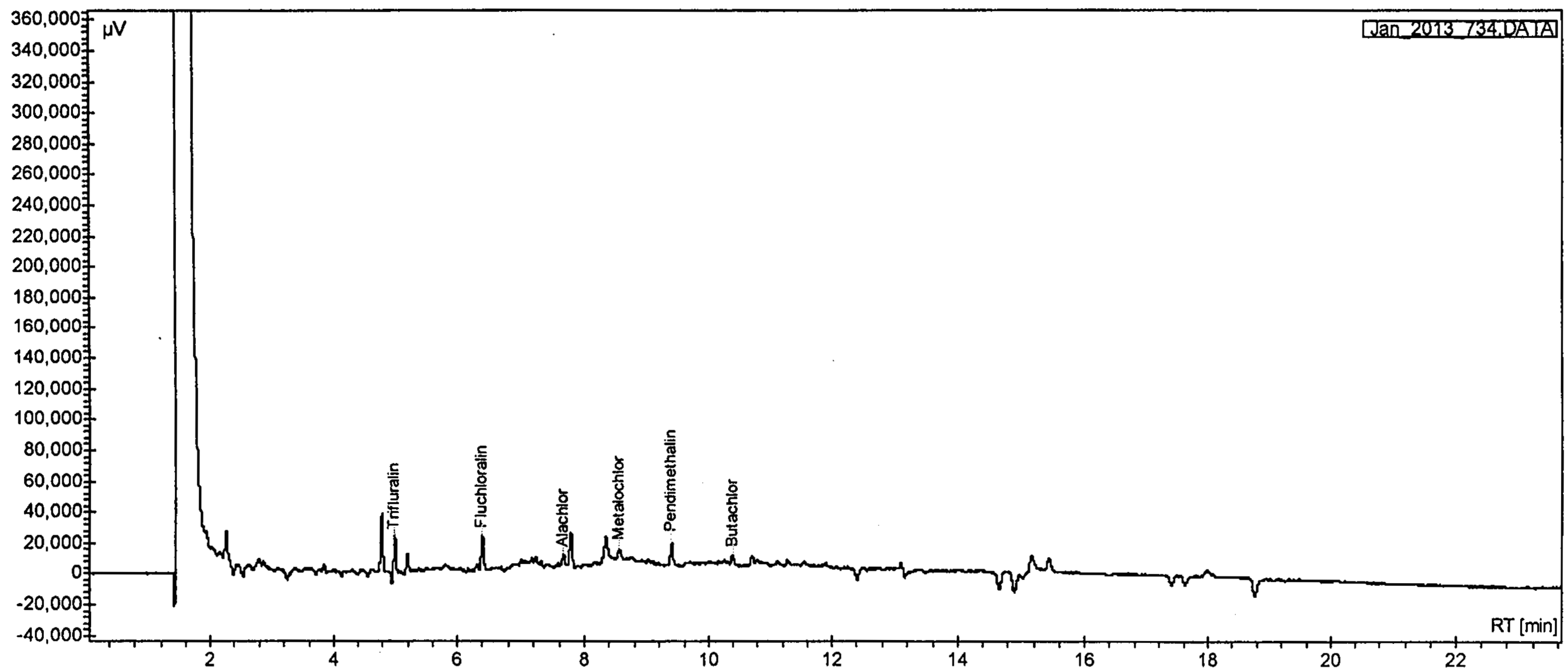
Chromatogram: Soil Rec SPs 0.01 ppm R-I



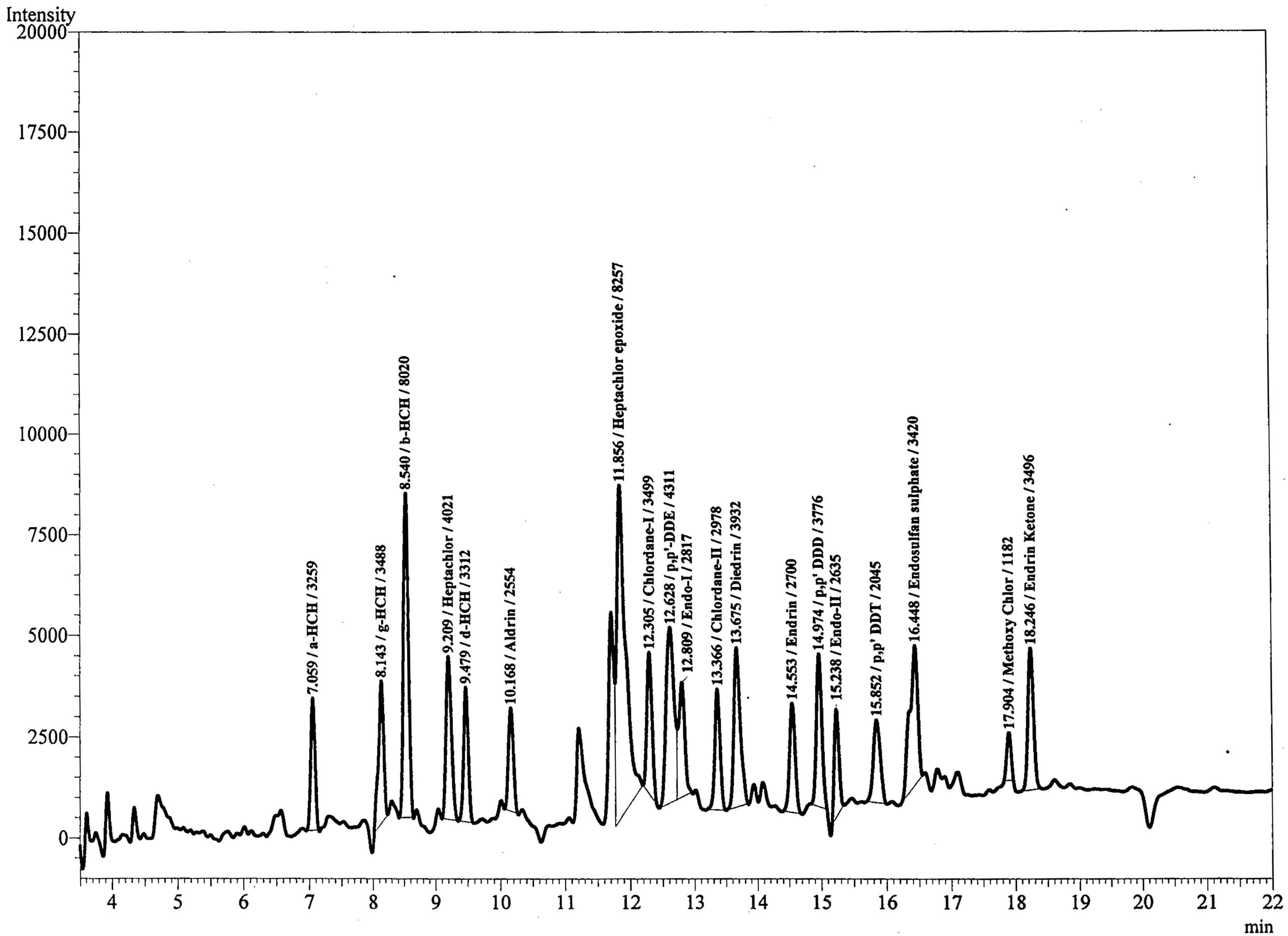
Chromatogram: Std SPs 0.025 ppm



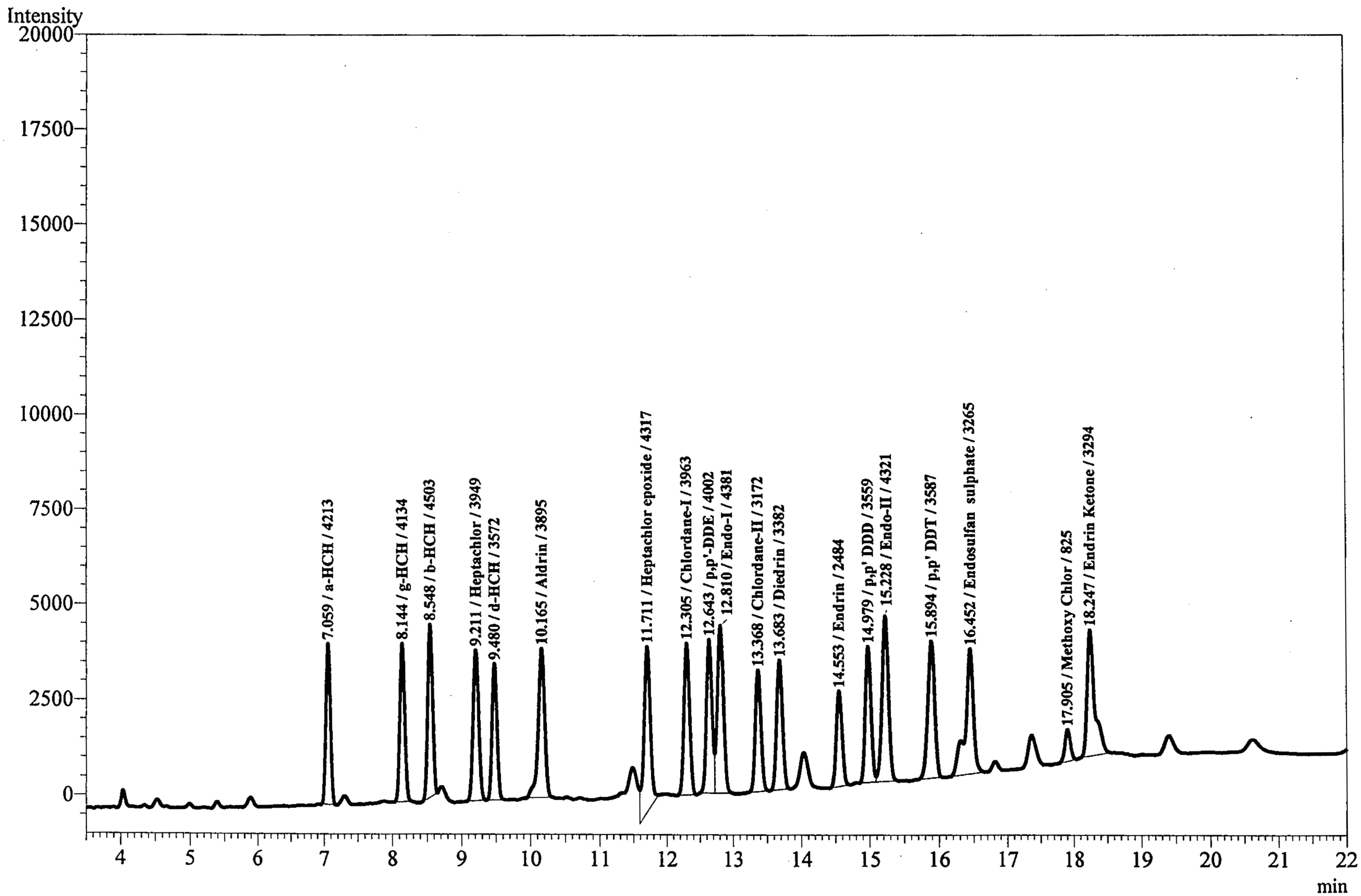
Chromatogram: Soil Rec HBs 0.01 ppm R-I



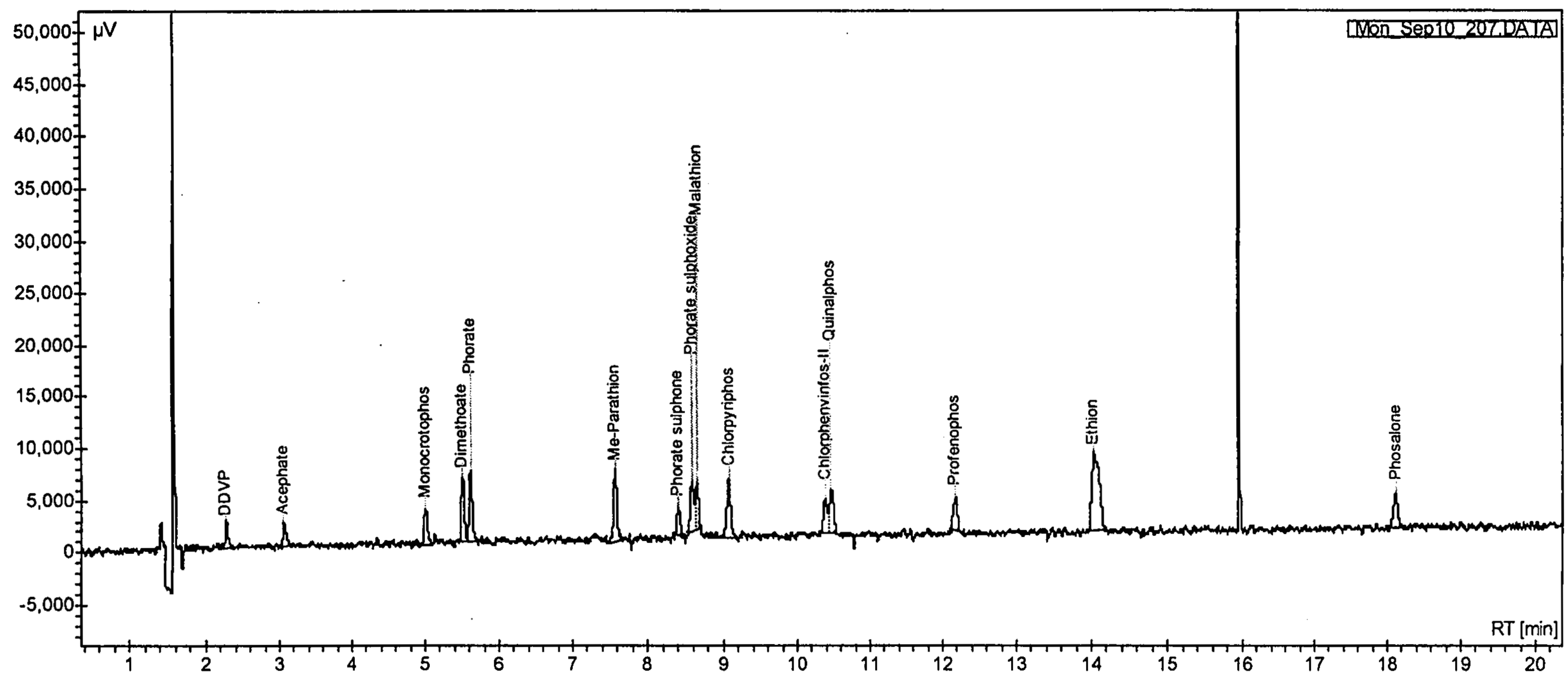
Chromatogram: Std HBs 0.025 ppm



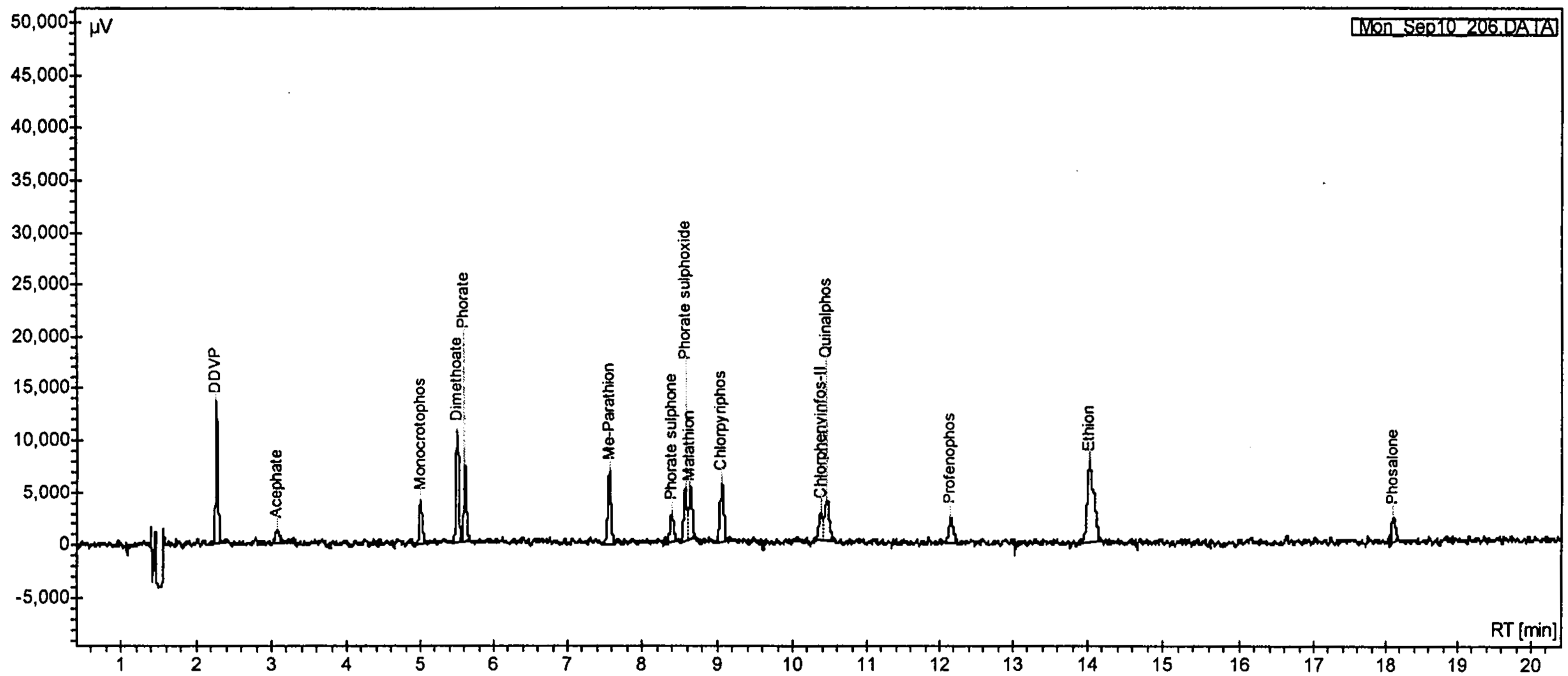
Chromatogram : Brinjal OCs Rec 0.01 FL R-I



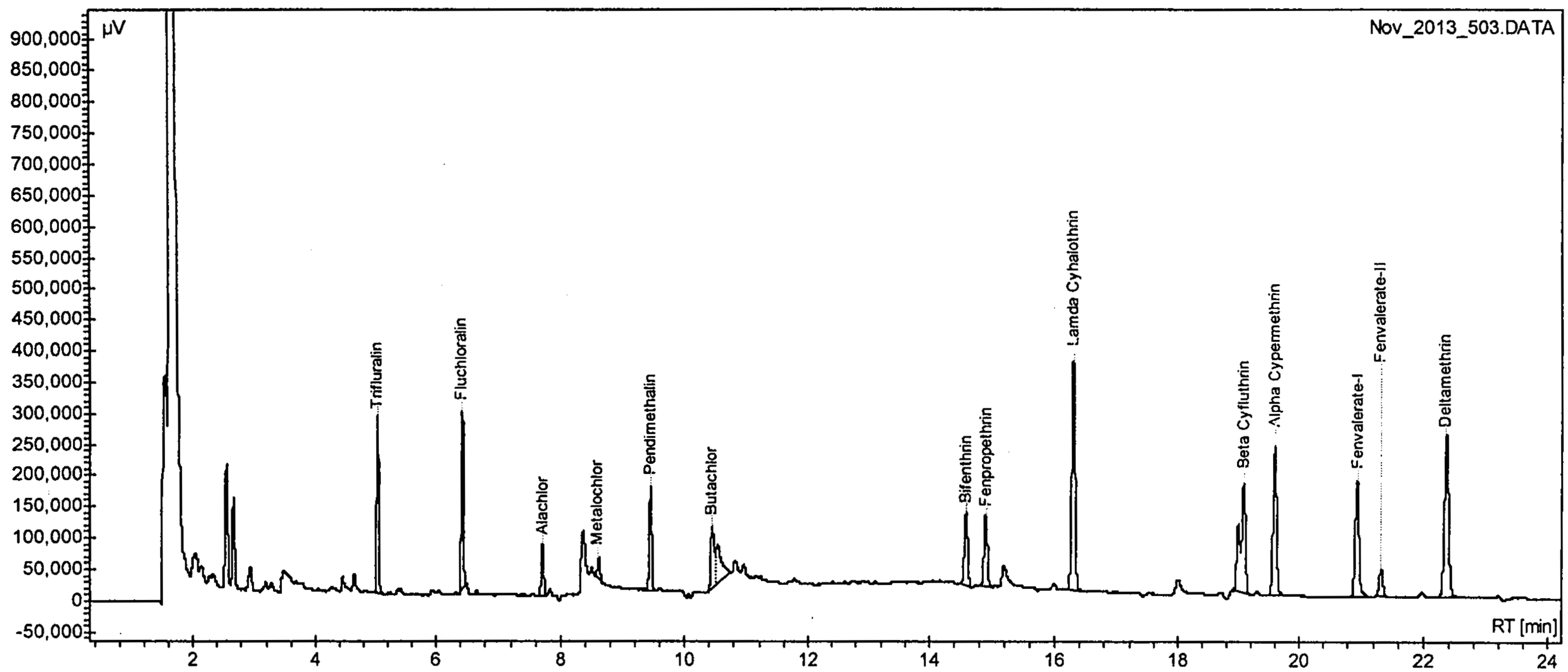
Chromatogram : Std OCs-20 0.01 ppm



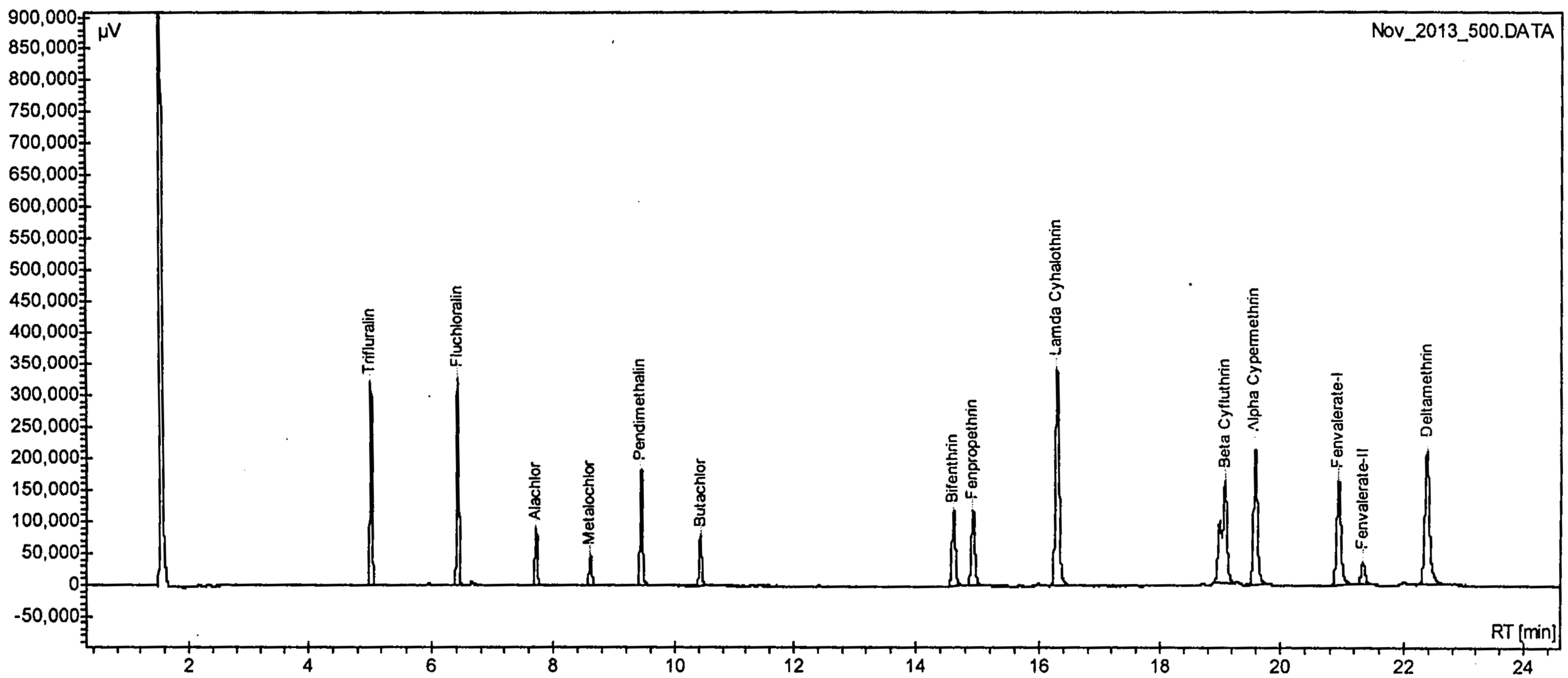
Chromatogram: Brinjal Rec OPs 0.05 ppm R-I



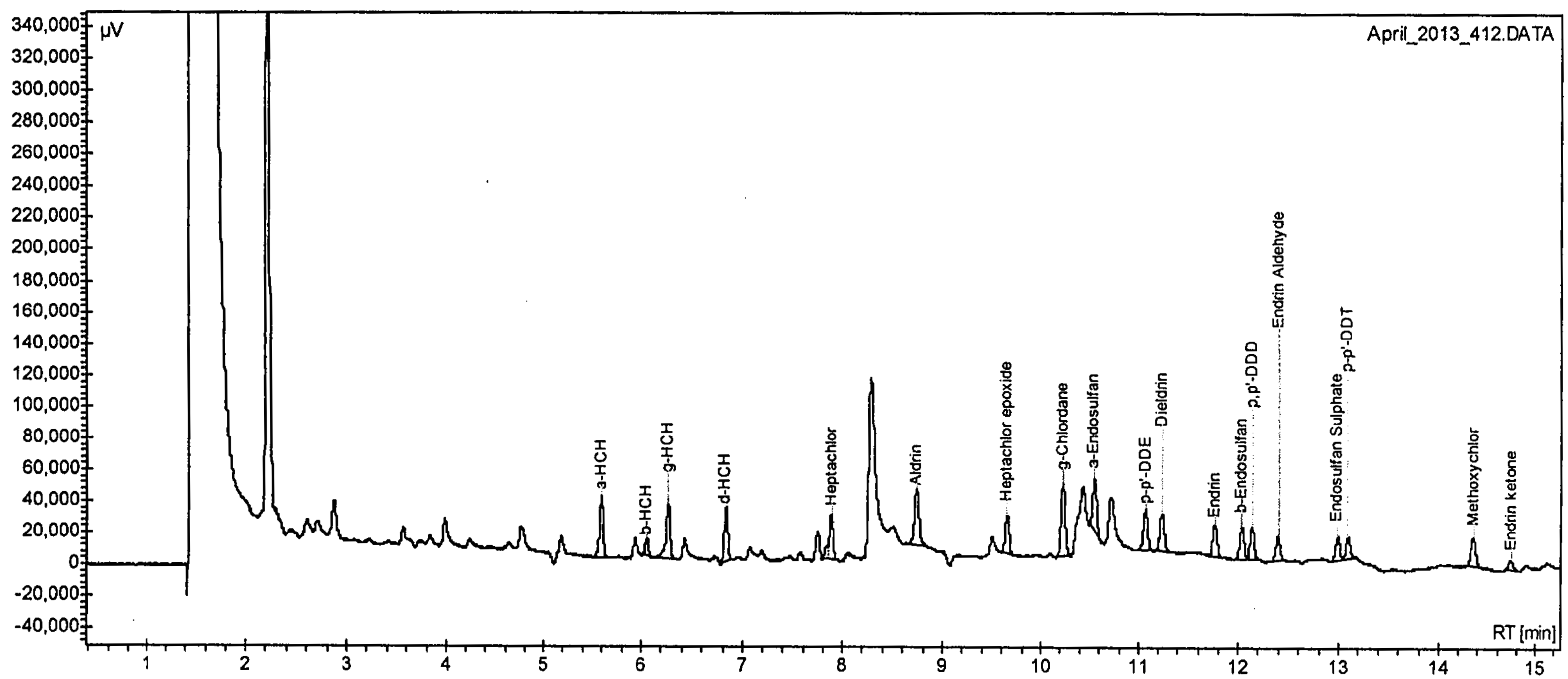
Chromatogram: Std OPs 0.05 ppm



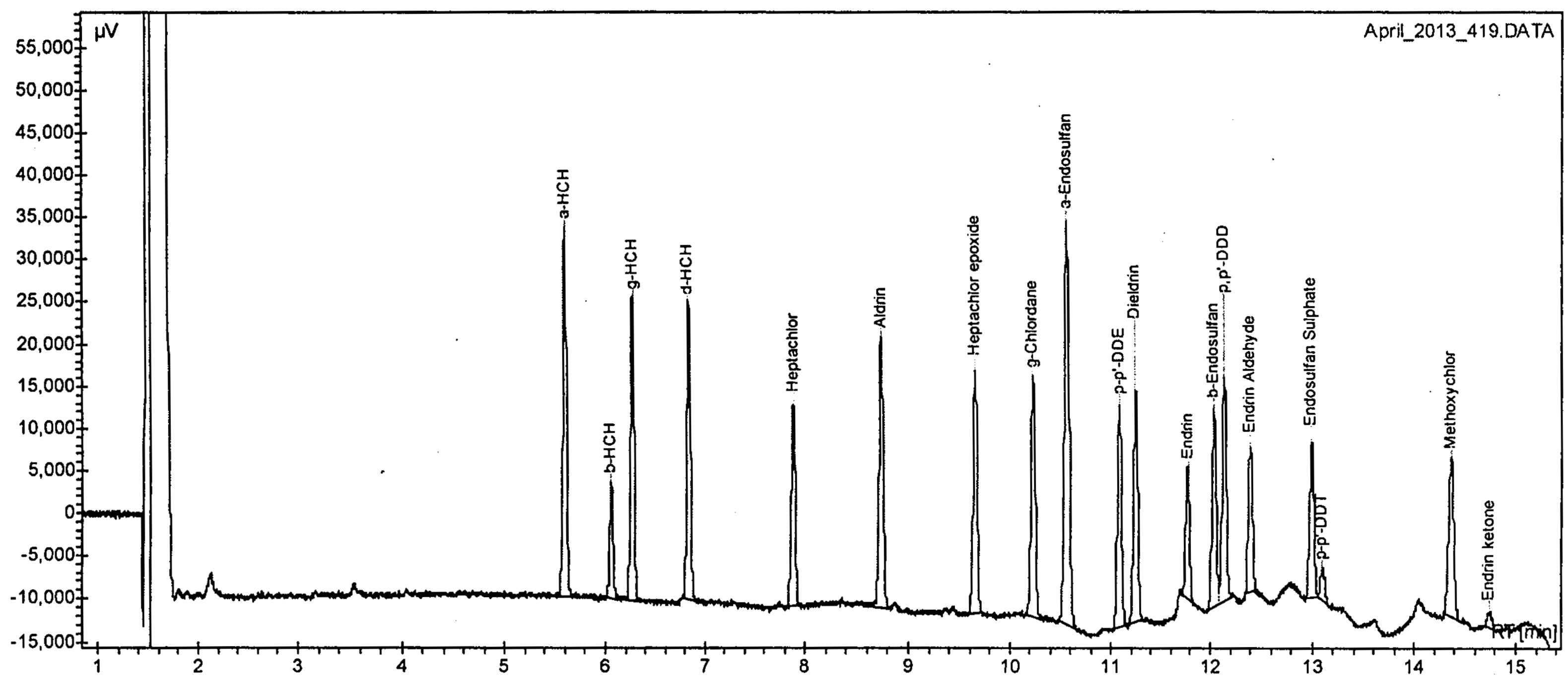
Chromatogram: Brinjal SP+HBs Rec 0.1 ppm R-I



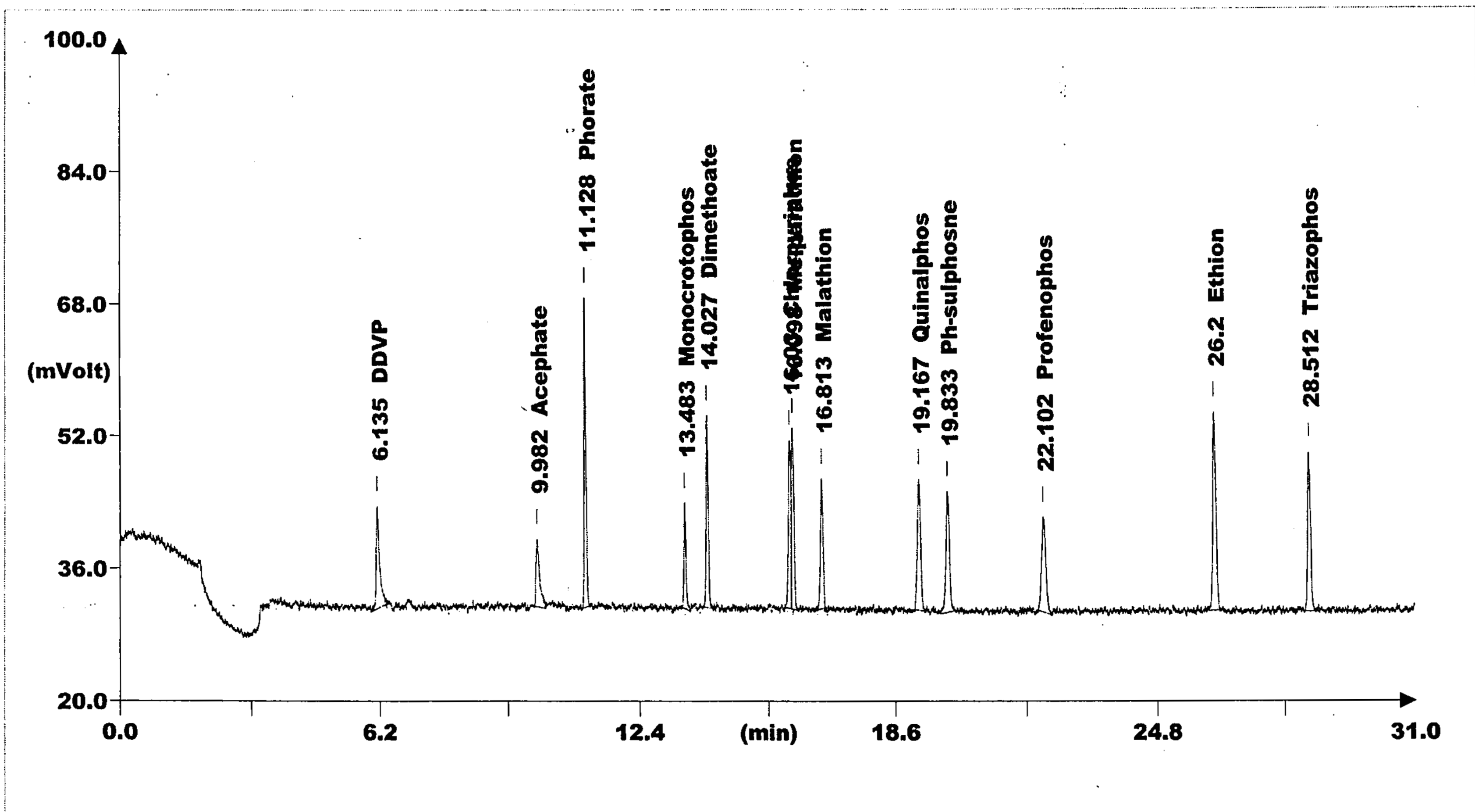
Chromatogram: Std SP+HBs 0.1 ppm



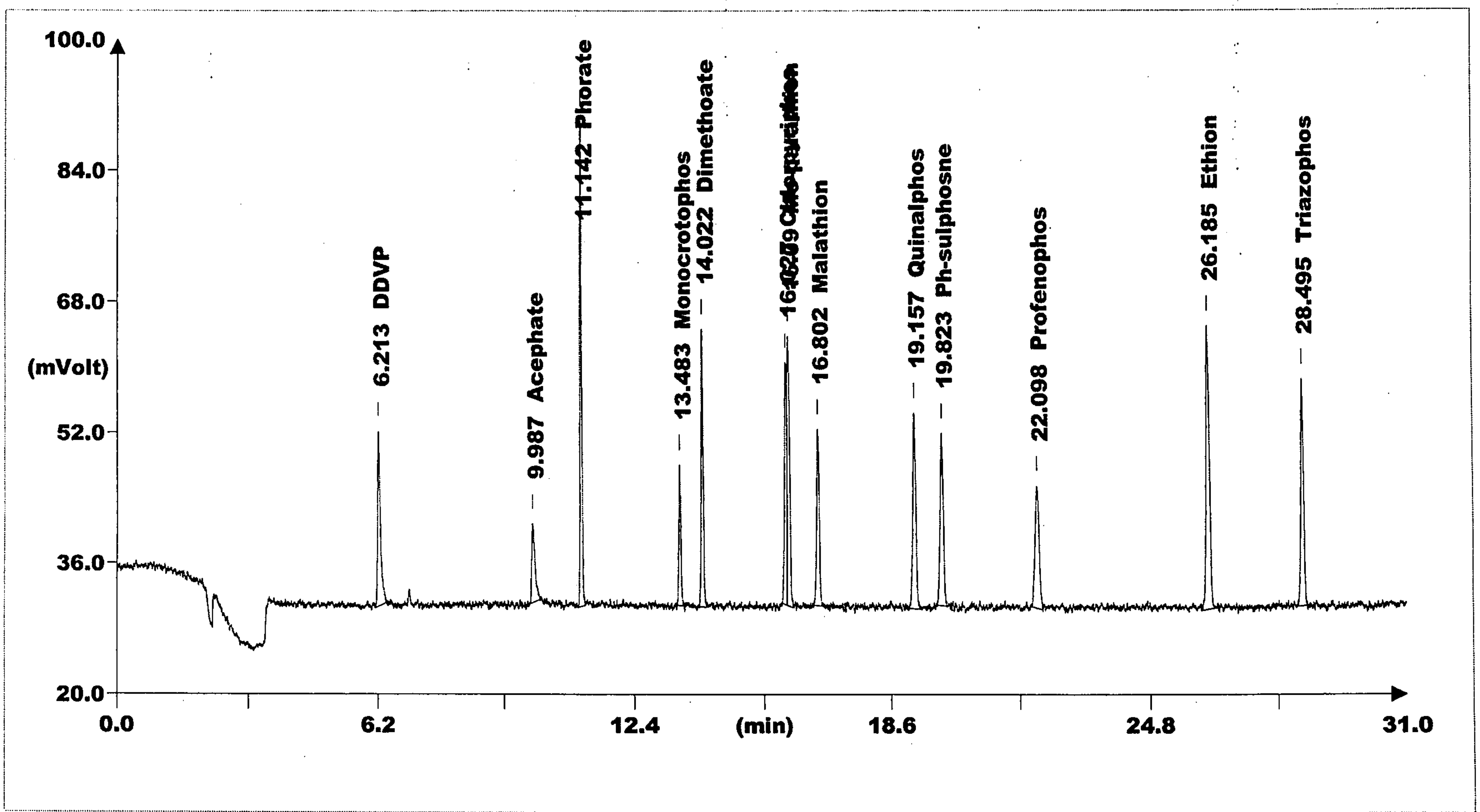
Chromatogram: Cotton lint Rec OCs 0.01 ppm R-I



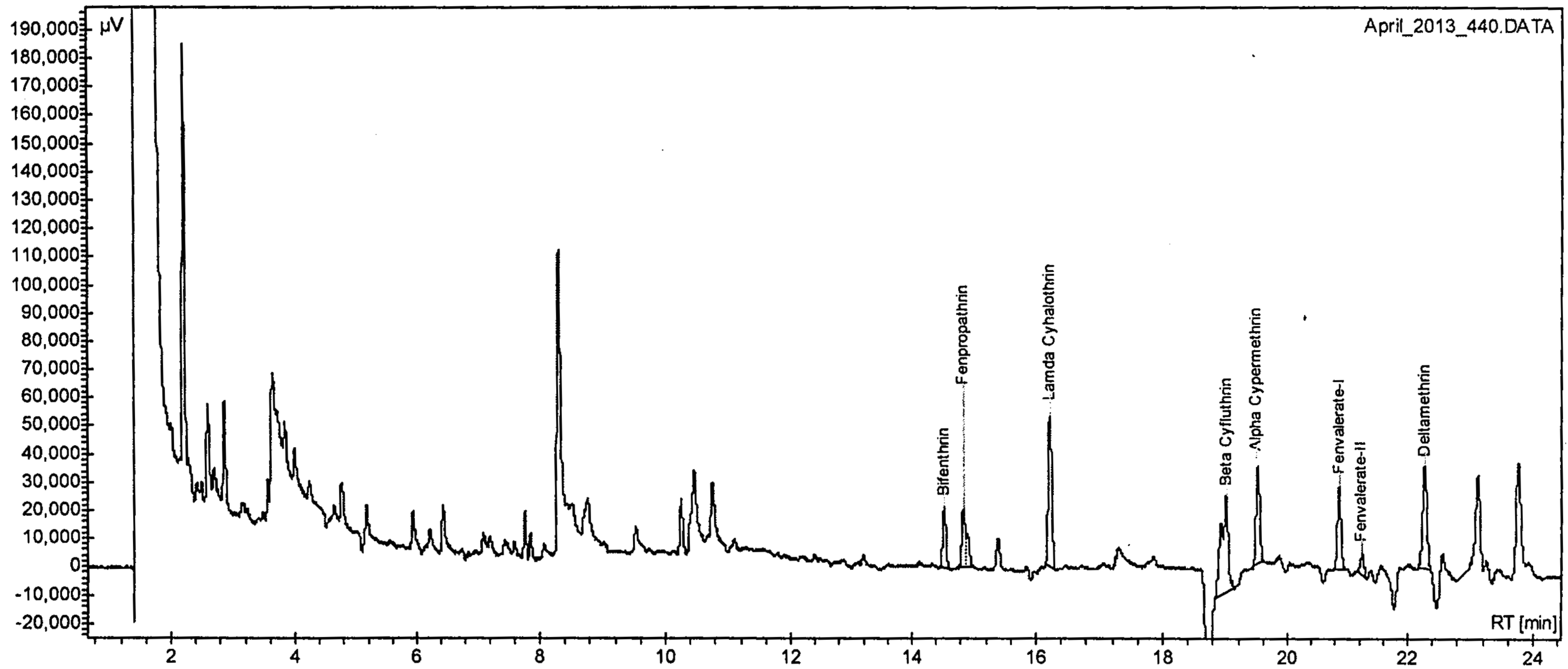
Chromatogram: Std OCs 0.01 ppm



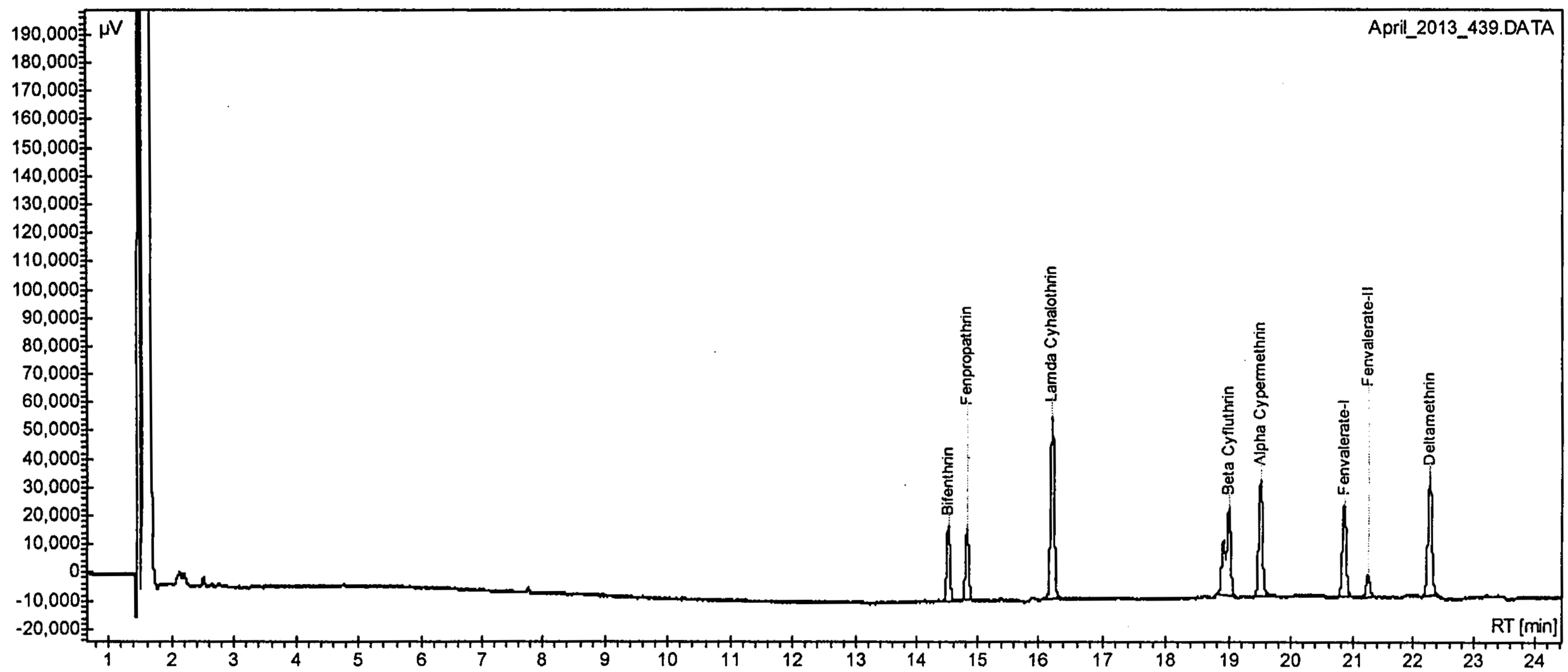
Cotton Lint Rec OPs 0.05 ppm R-I



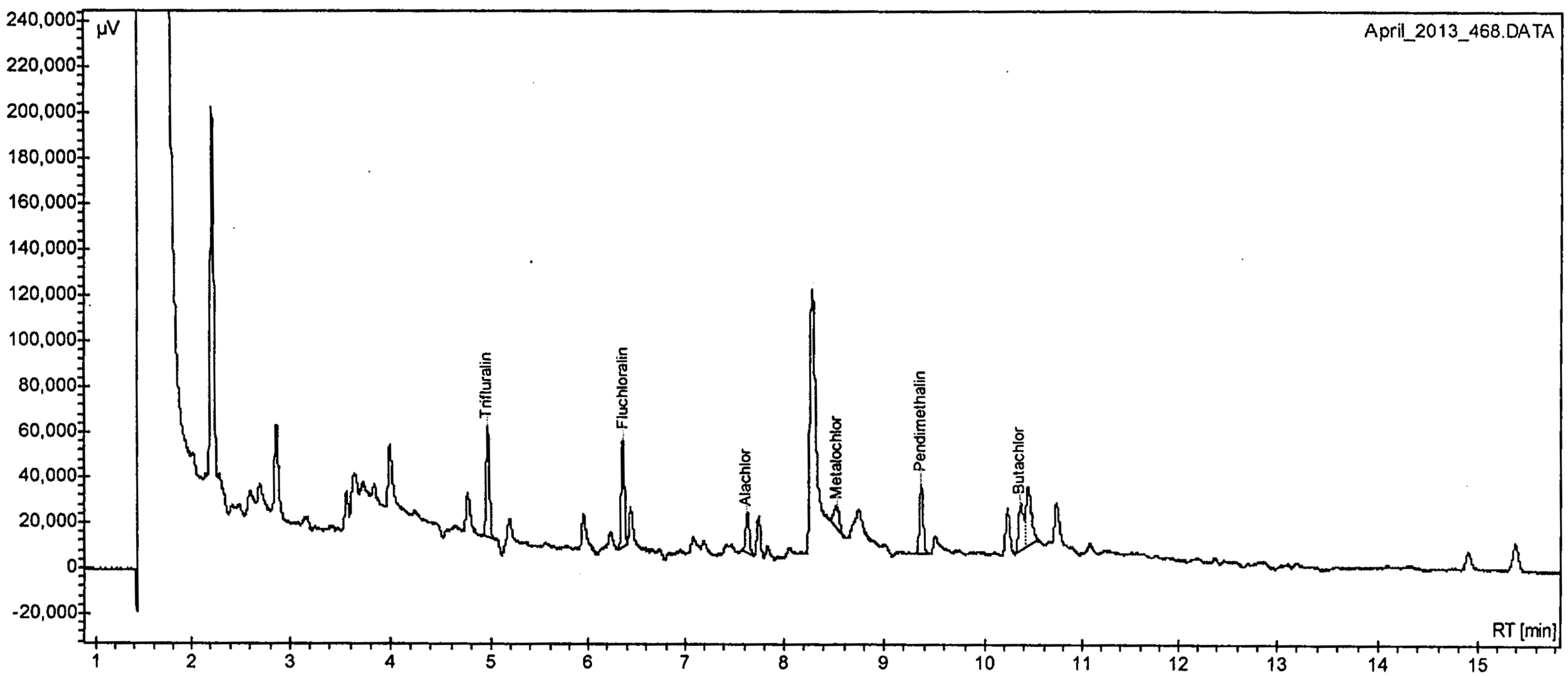
Std OPs 0.05 ppm



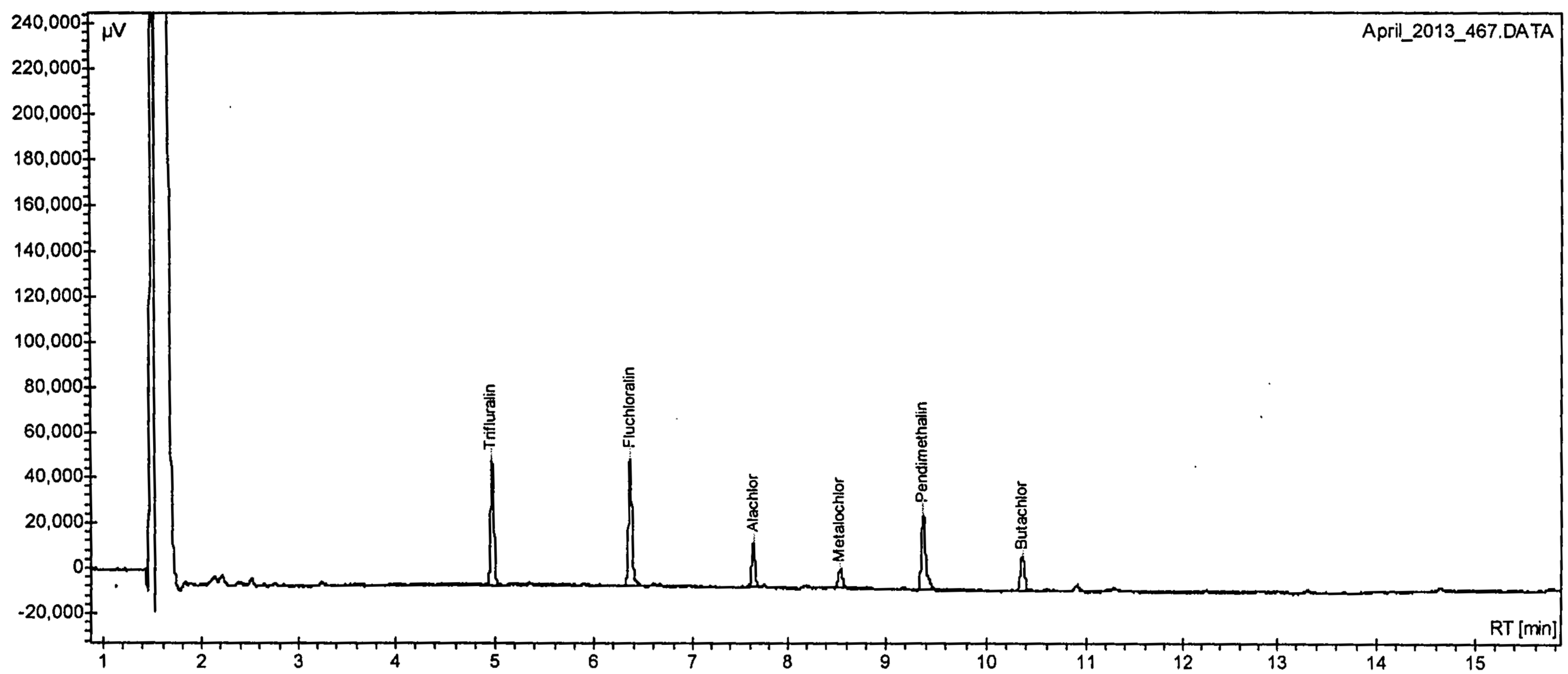
Chromatogram: Cotton lint Rec SPs 0.05 ppm R-I



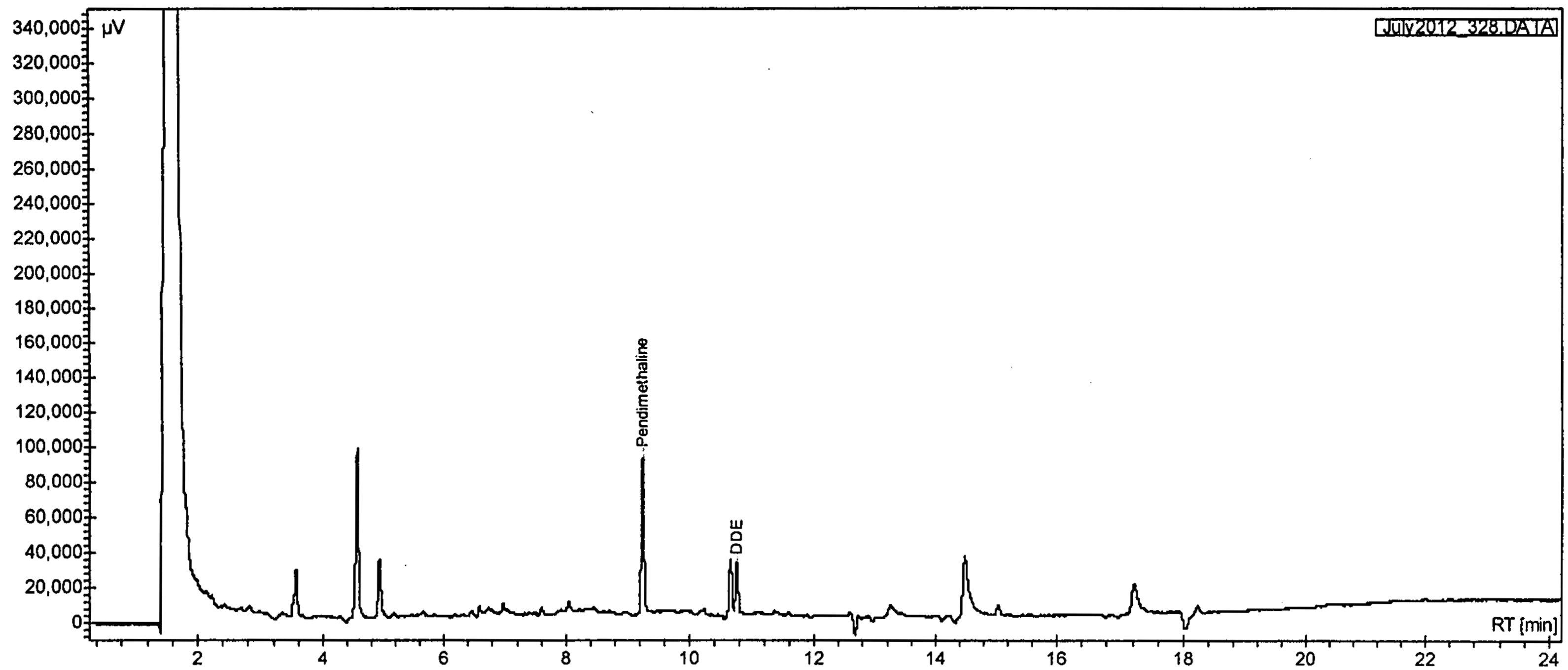
Chromatogram: Std SPs 0.05 ppm



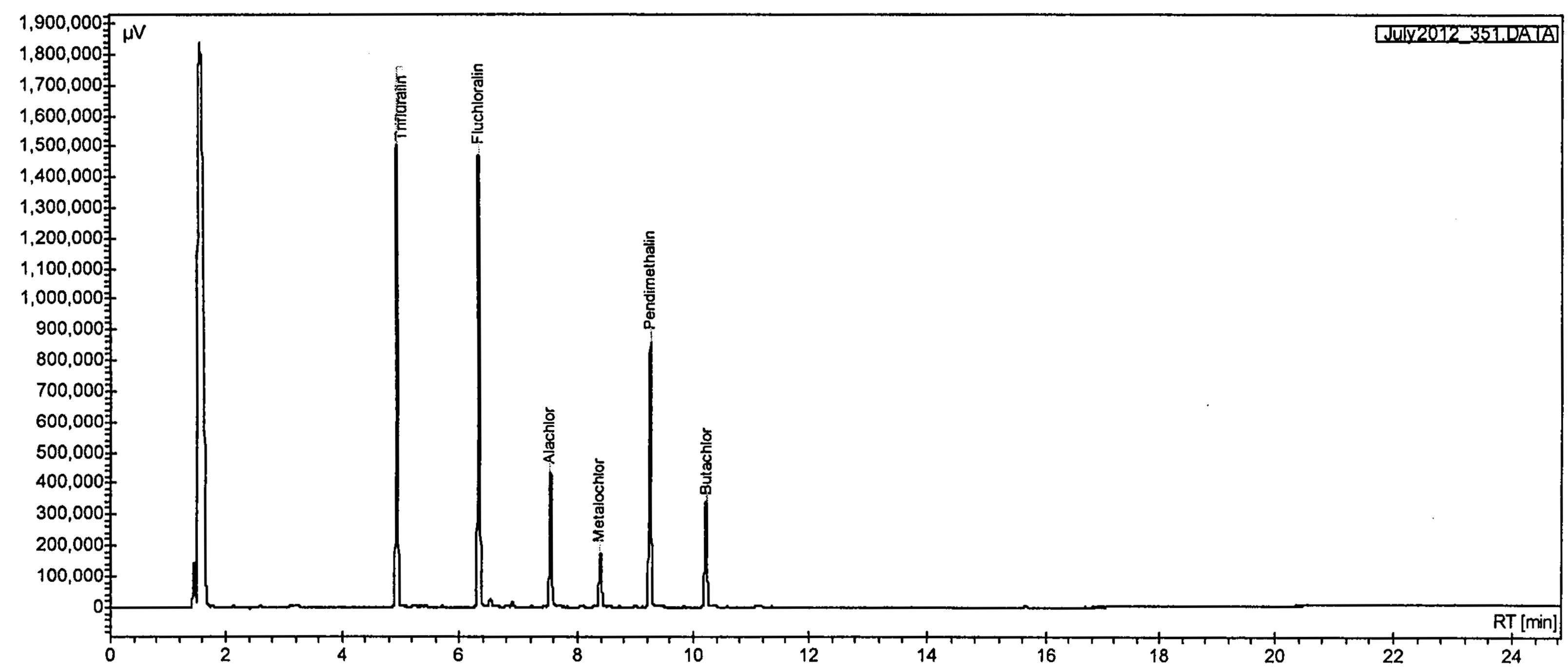
Chromatogram: Cotton lint Rec HBs 0.05 ppm R-I



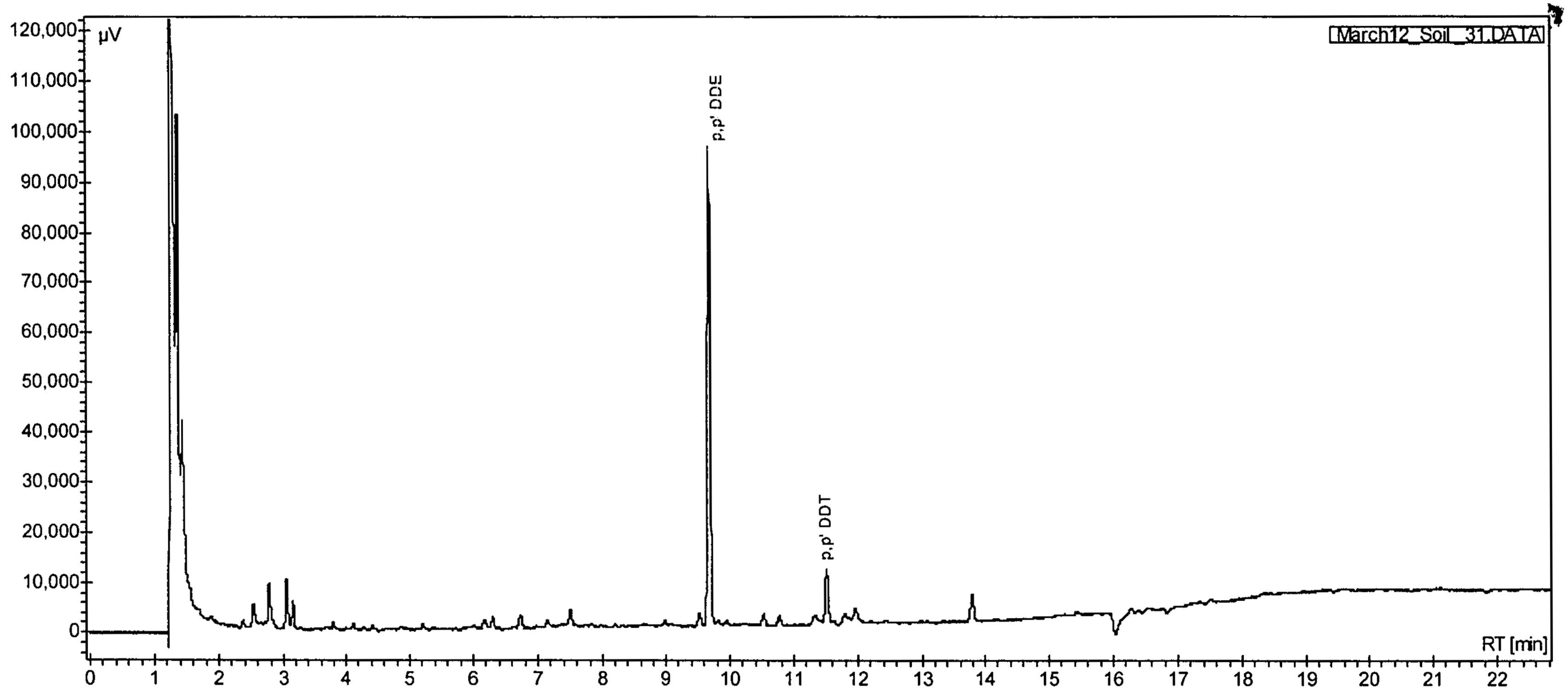
Chromatogram: Std HBs 0.05 ppm



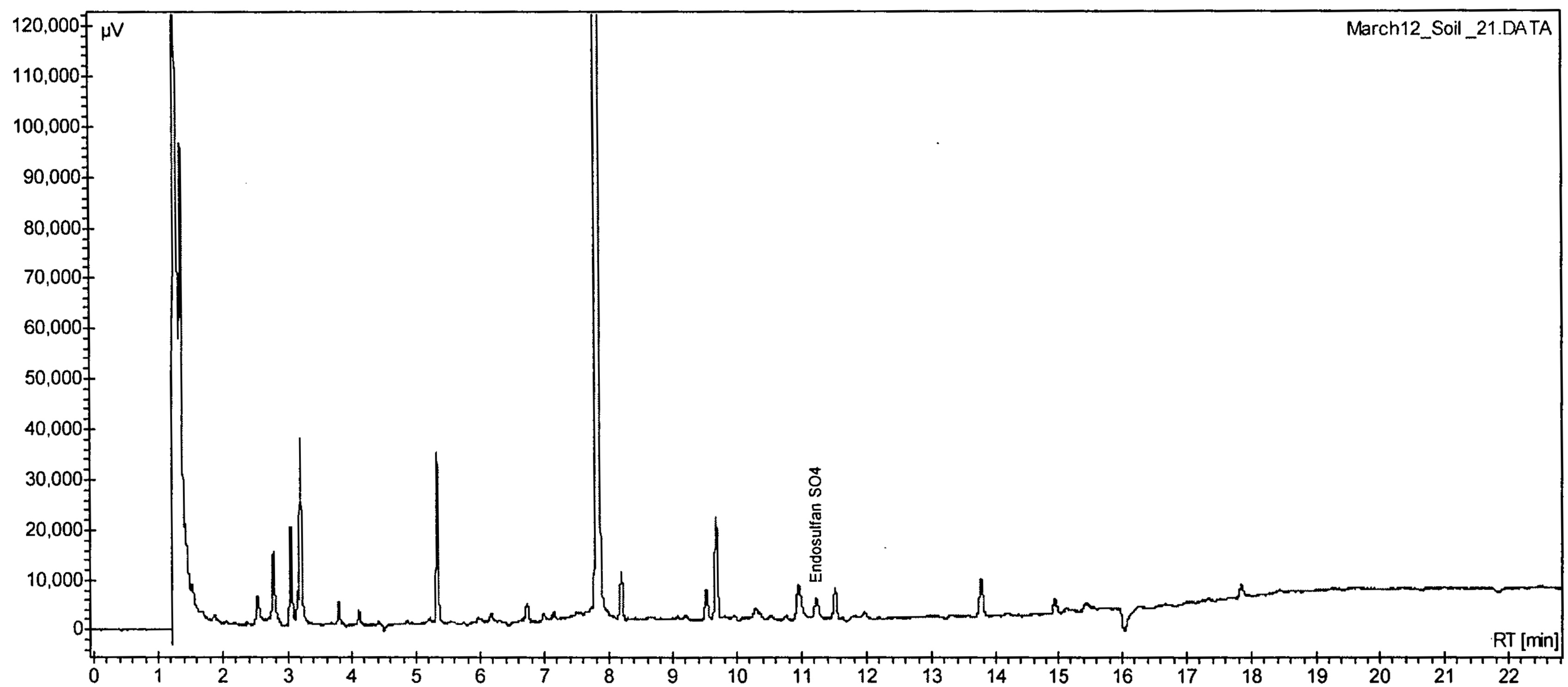
Chromatogram of soil: Sombhoi



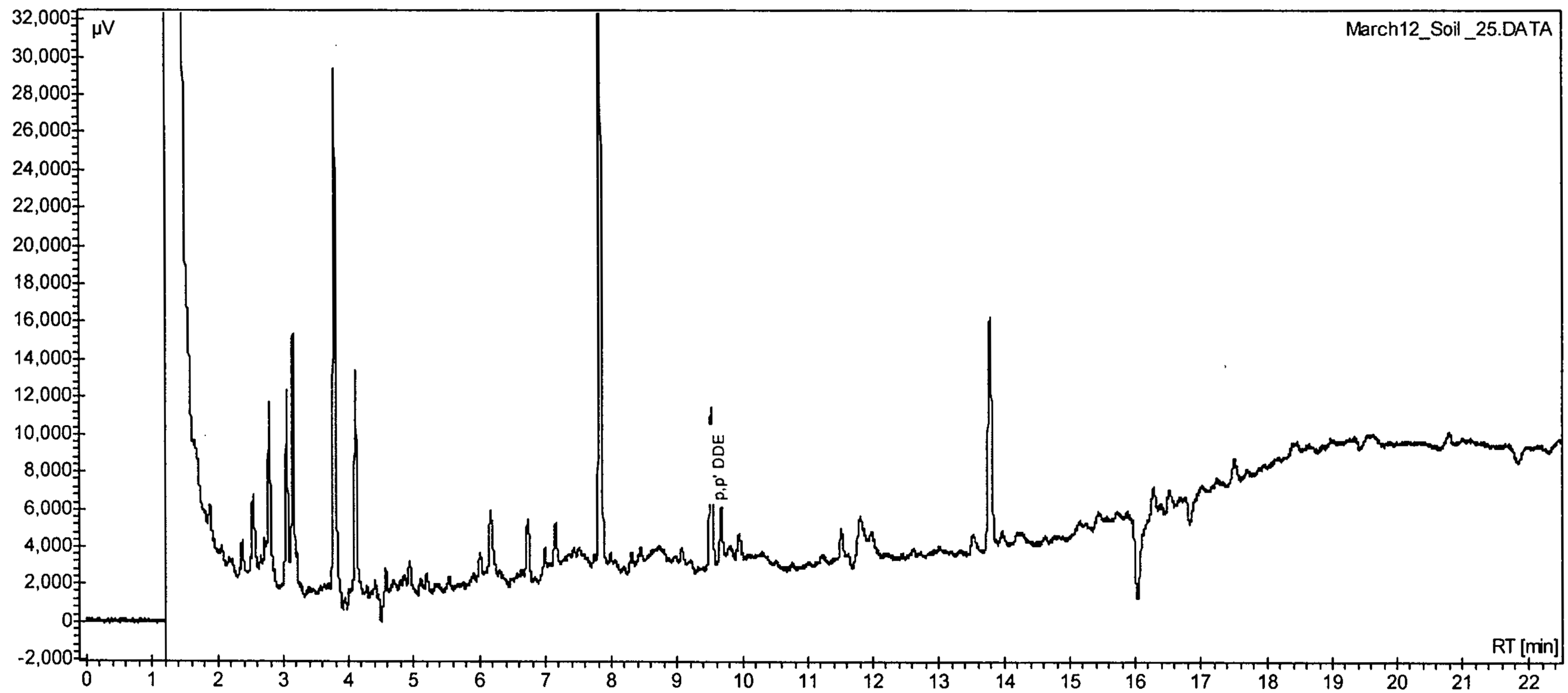
Chromatogram: Std HBs 1.0 ppm



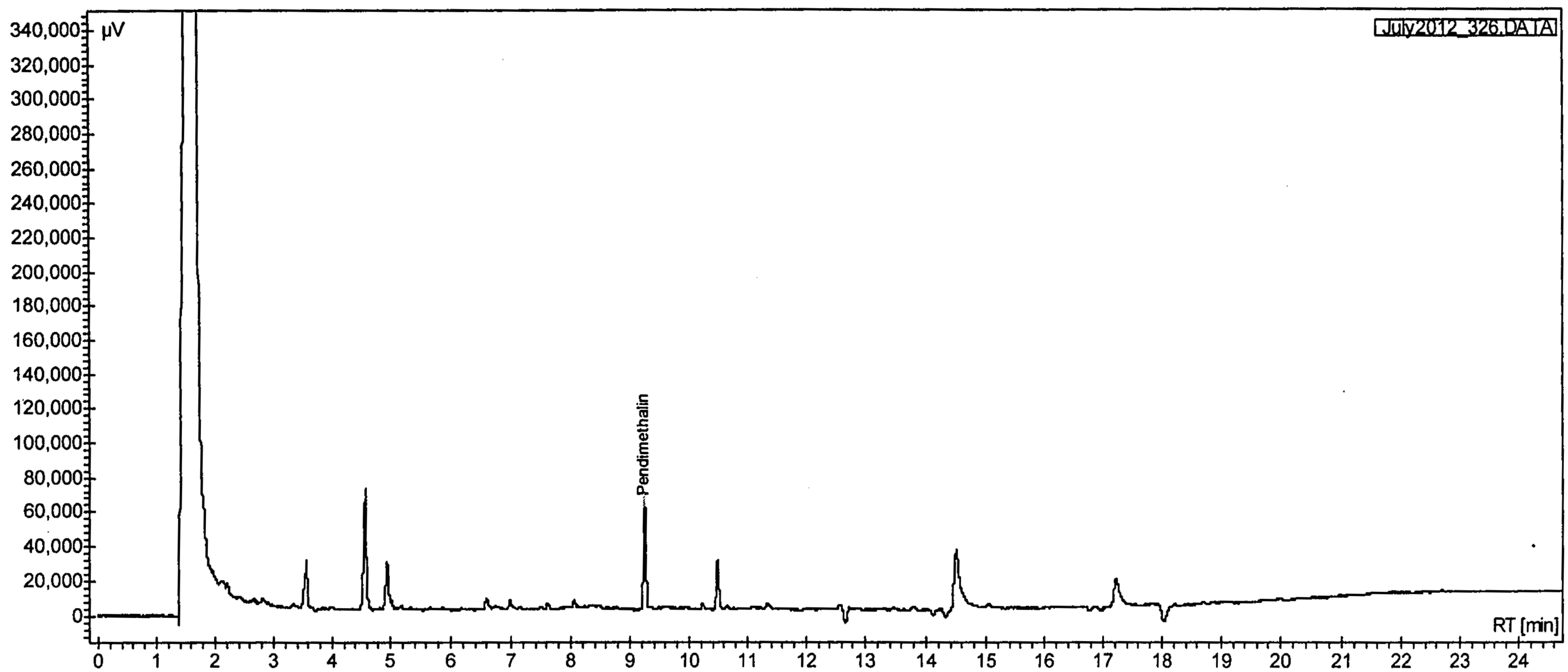
Chromatogram of soil: Totarmata



Chromatogram of soil: Jambusar-1



Chromatogram of soil: Kurali



Chromatogram of soil: Ranu-1

AINP ON PESTICIDE RESIDUE LABORATORY
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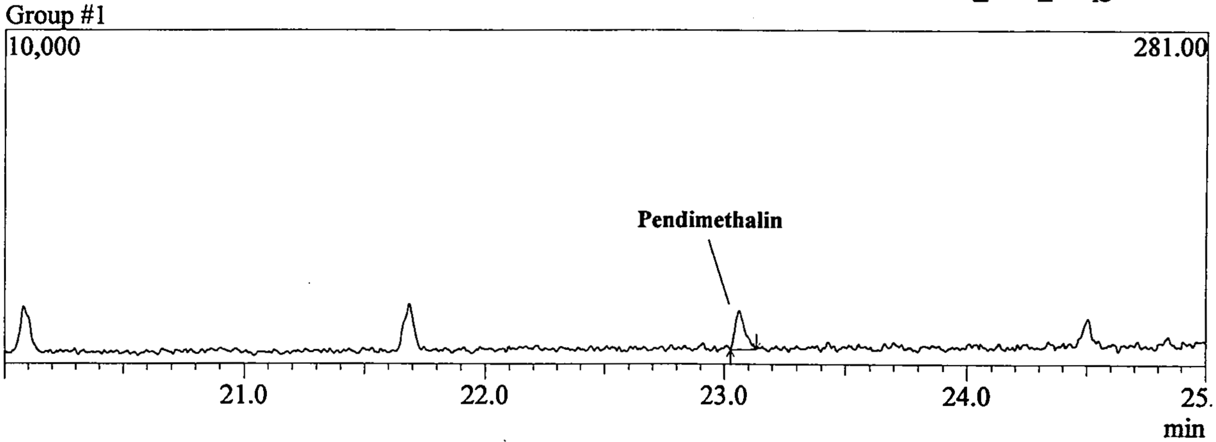
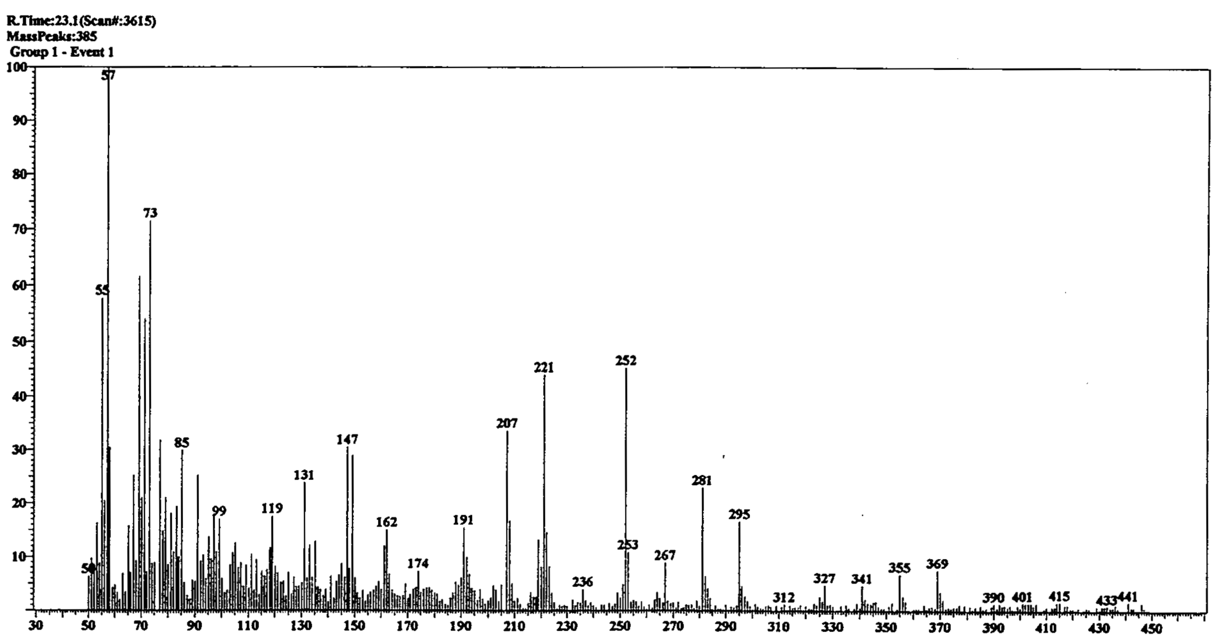
Sample Information

Analyzed	: 8/24/2012 11:23:40 AM
Sample Name	: Soil-5 Ranu Pendi
Sample ID	: Soil-5 Ranu Pendi
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_31.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram Soil-5 Ranu Pendi C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_31.qgd



Spectrum



Peak Report

Peak#	R.Time	Name	Area	Height
1	23.062	Pendimethalin	3205	1142
			3205	1142

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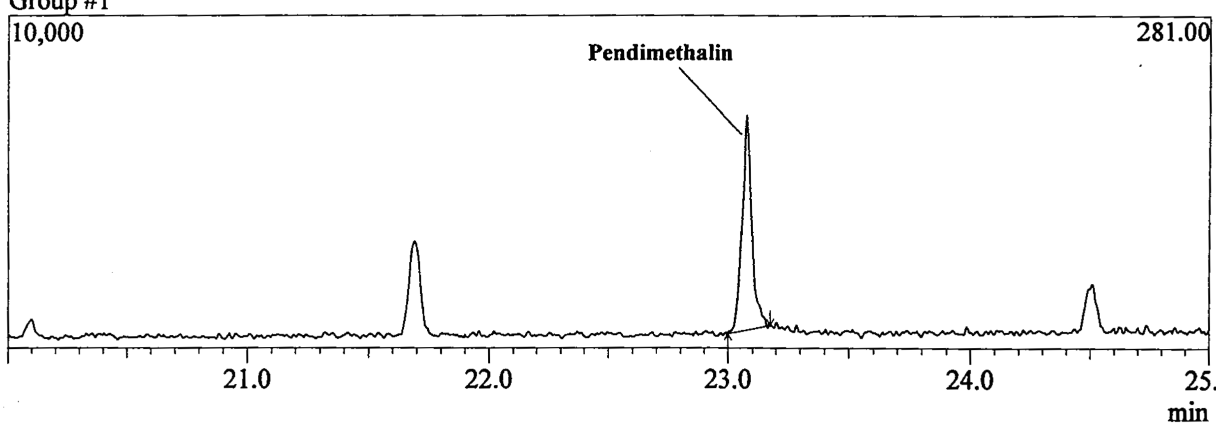
Sample Information

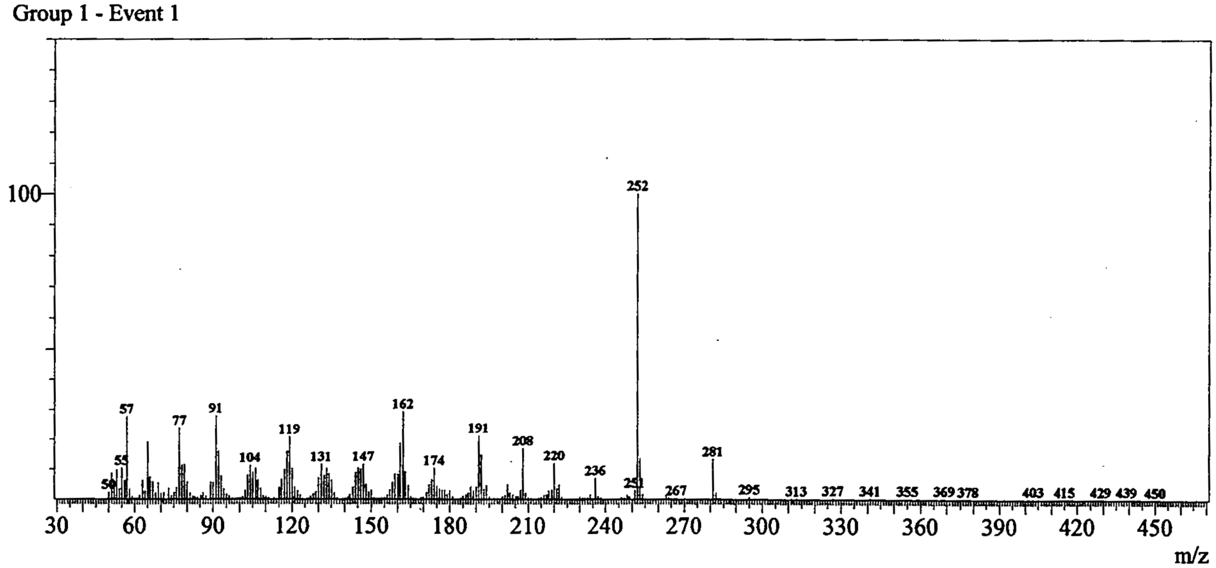
Analyzed	: 8/24/2012 3:24:25 PM
Sample Name	: HBs-6 1.0ppm
Sample ID	: HBs-6 1.0ppm
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_36.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram HBs-6 1.0ppm C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_36.qgd



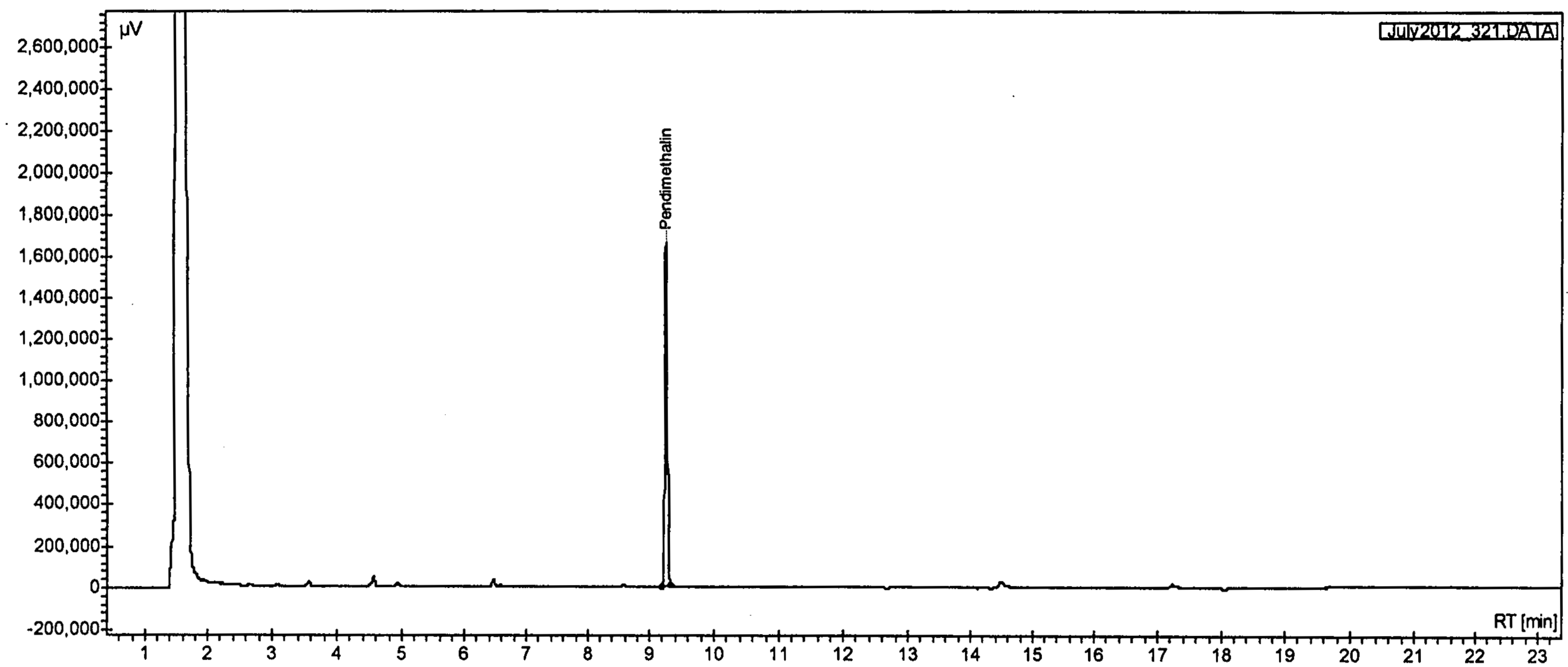
Spectrum

R.Time:23.1(Scan#:3615)
MassPeaks:393
Group 1 - Event 1



Peak Report

Peak#	R.Time	Name	Area	Height
1	23.075	Pendimethalin	17661	6501
			17661	6501



Chromatogram of soil: Latipura-1

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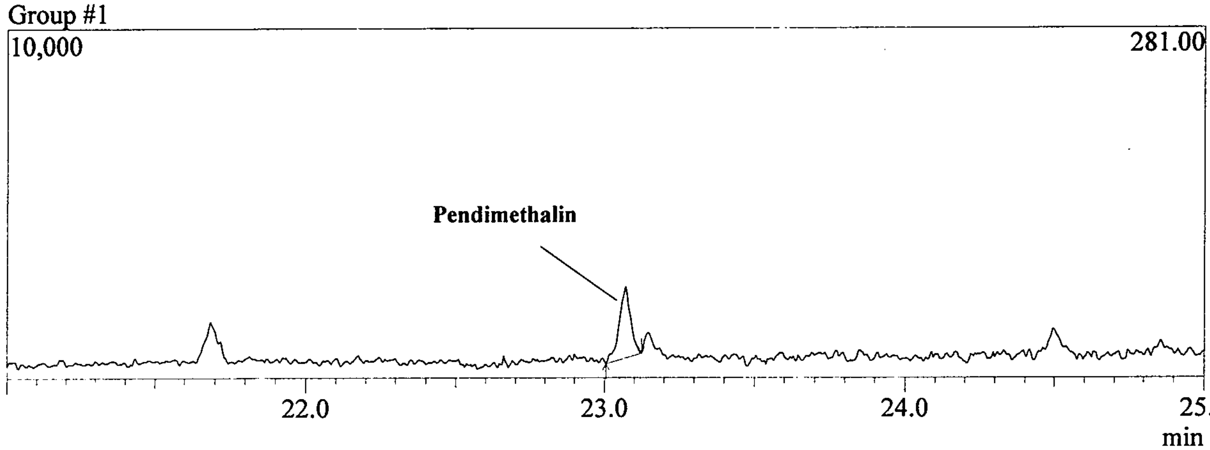
Sample Information

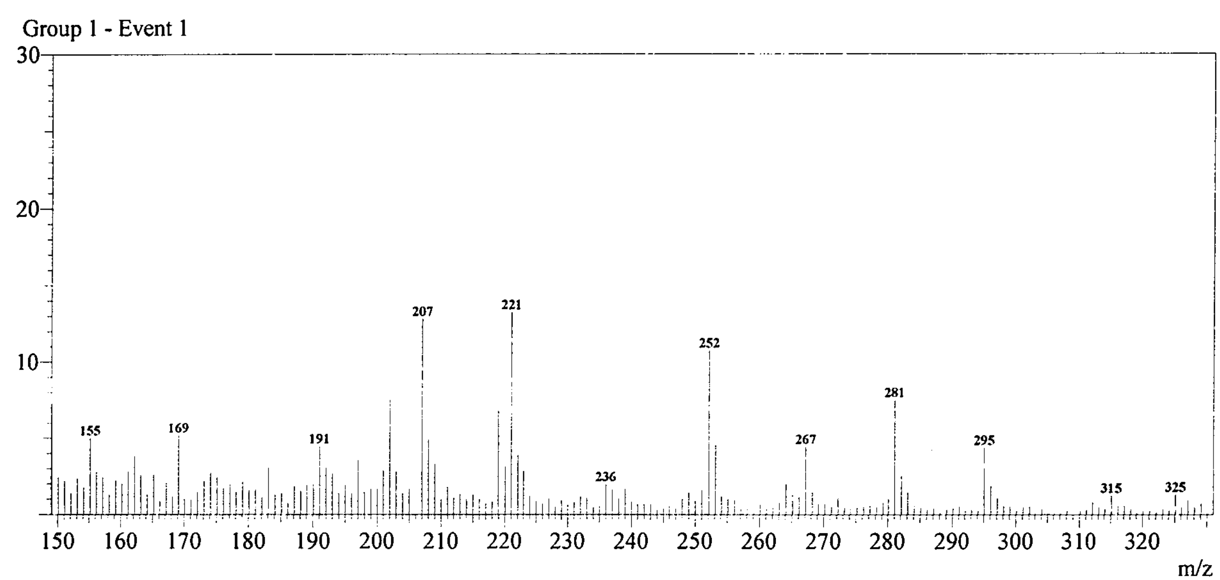
Analyzed	: 8/23/2012 6:56:59 PM
Sample Name	: Soil-3 Latipura Pendimethaline
Sample ID	: Soil-3 Latipura Pendimethaline
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_11.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram Soil-3 Latipura Pendimethaline C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_11.qgd



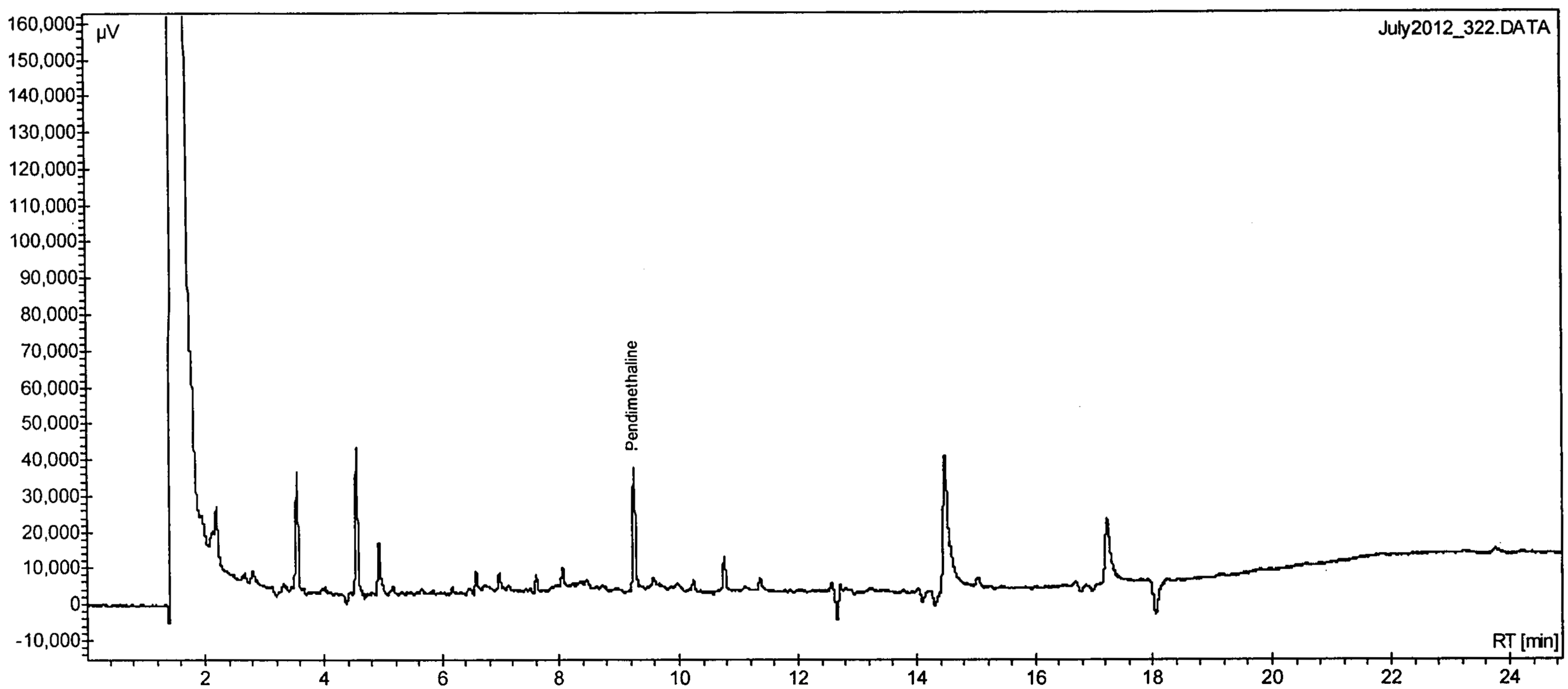
Spectrum

R.Time:23.1(Scan#:3621)



Peak Report

Peak#	R.Time	Name	Area	Height
1	23.070	Pendimethalin	5483	2046
			5483	2046



Chromatogram of soil: Latipura-2

AINP ON PESTICIDE RESIDUE LABORATORY
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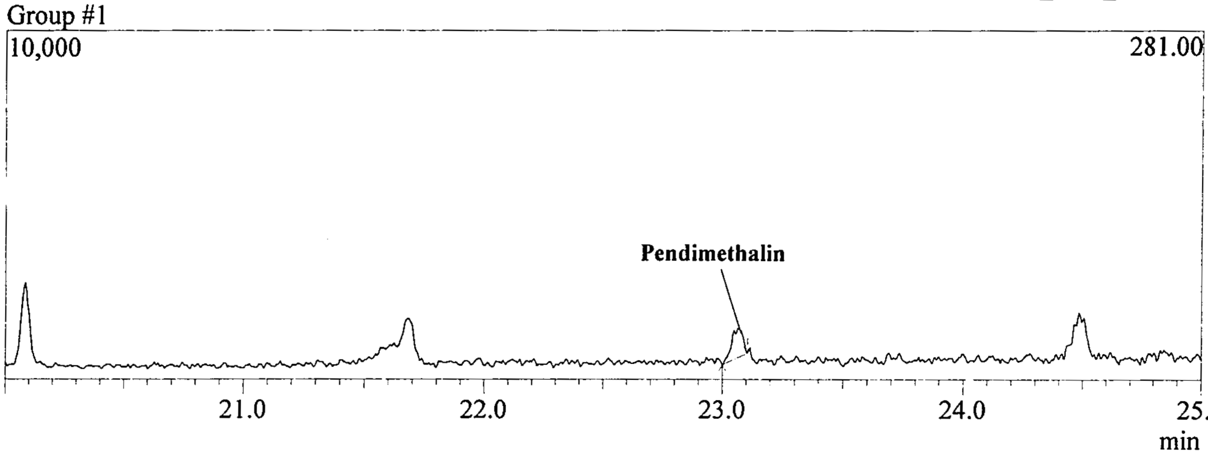
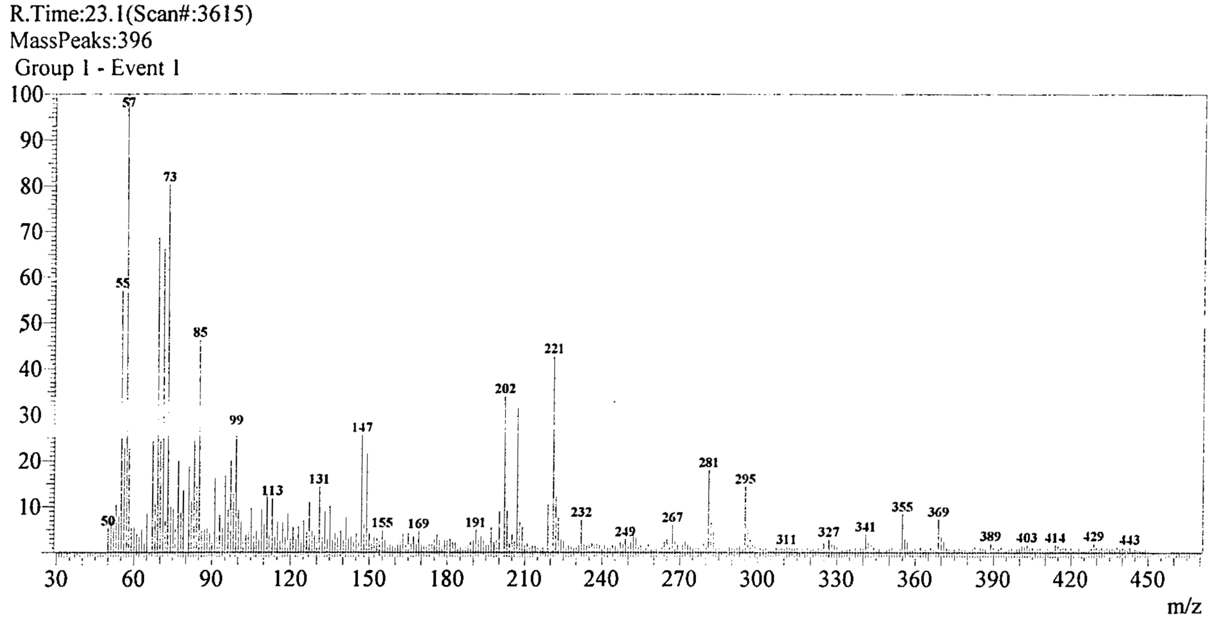
Sample Information

Analyzed	: 8/24/2012 9:47:14 AM
Sample Name	: Soil-4 Latipura Pendimethaline
Sample ID	: Soil-4 Latipura Pendimethaline
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_29.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram Soil-4 Latipura Pendimethaline C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_29.qgd

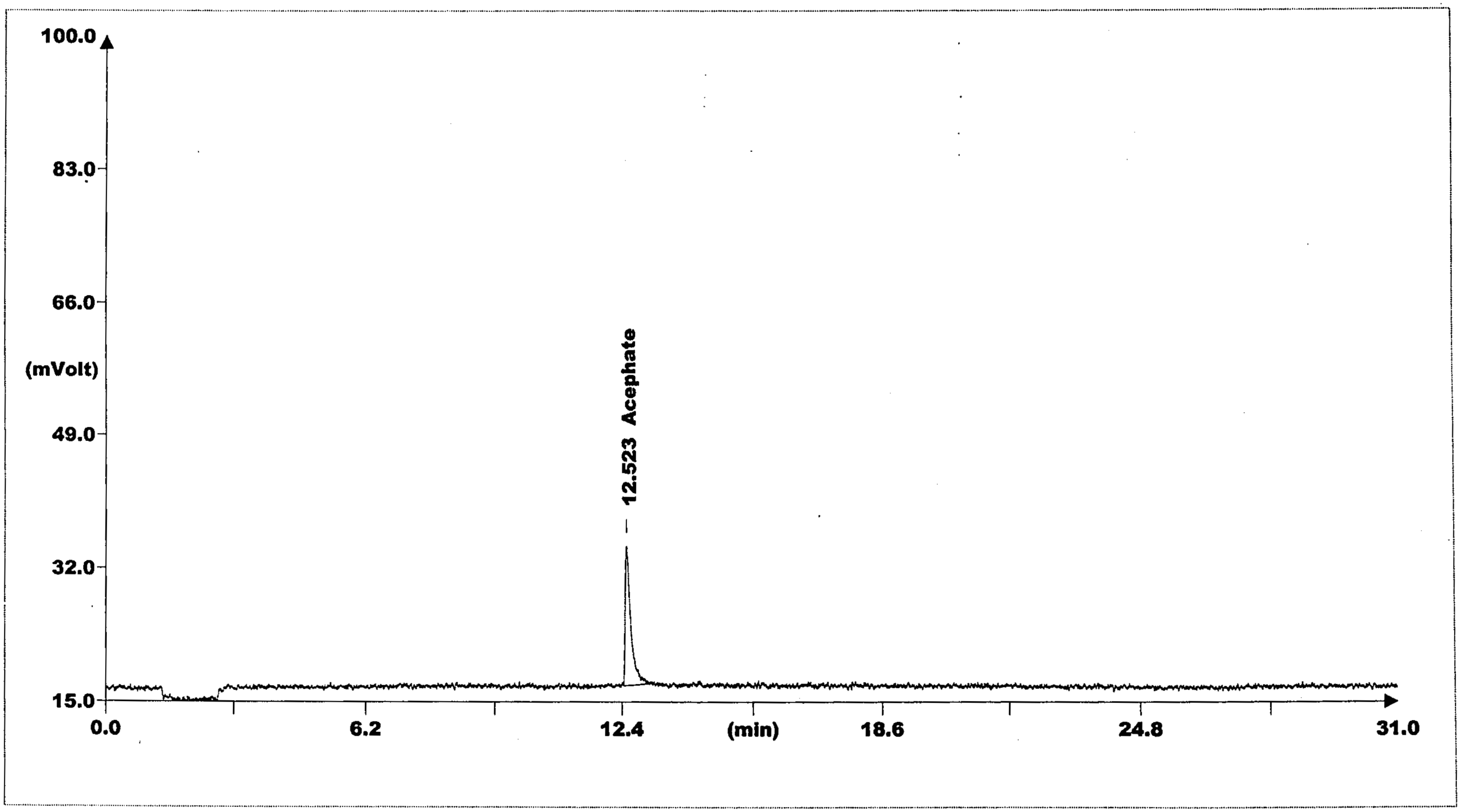


Spectrum

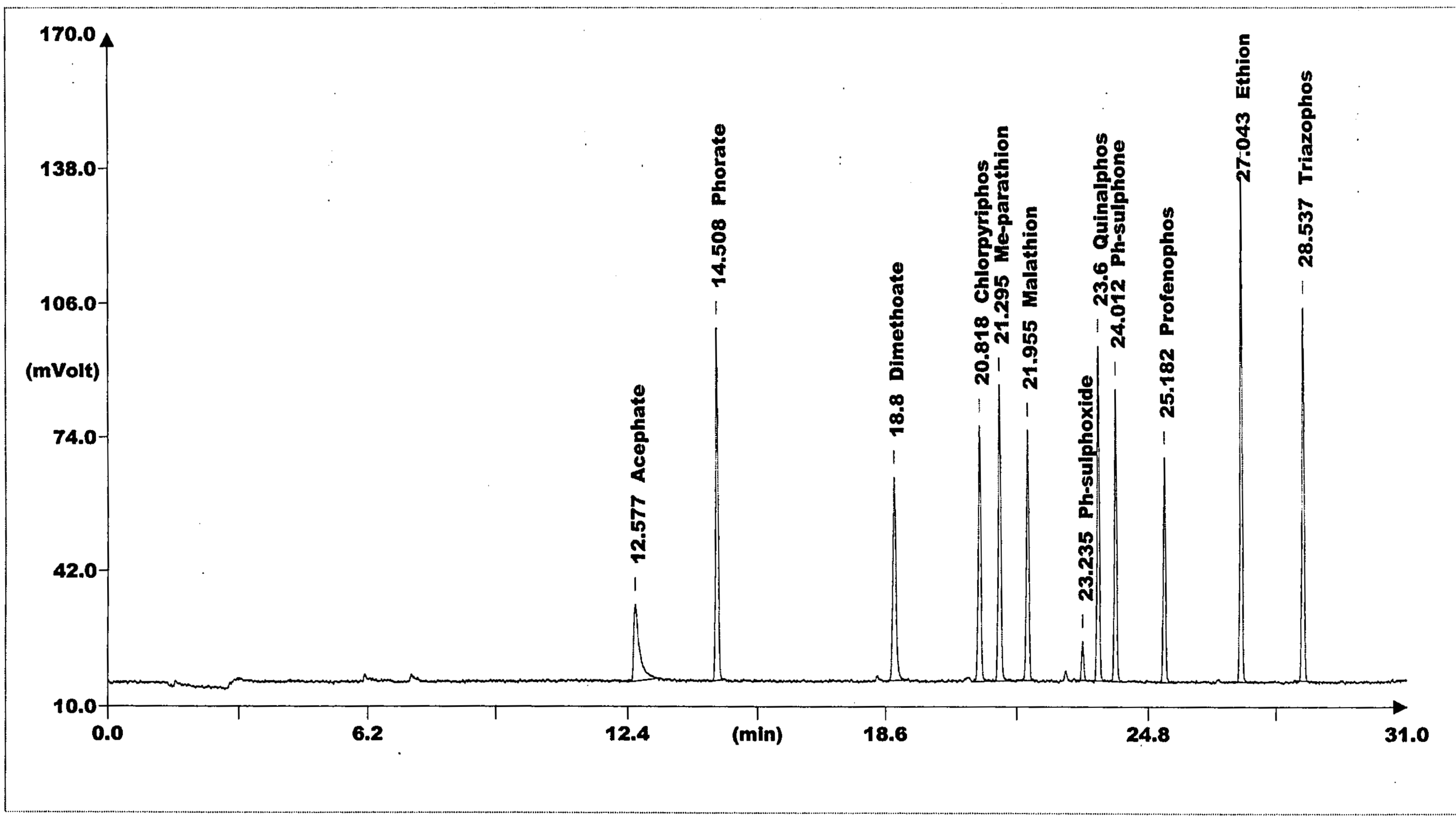


Peak Report

Peak#	R.Time	Name	Area	Height
1	23.068	Pendimethalin	2781	839
			2781	839



Ode-5 (capsicum)



Std OPs 0.1 ppm

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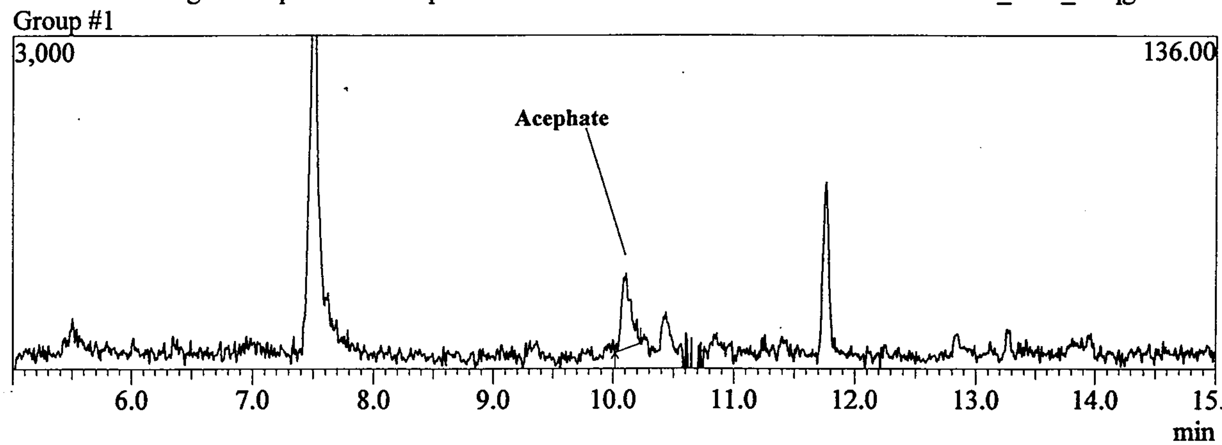
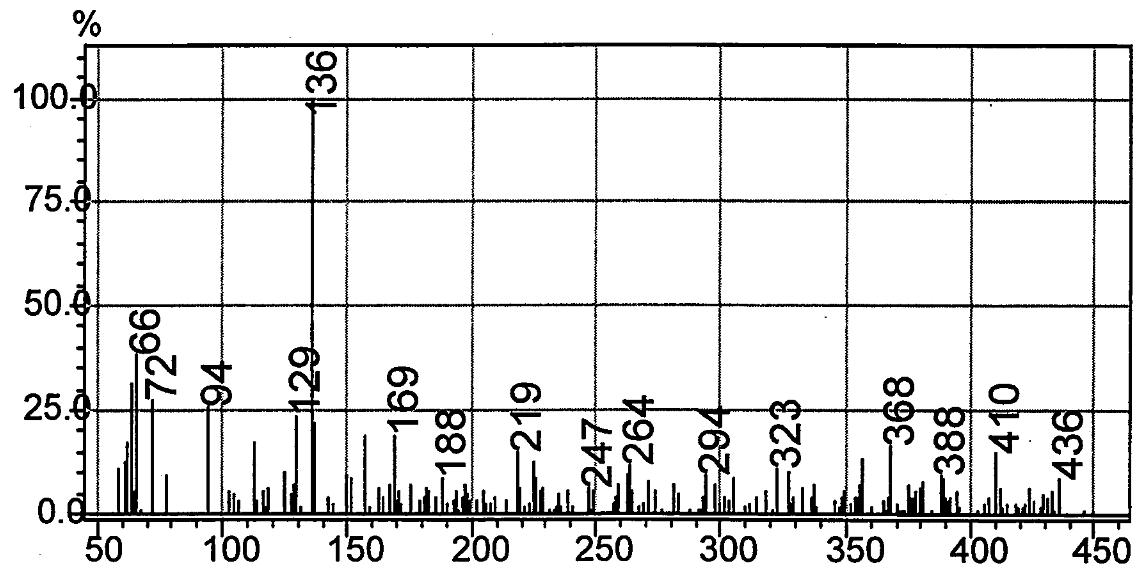
Sample Information

Analyzed	: 8/23/2012 3:44:25 PM
Sample Name	: Capsicum-5 acephate
Sample ID	: Capsicum-5 acephate
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_07.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

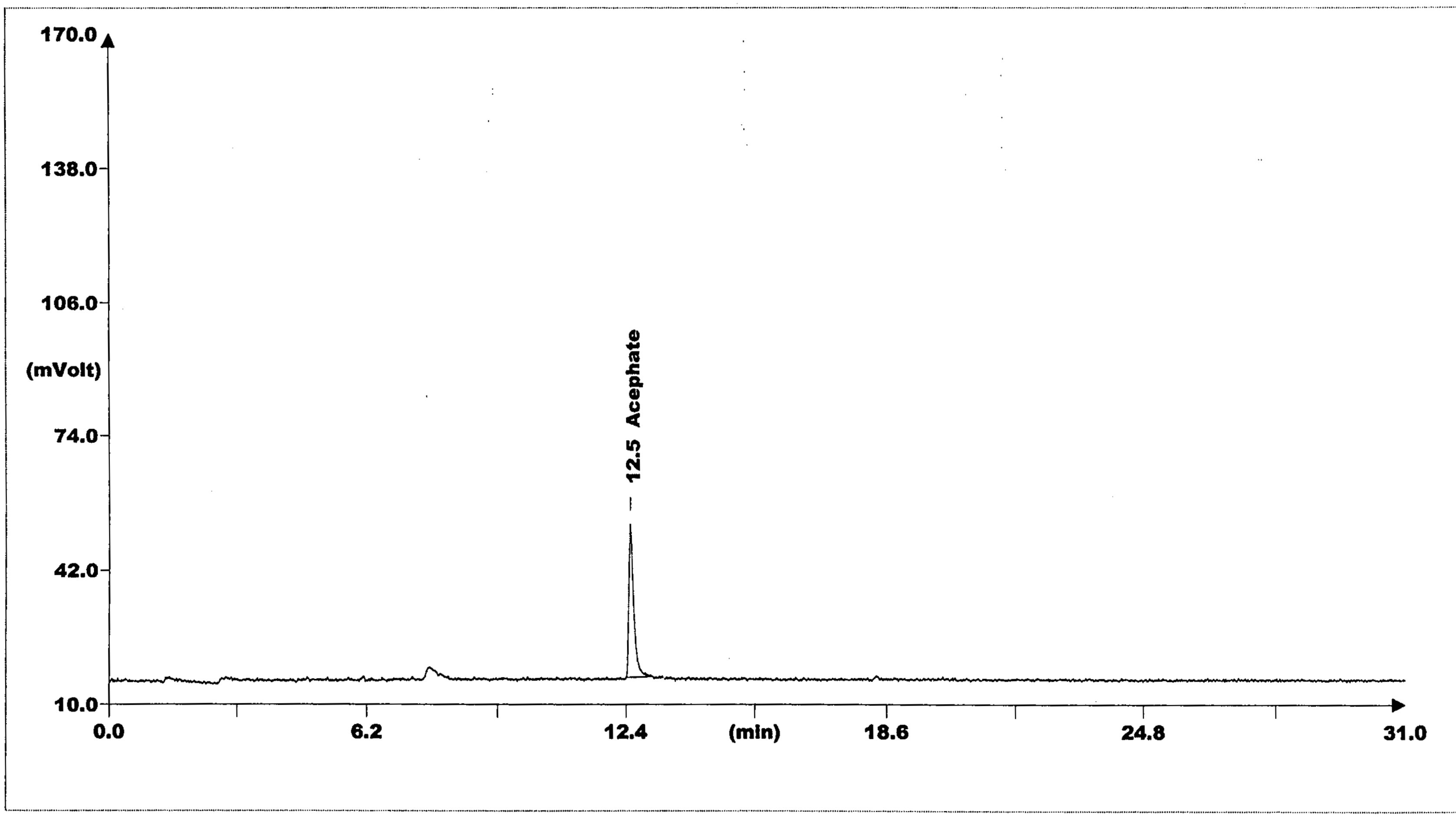
Chromatogram Capsicum-5 acephate C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_07.qgd



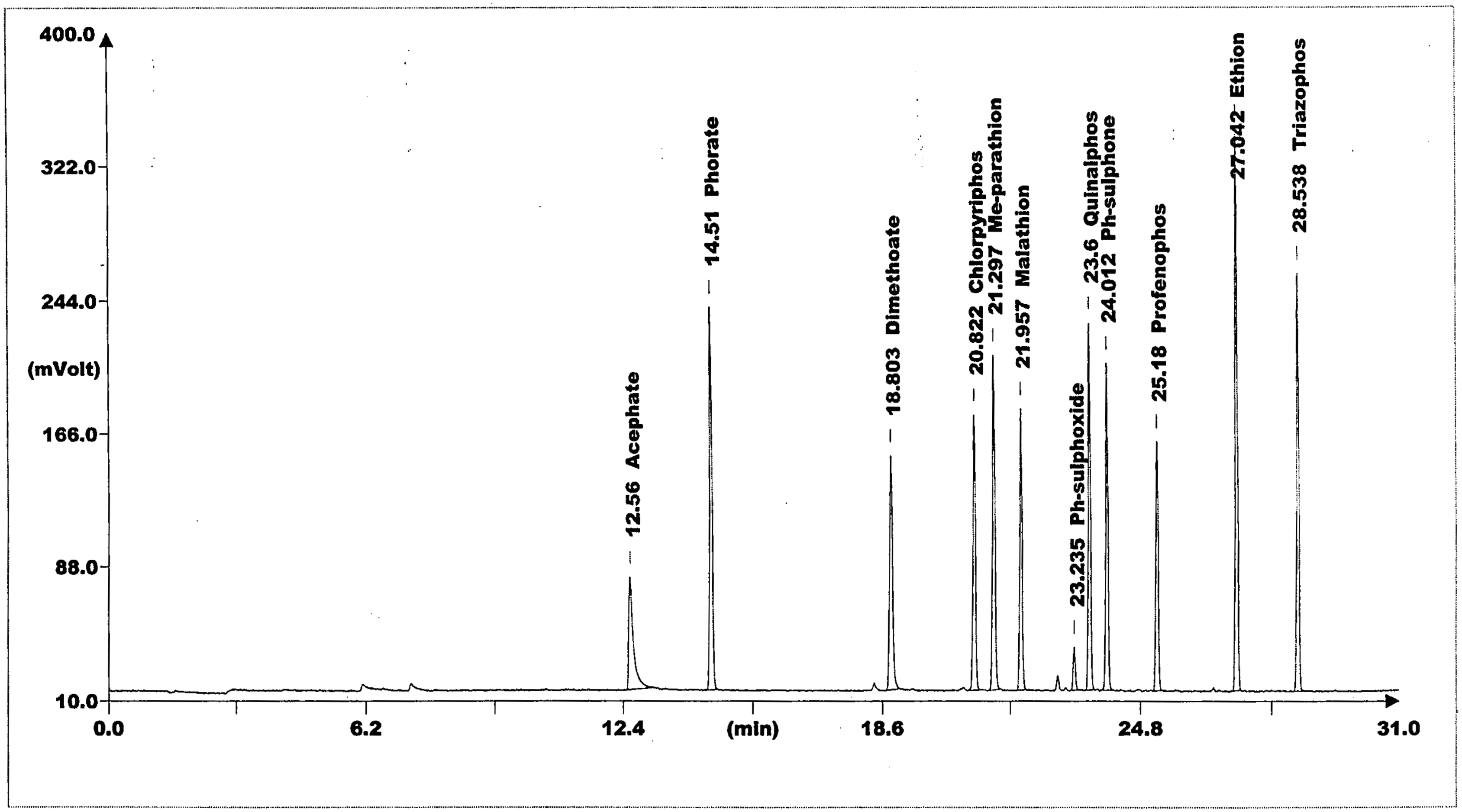


Peak Report

Peak#	R.Time	Name	Area	Height
1	10.109	Acephate	3405	641
			3405	641



Anandi B (Okra)



Std OPs 0.25ppm

AINP ON PESTICIDE RESIDUE LABORATORY
ANAND AGRICULTURAL UNIVERSITY
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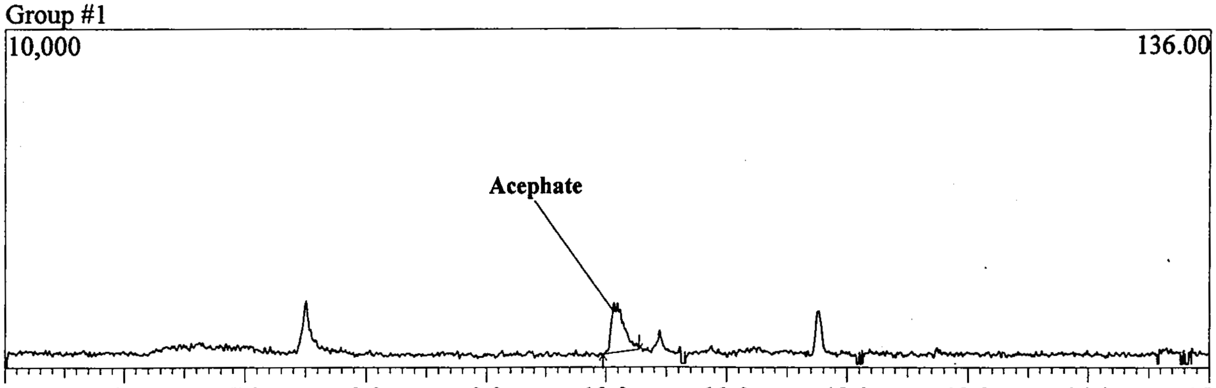
Sample Information

Analyzed	: 8/23/2012 4:32:32 PM
Sample Name	: PRL-048 ok/24/05/12 Acephate
Sample ID	: PRL-048 ok/24/05/12 Acephate
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_08.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram PRL-048 ok/24/05/12 Acephate C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_08.qgd

Group #1

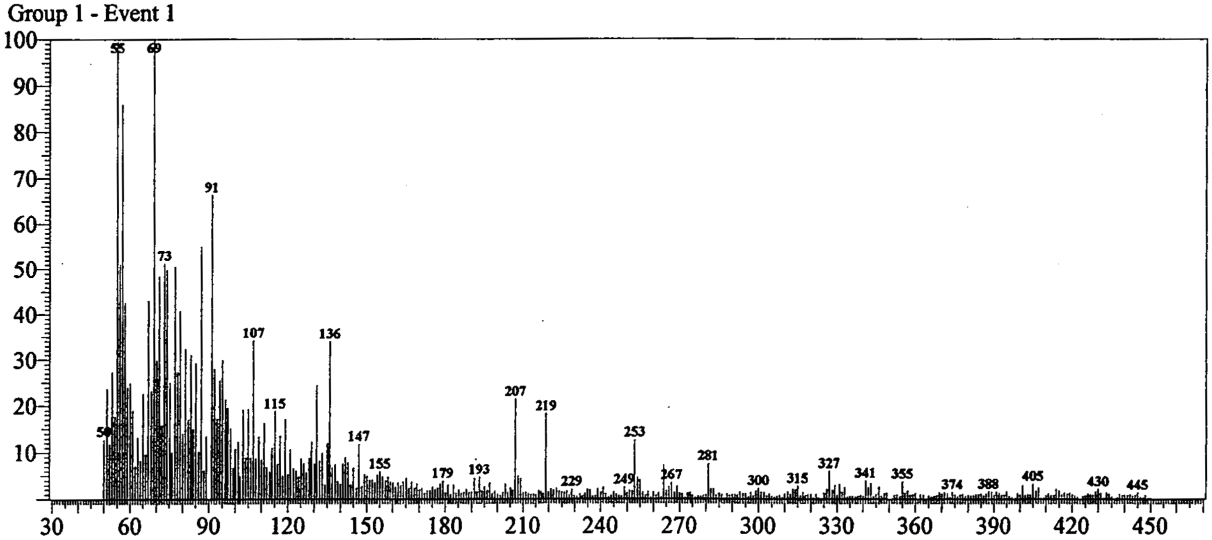


Spectrum

R.Time:10.1(Scan#:1014)

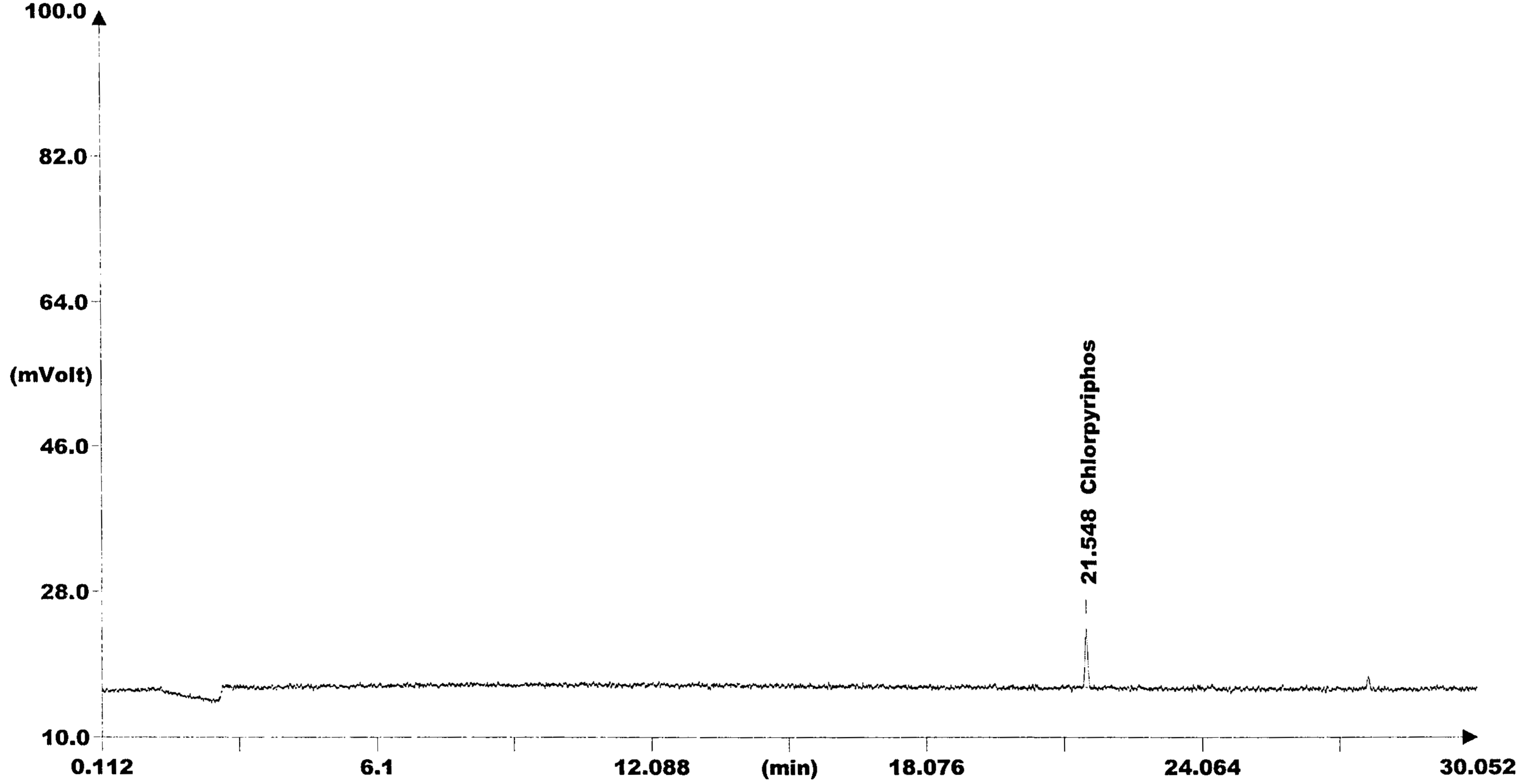
MassPeaks:395

Group 1 - Event 1



Peak Report

Peak#	R.Time	Name	Area	Height
1	10.067	Acephate	10618	1504
			10618	1504



Chromatogram : Cotton lint Ghora-4

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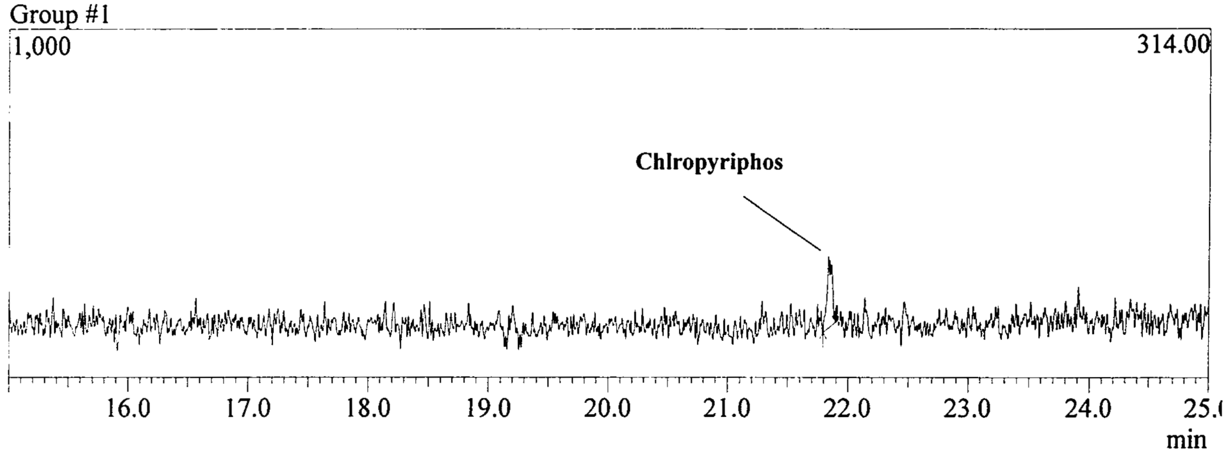
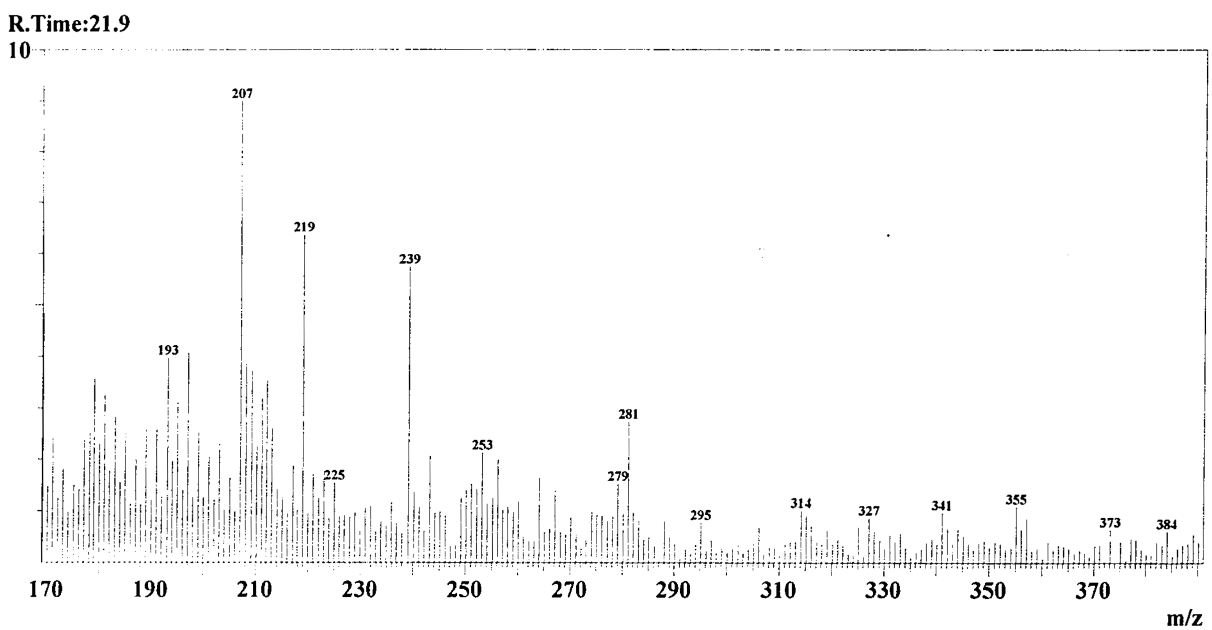
Sample Information

Sample Name	: Ghora-4
Sample ID	: Ghora-4
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_22.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm

Chromatogram

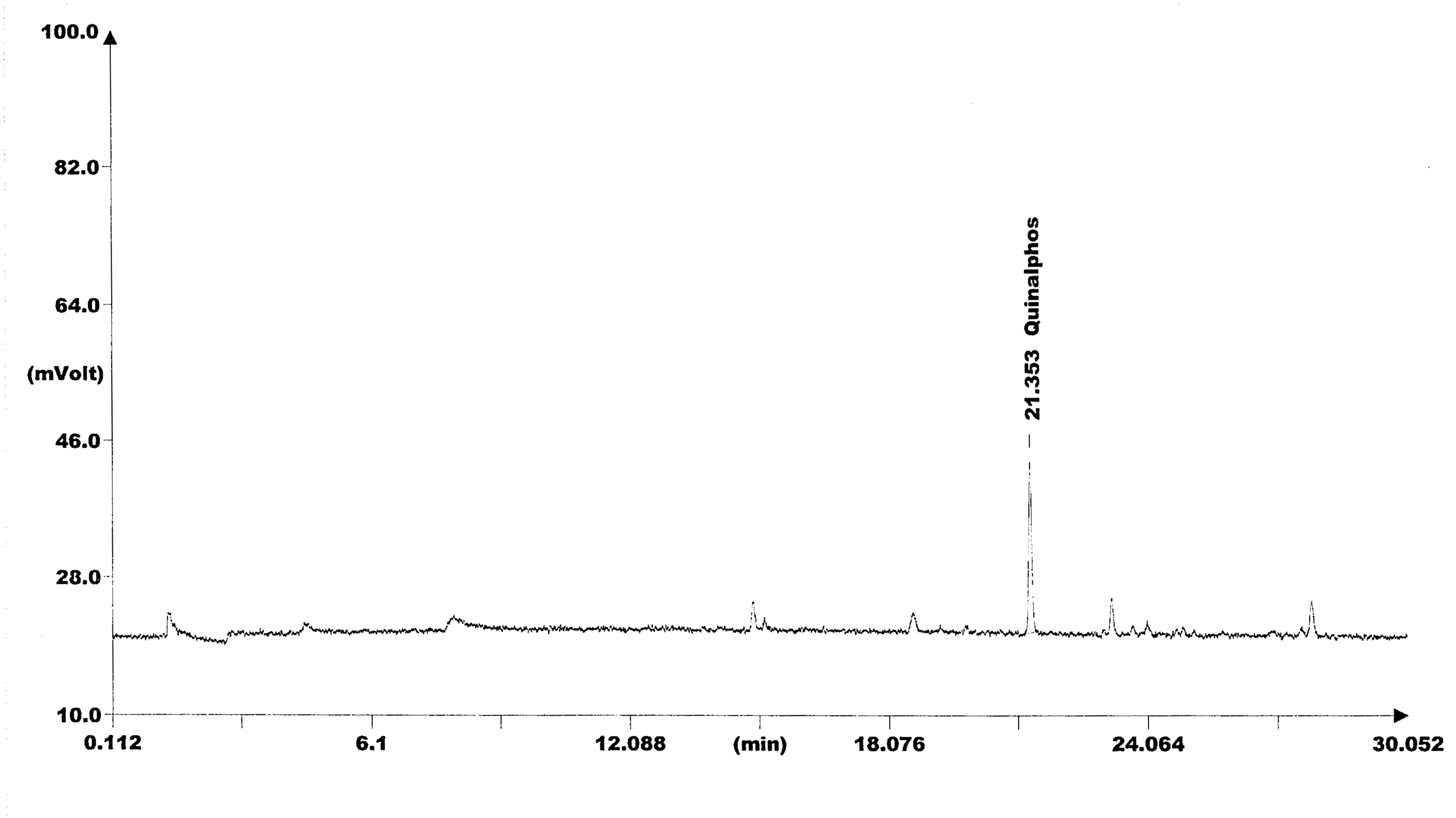


Spectrum



Peak Report

Peak#	R.Time	Name	Area	Height
1	21.837		674	226
			674	226



Chromatogram : Cotton lint Osaram-2

AINP ON PESTICIDE RESIDUE LABORATORY
ANAND AGRICULTURAL UNIVERSITY
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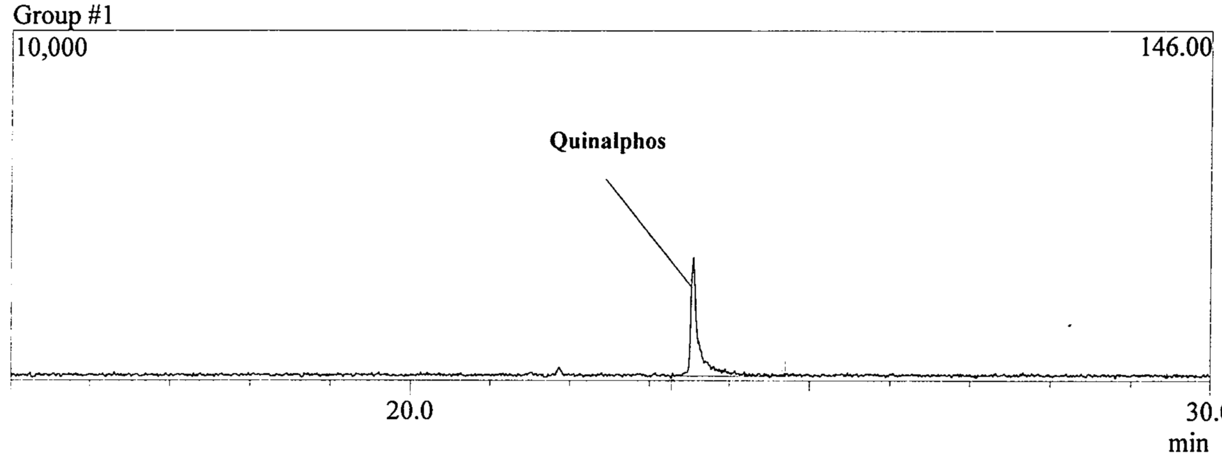
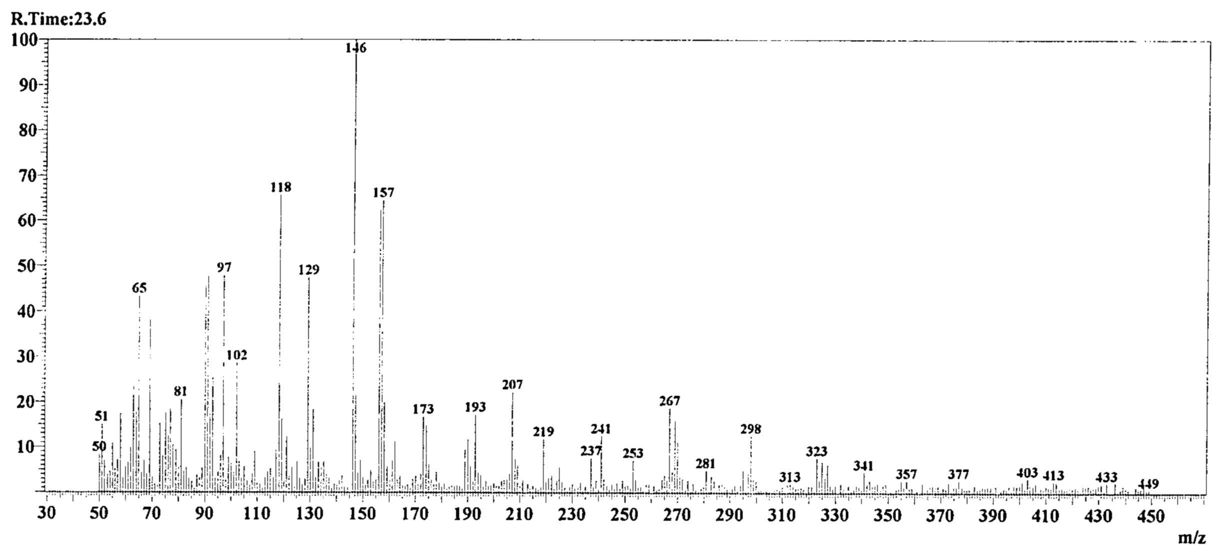
Sample Information

Sample Name : Osaram-2
Sample ID : Osaram-2
Data File : E:\Monitoring Data\4.Monitoring 2012\8. August-2012\Aug_2012_31.qgd
Method File : C:\GCMSsolution\Data\DATA\Monitoring 2012\8. August-2012\Mon_TIC_2012.

Chromatogram

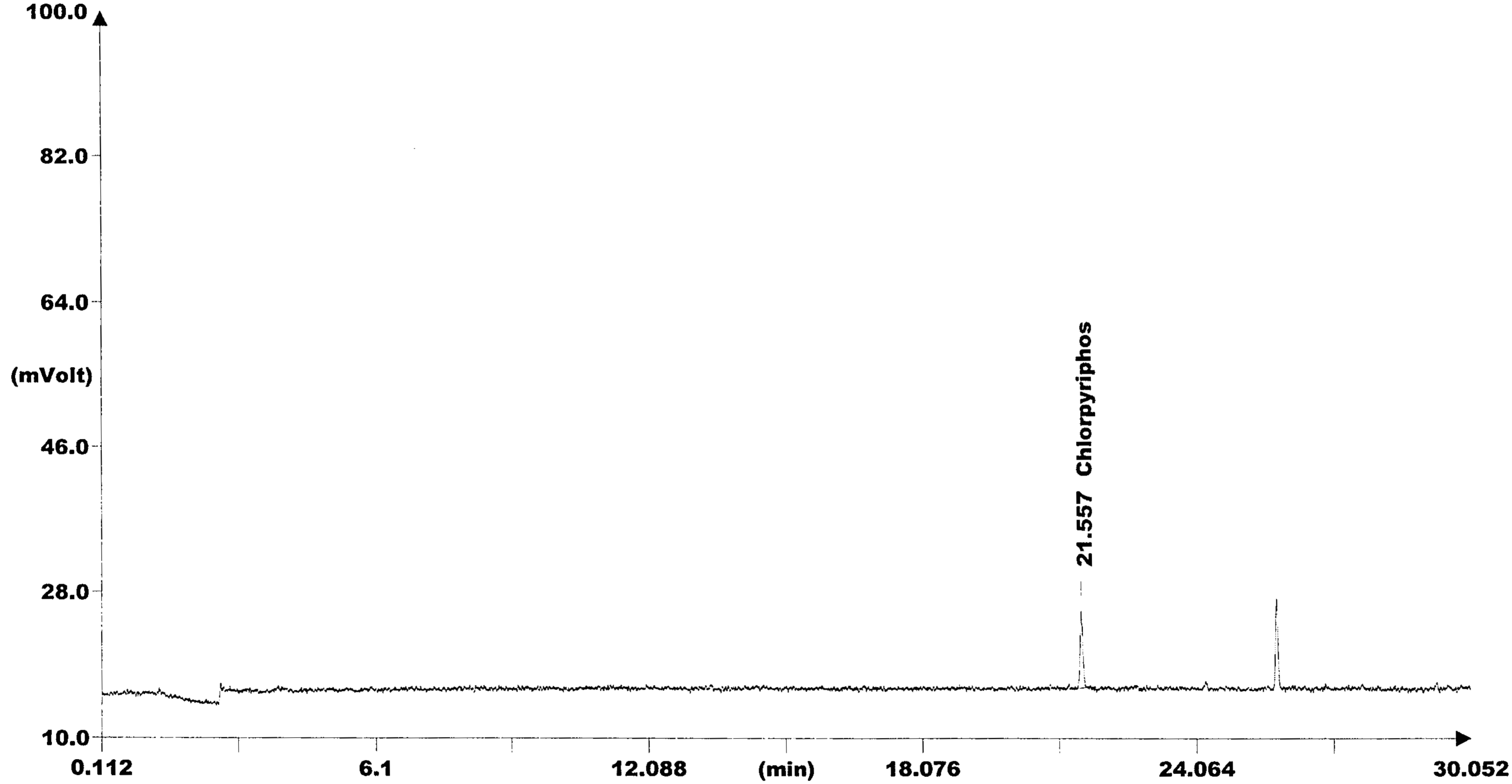


Spectrum



Peak Report

Peak#	R.Time	Name	Area	Height
1	23.549	Quinalphos	24275	3463
			24275	3463



Chromatogram : Cotton lint Sahera-10

AINP ON PESTICIDE RESIDUE LABORATORY
ANAND AGRICULTURAL UNIVERSITY
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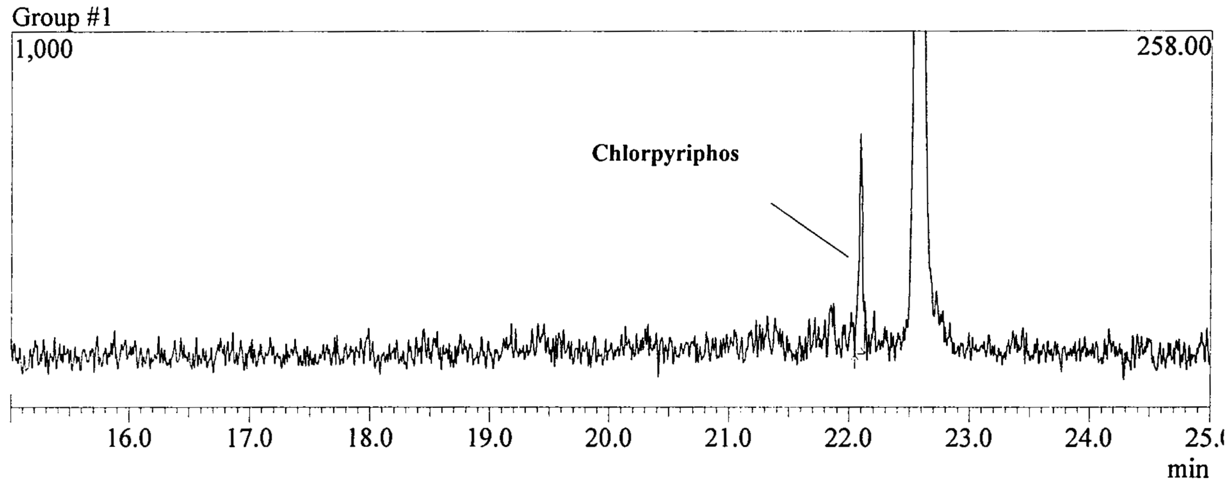
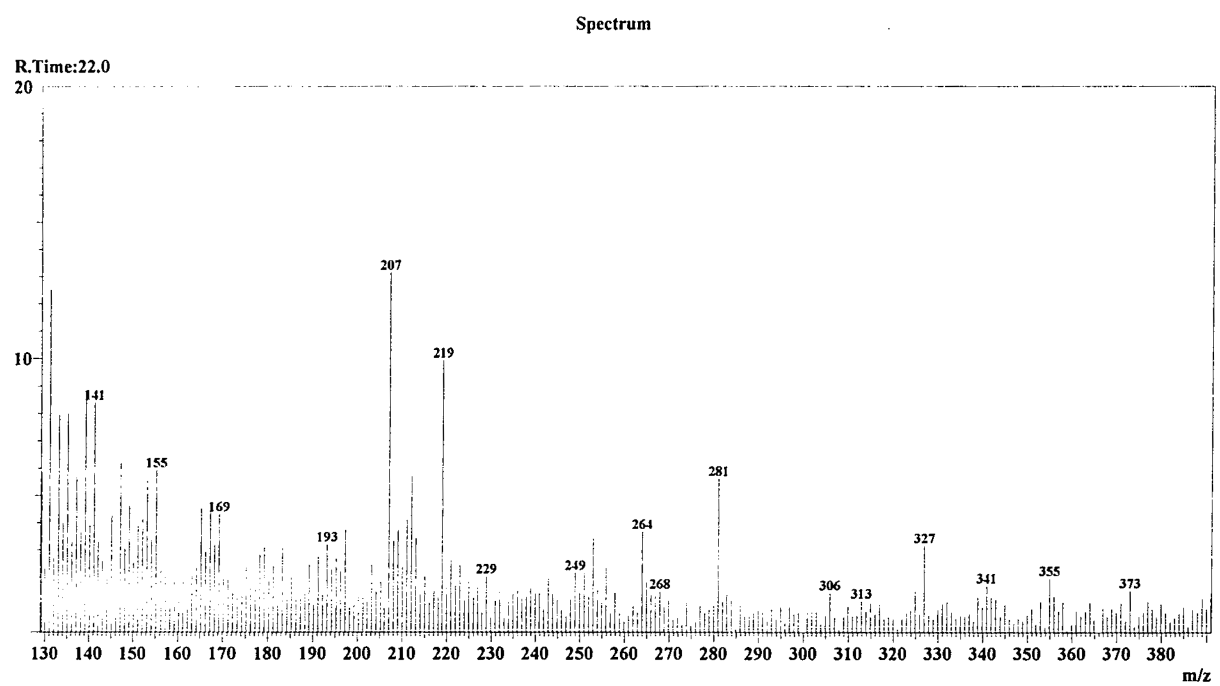
Sample Information

Sample Name	: Sahera-10
Sample ID	: Sahera-10
Data File	: C:\GCMSsolution\Data\DATA\Priti MSc\Priti_MSc_33.qgd
Method File	: C:\GCMSsolution\Data\DATA\Priti MSc\Mon_TIC_2012.qgm





Peak Report

Peak#	R.Time	Name	Area	Height
1	22.091	Chlorpyriphos	1336	643
			1336	643

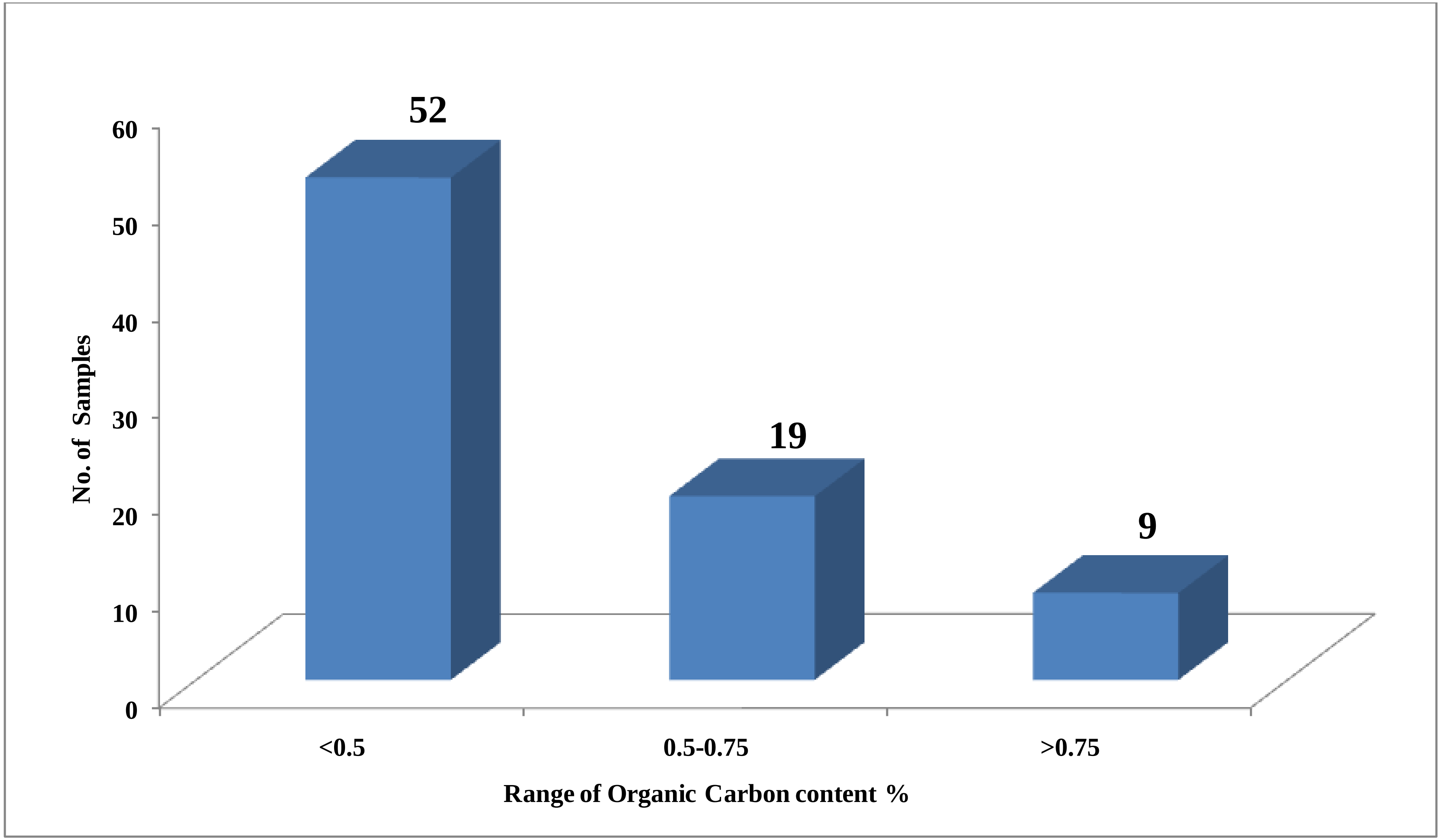


Fig : 11 Organic carbon content in cotton growing soil

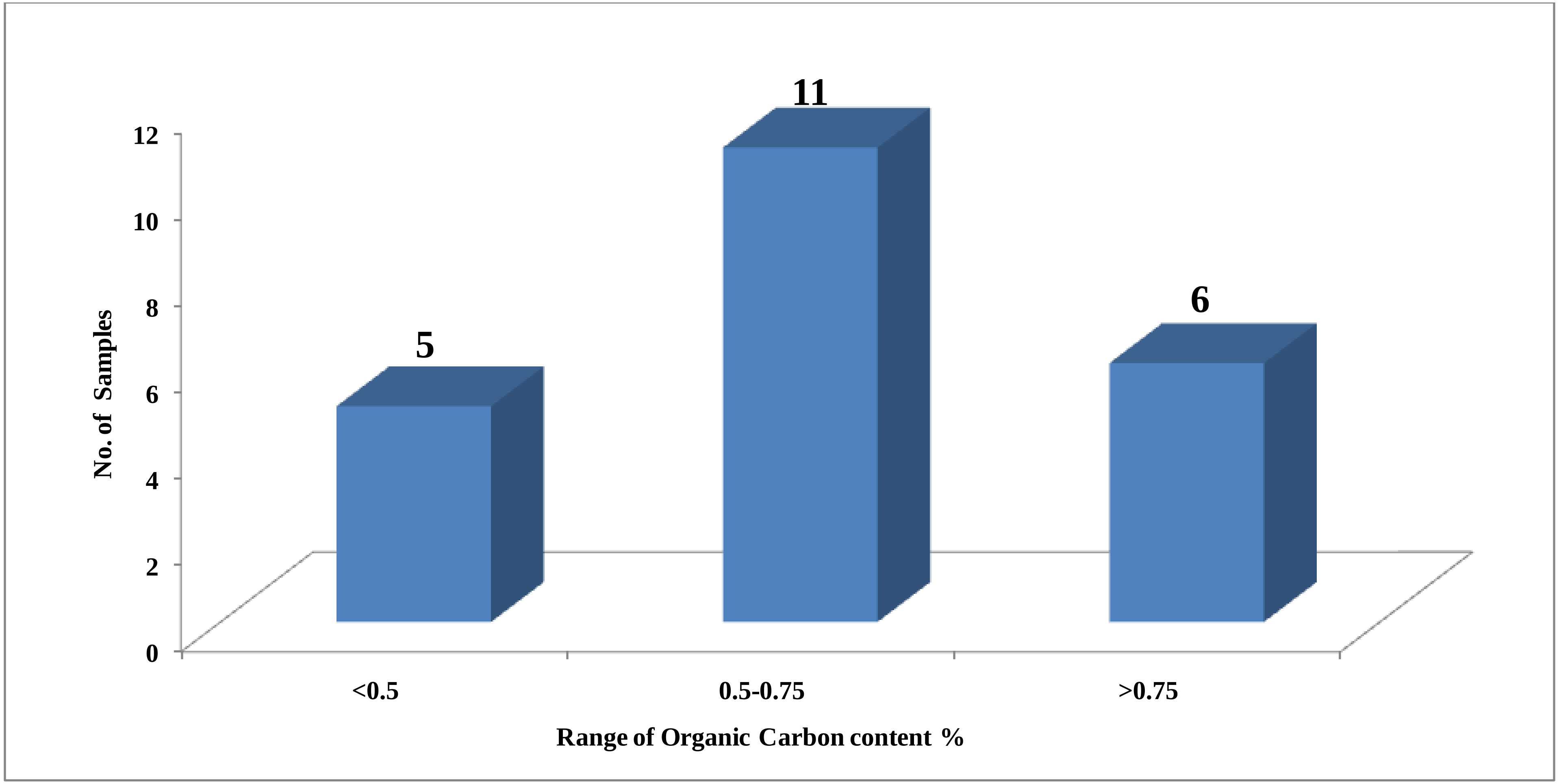


Fig: 12 Organic carbon content from vegetable growing soil

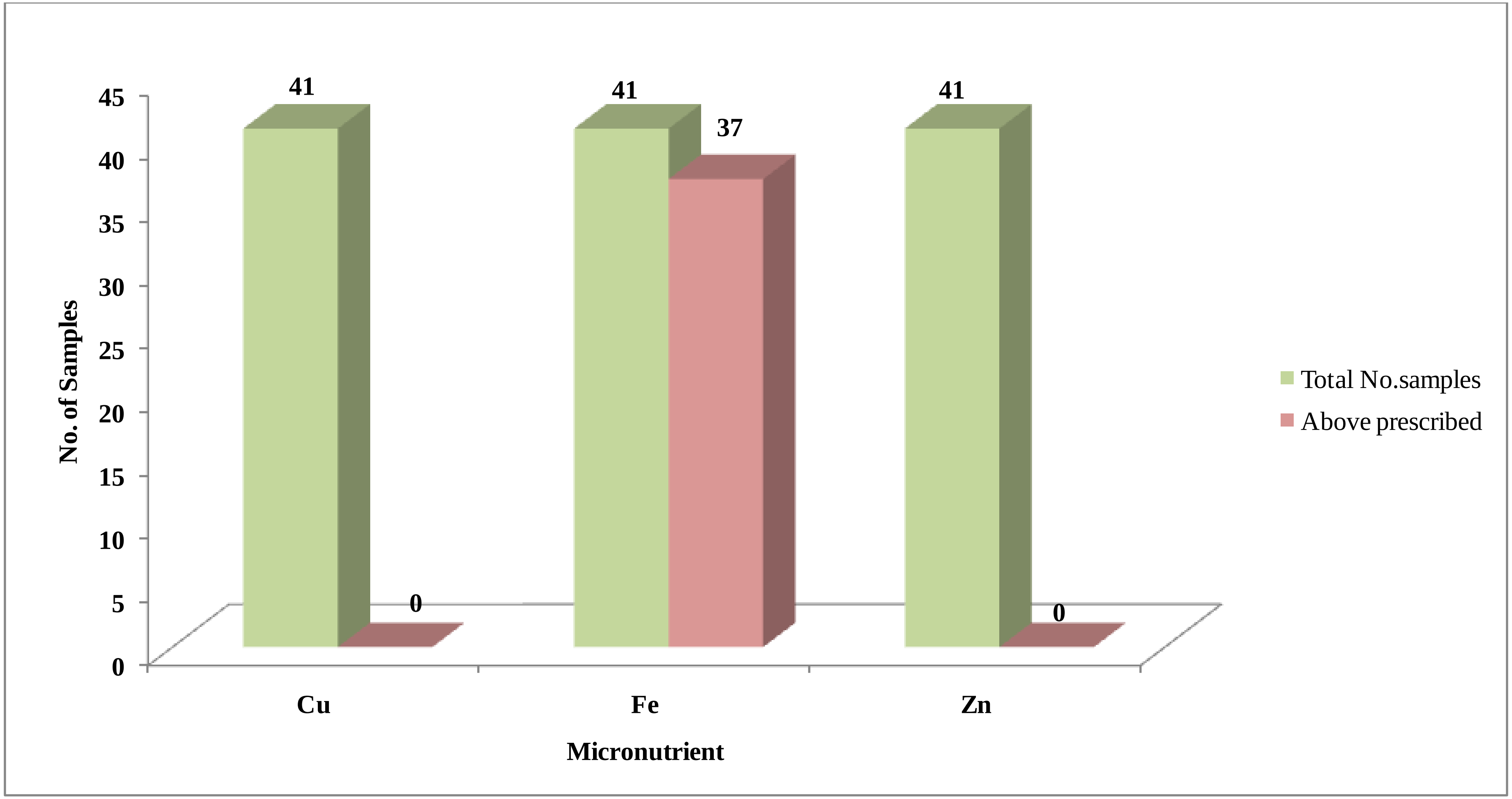


Fig: 5 Micronutrient status in ground water

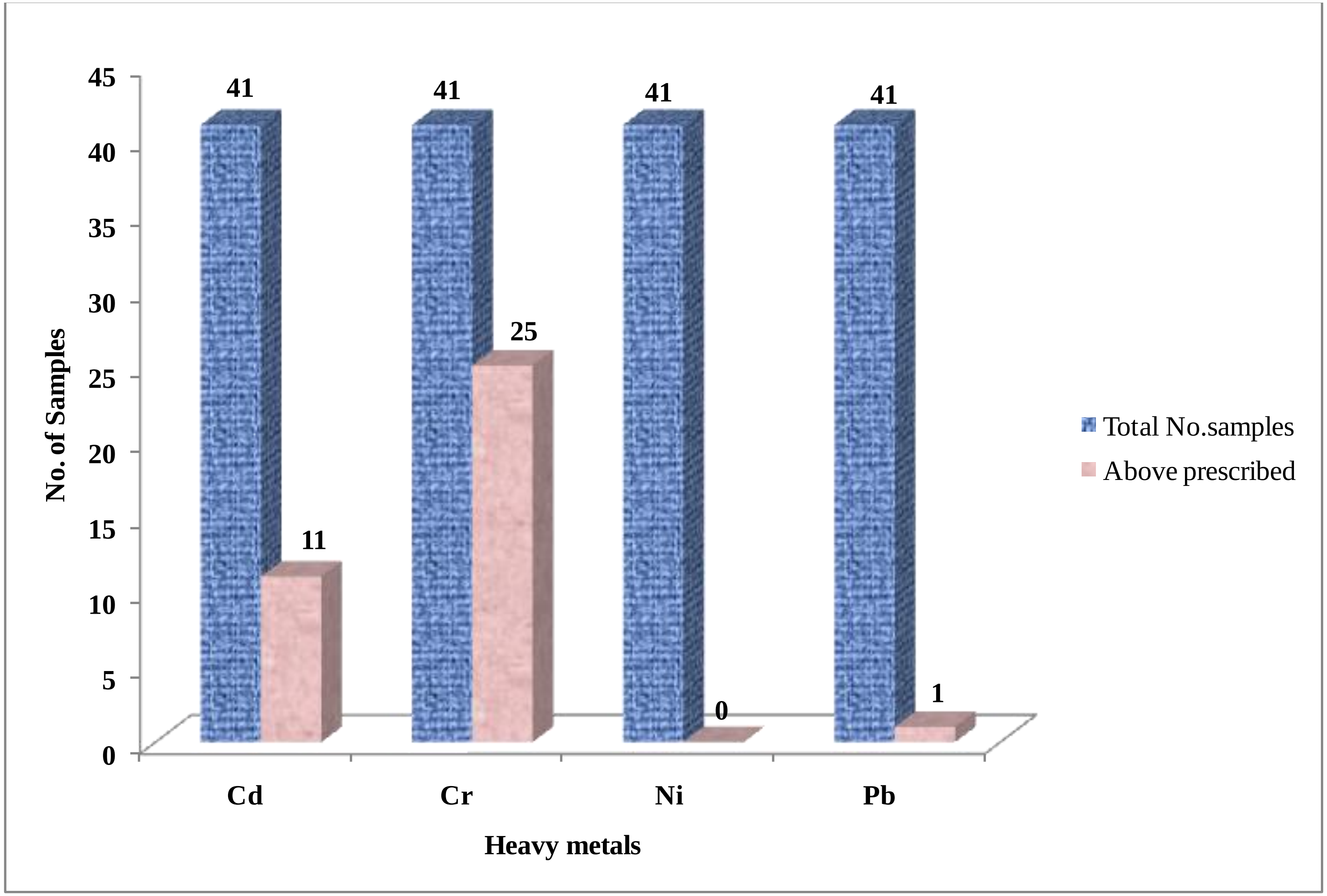


Fig: 6 Heavy metal status in ground water

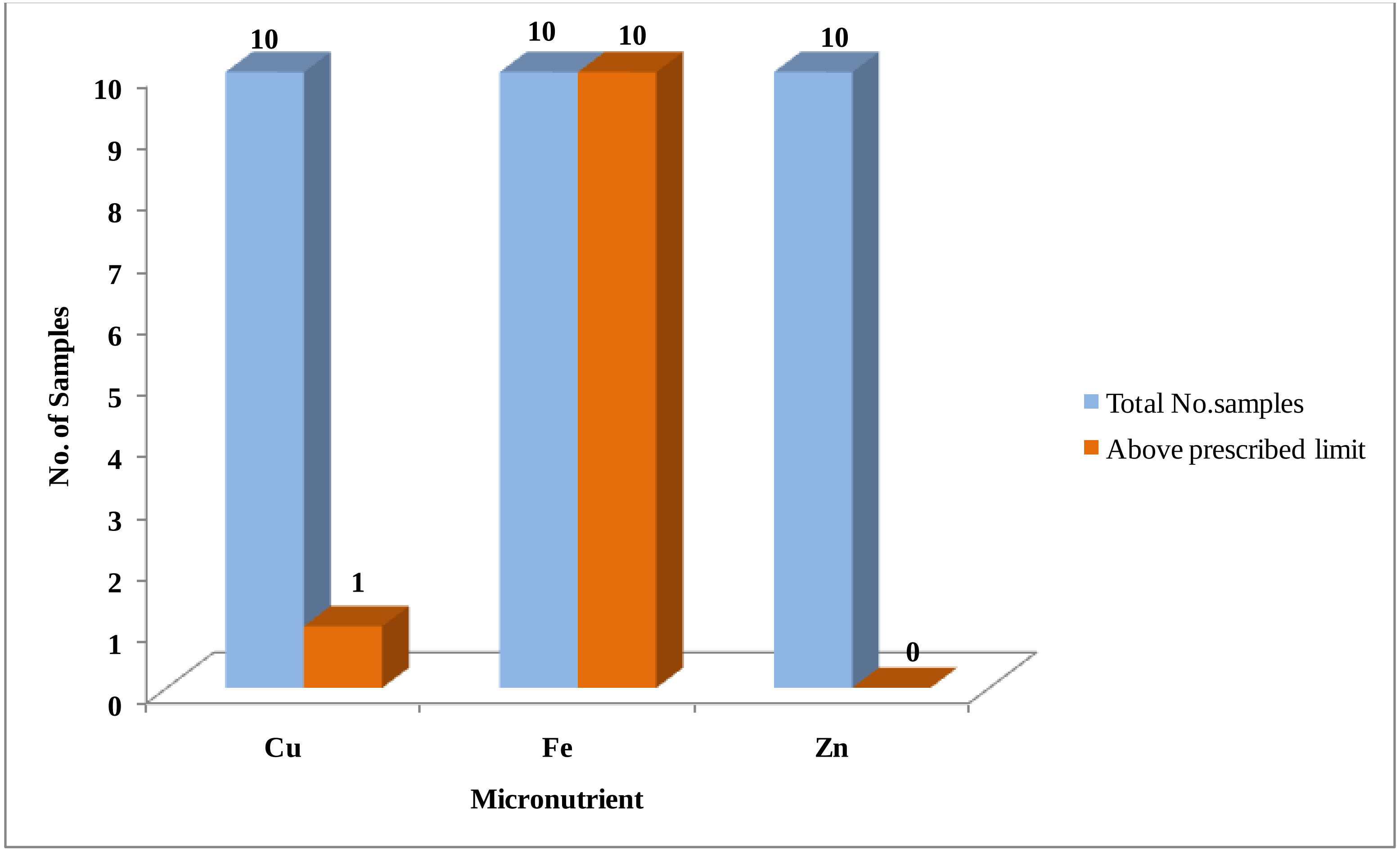


Fig: 7 Micronutrient status in surface water

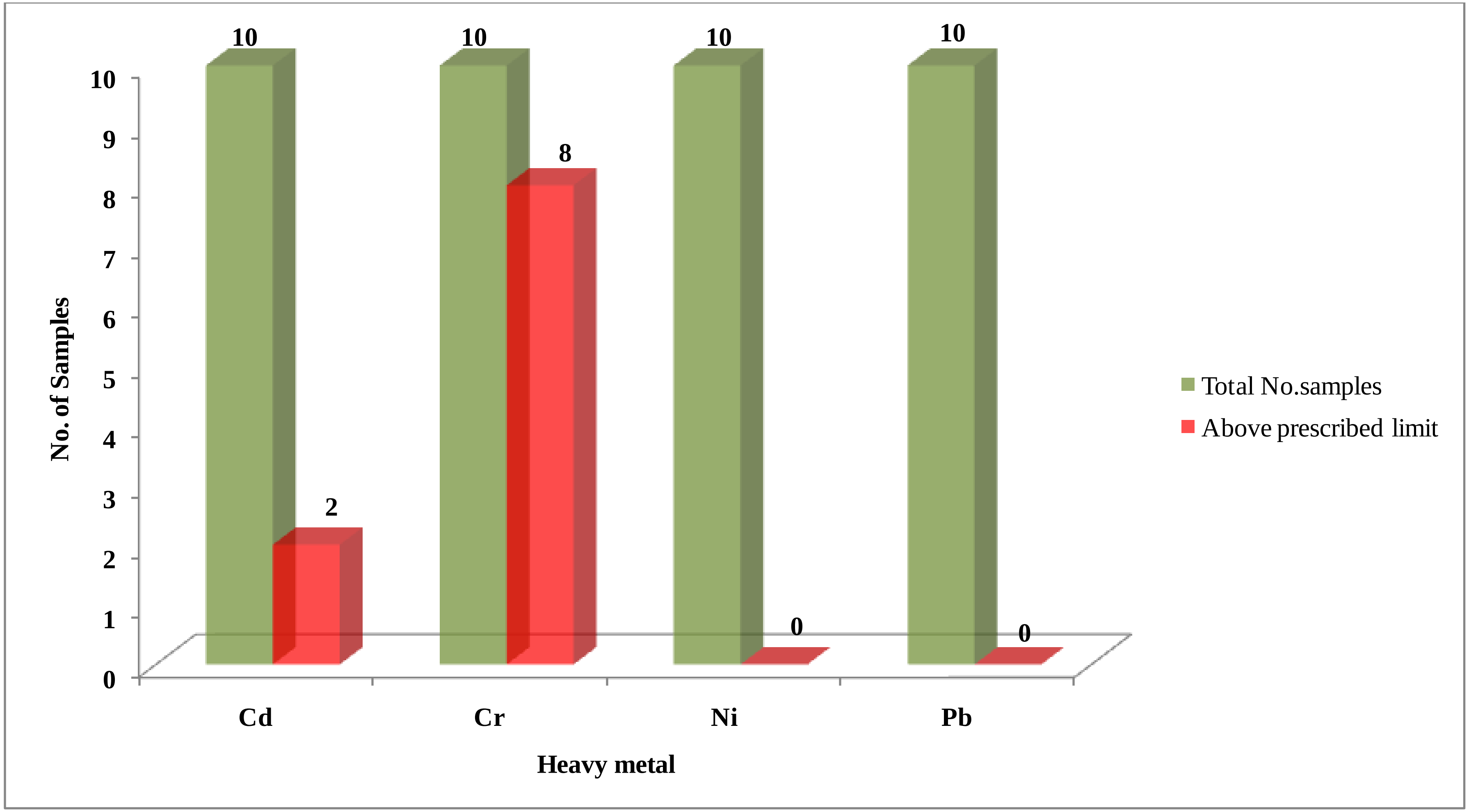


Fig: 8 Heavy metal status in surface water

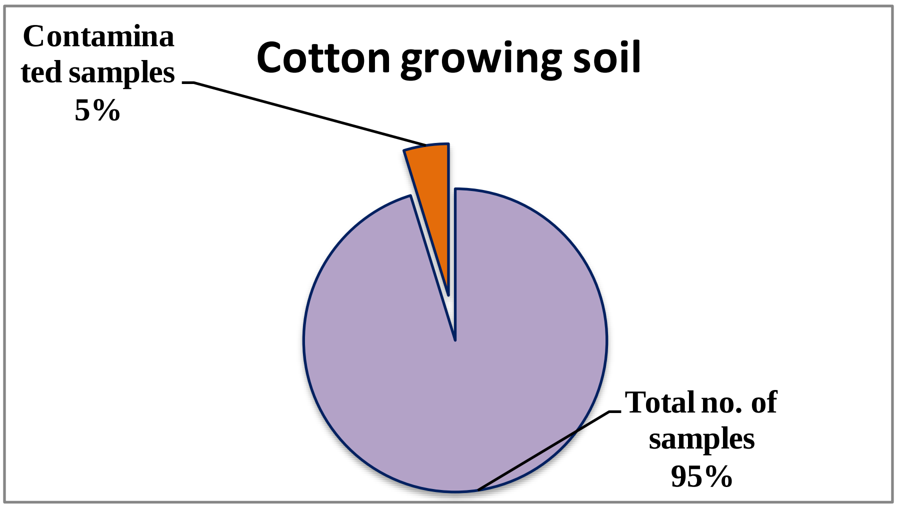


Fig:9 Pesticide contamination in cotton growing soil

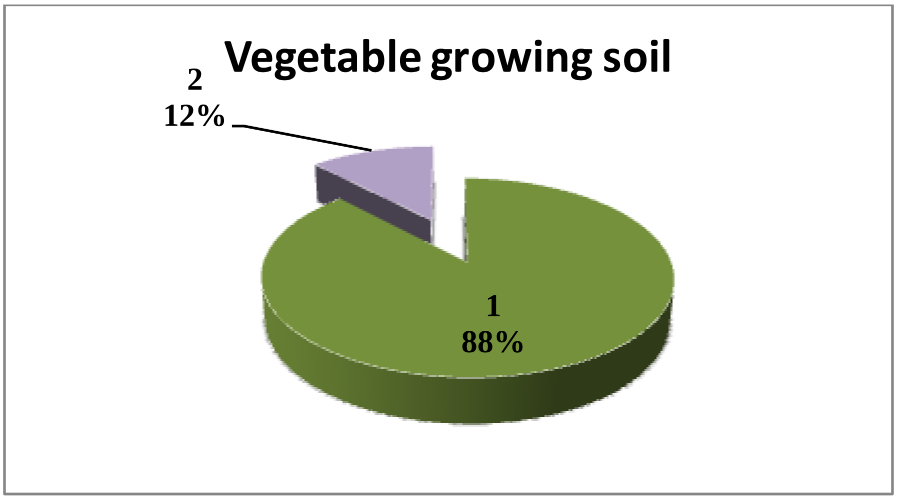


Fig:10 Pesticide contamination in vegetable growing soil

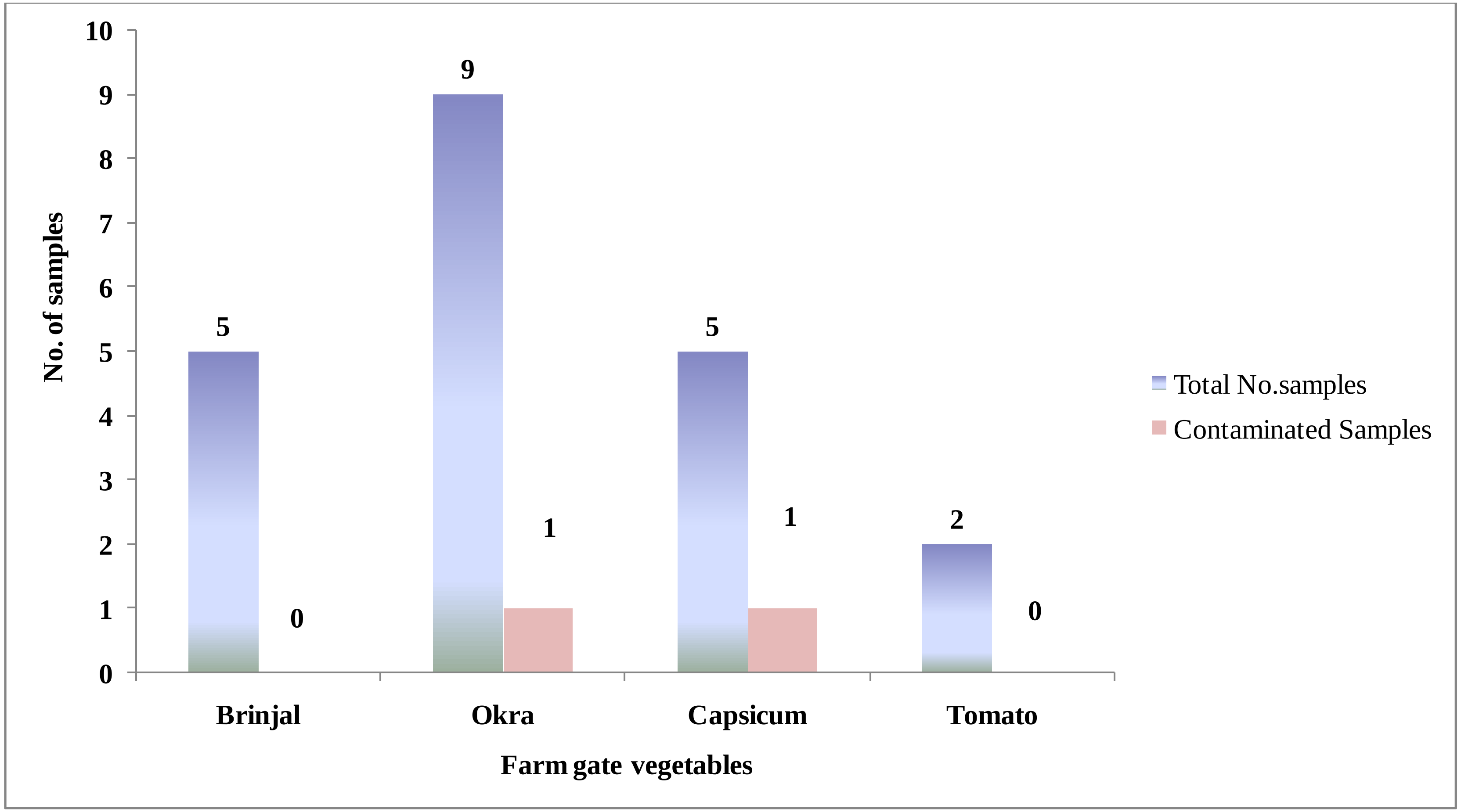


Fig: 13 Pesticide contamination in farm gate vegetables

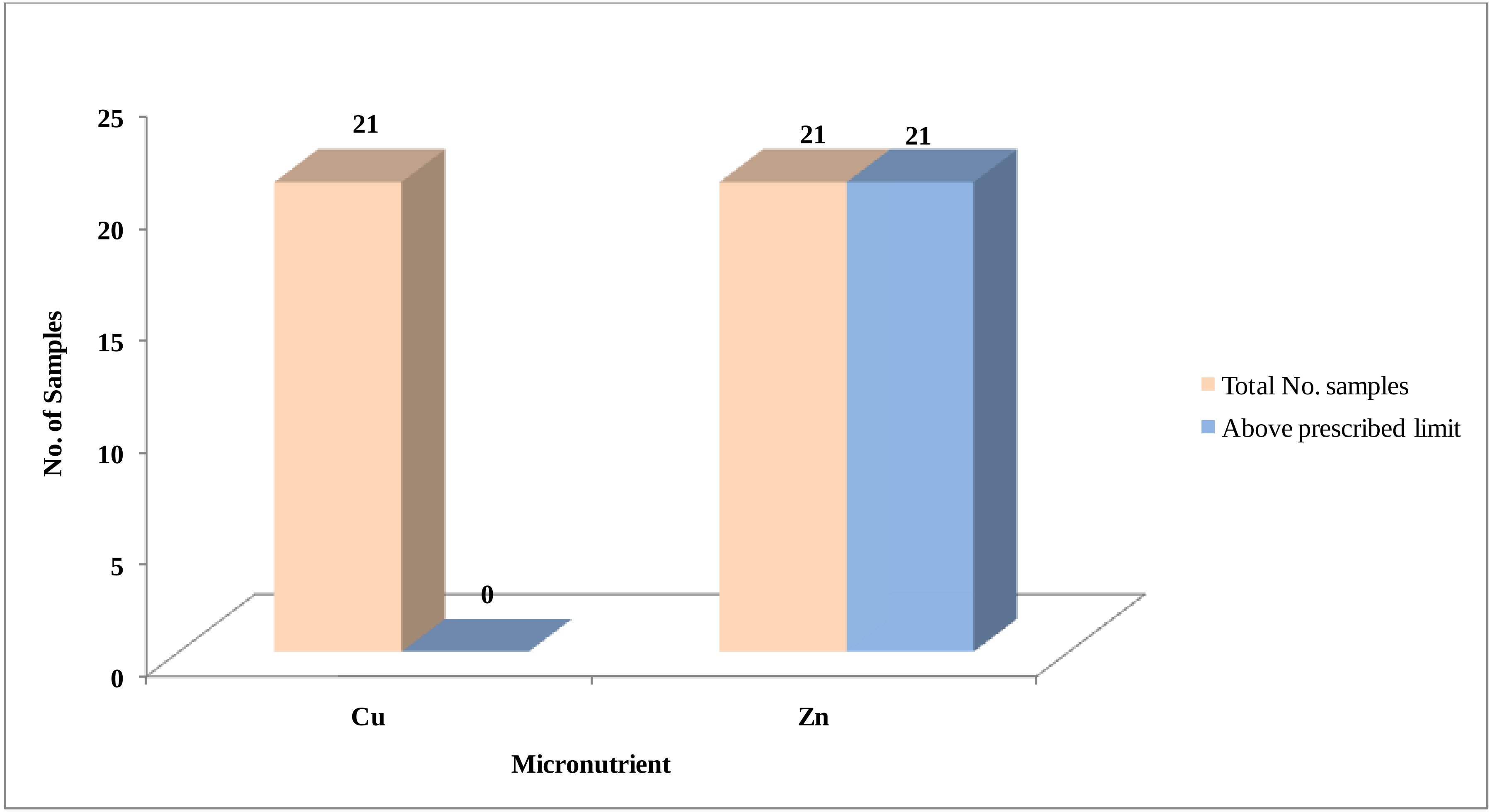


Fig: 14 Micronutrient in farm gate vegetables

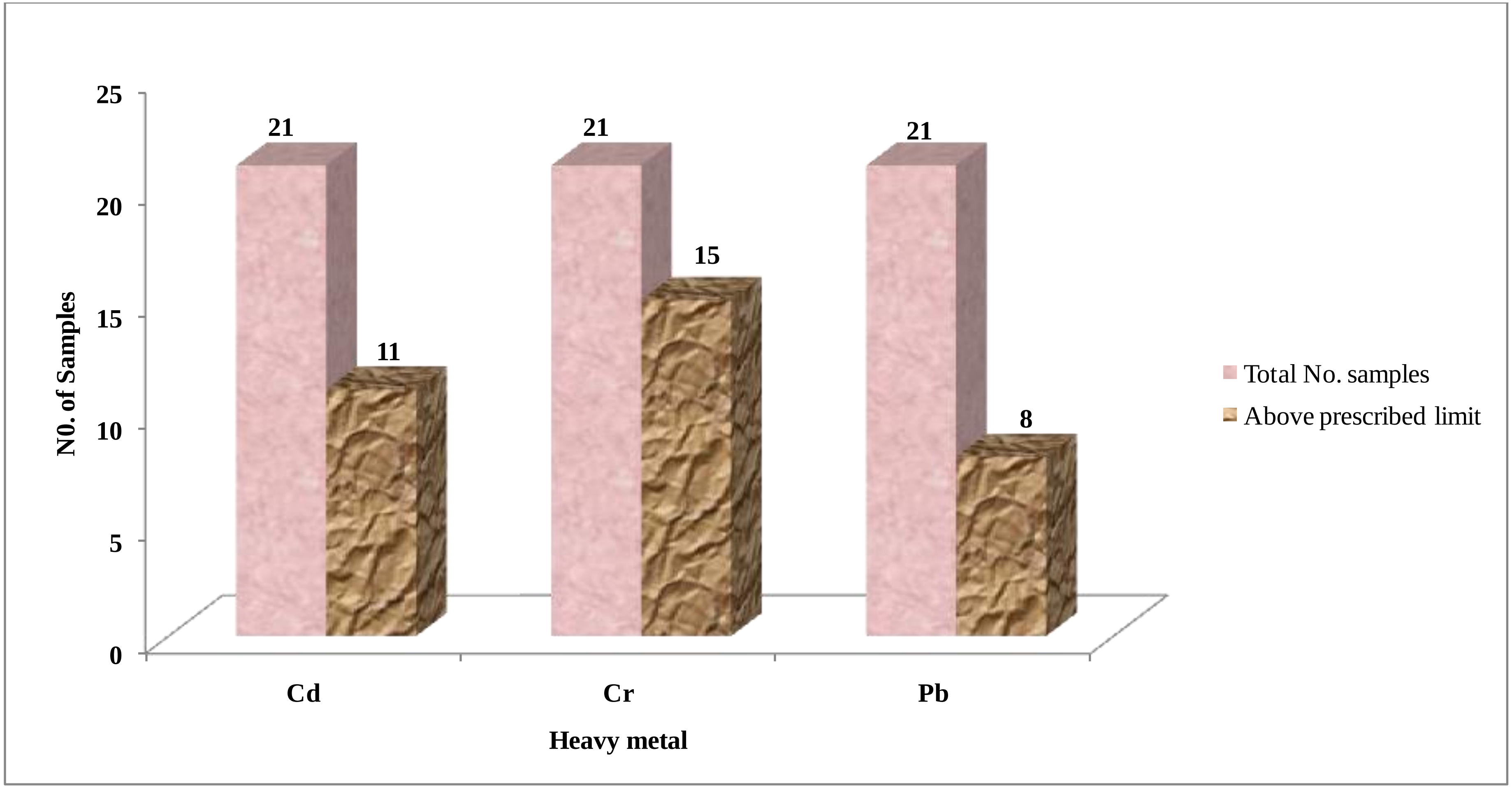


Fig: 15 Heavy metal in farm gate vegetable

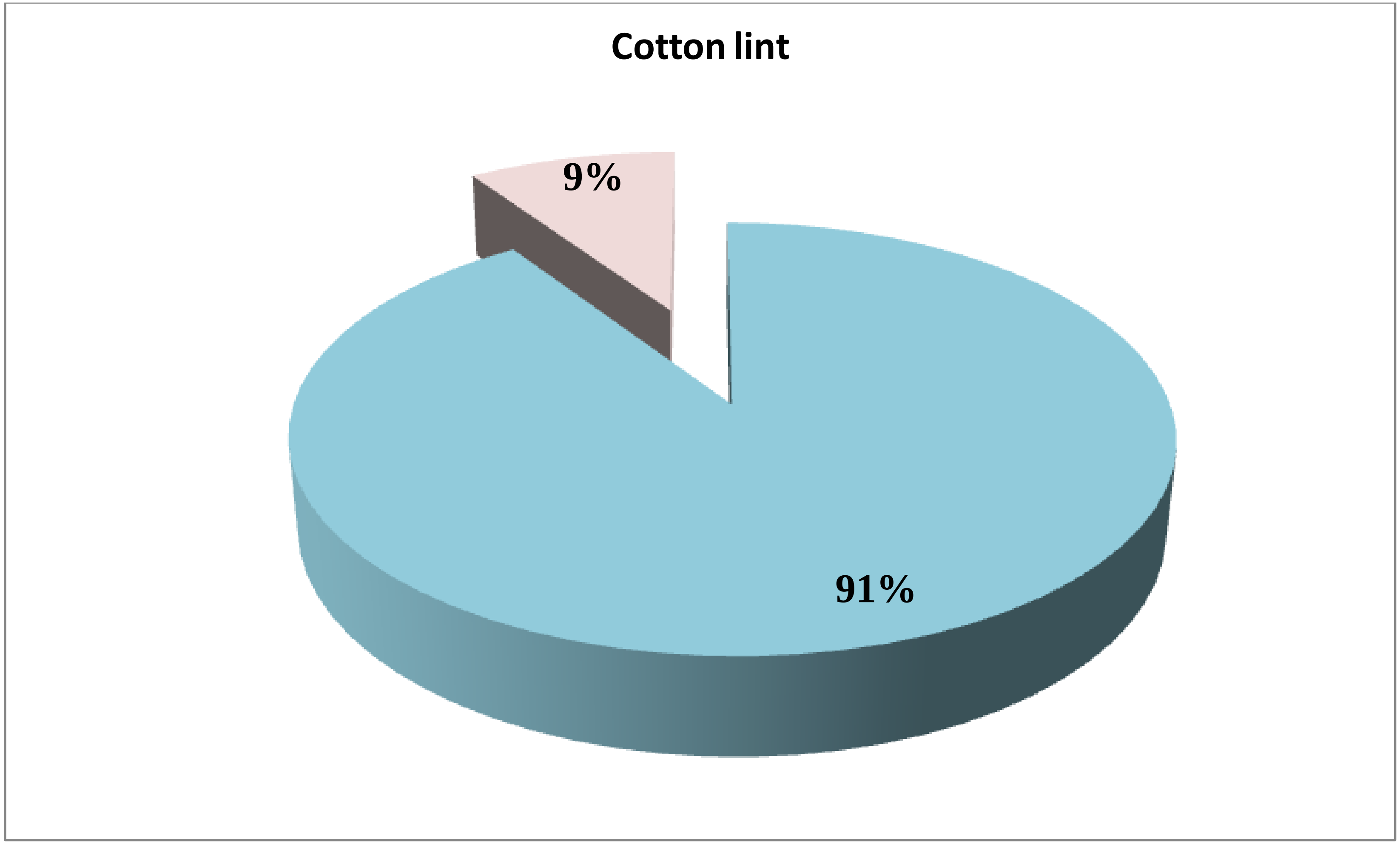


Fig: 16 Pesticide contamination in cotton lint (29 samples)

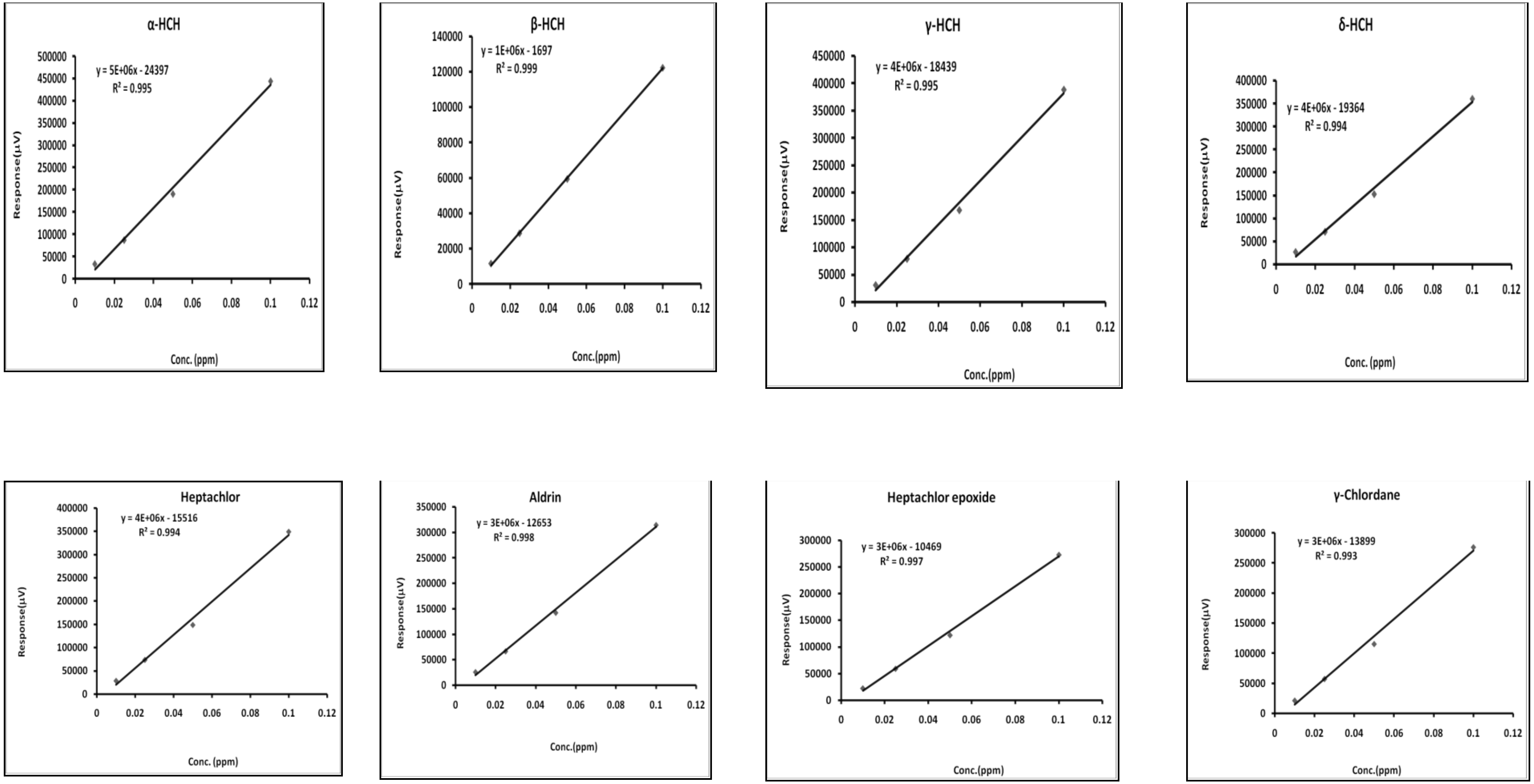


Fig : 1 Linearity graph : Organochlorines

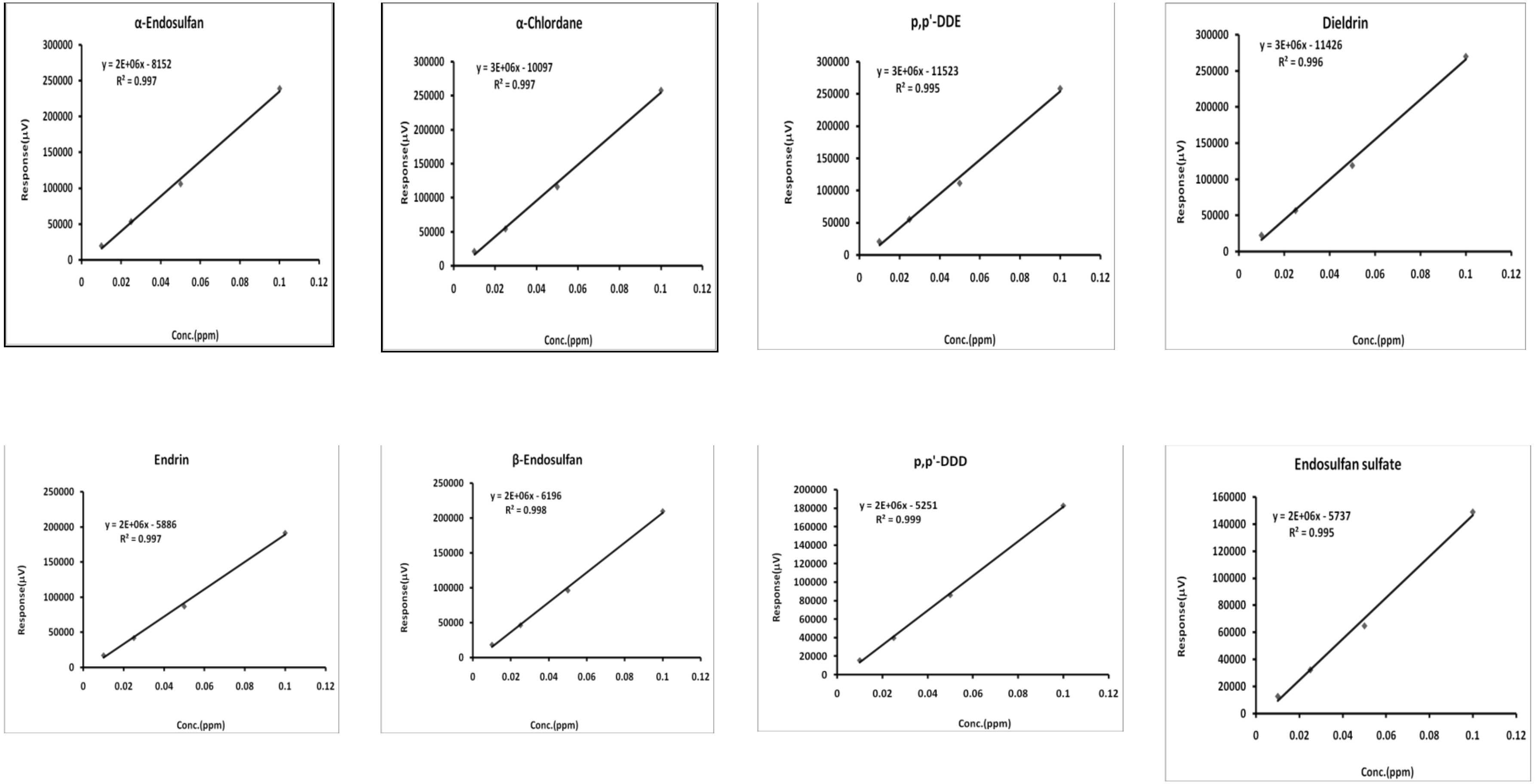


Fig : 1 Linearity graph : Organochlorines (cont.)

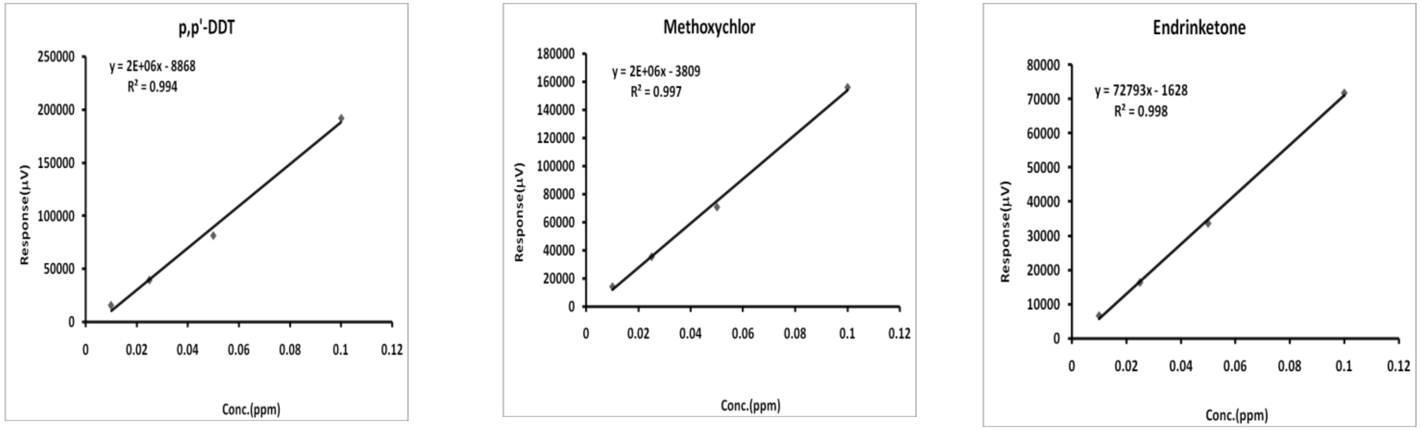


Fig : 1 Linearity graph : Organochlorines (cont.)

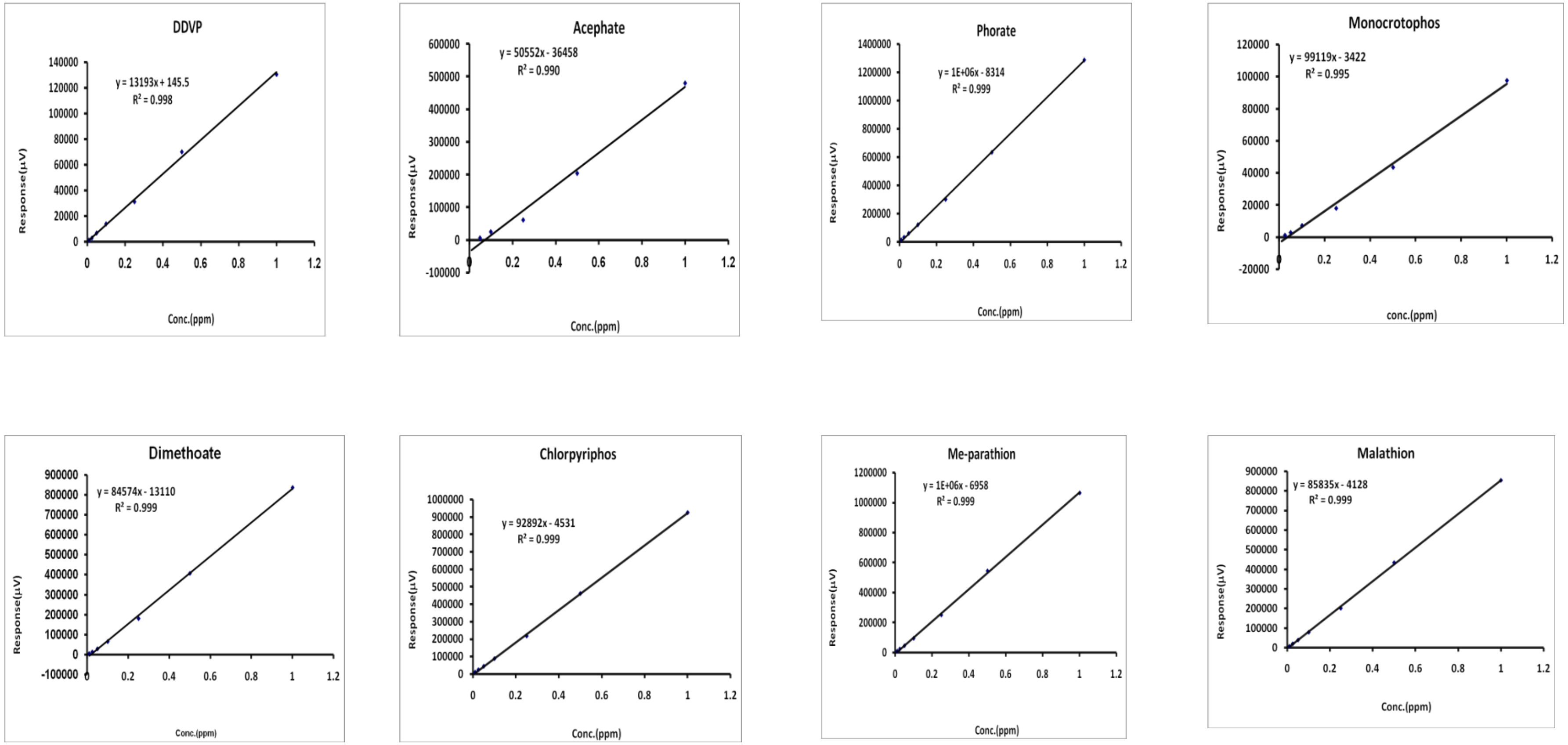


Fig:2 Linearity graph of Organo phosphates

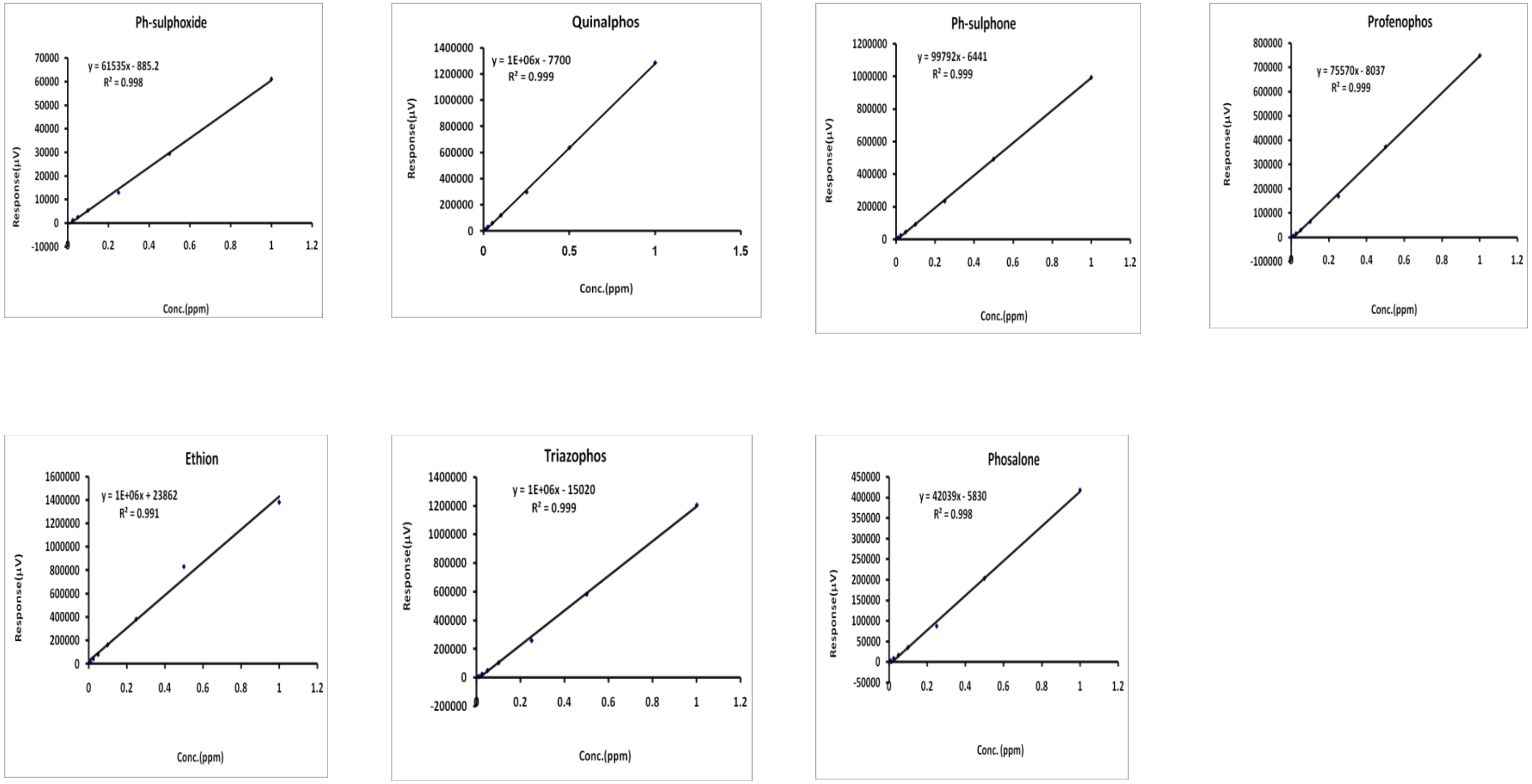


Fig:2 Linearity graph of Organo phosphates (cont.)

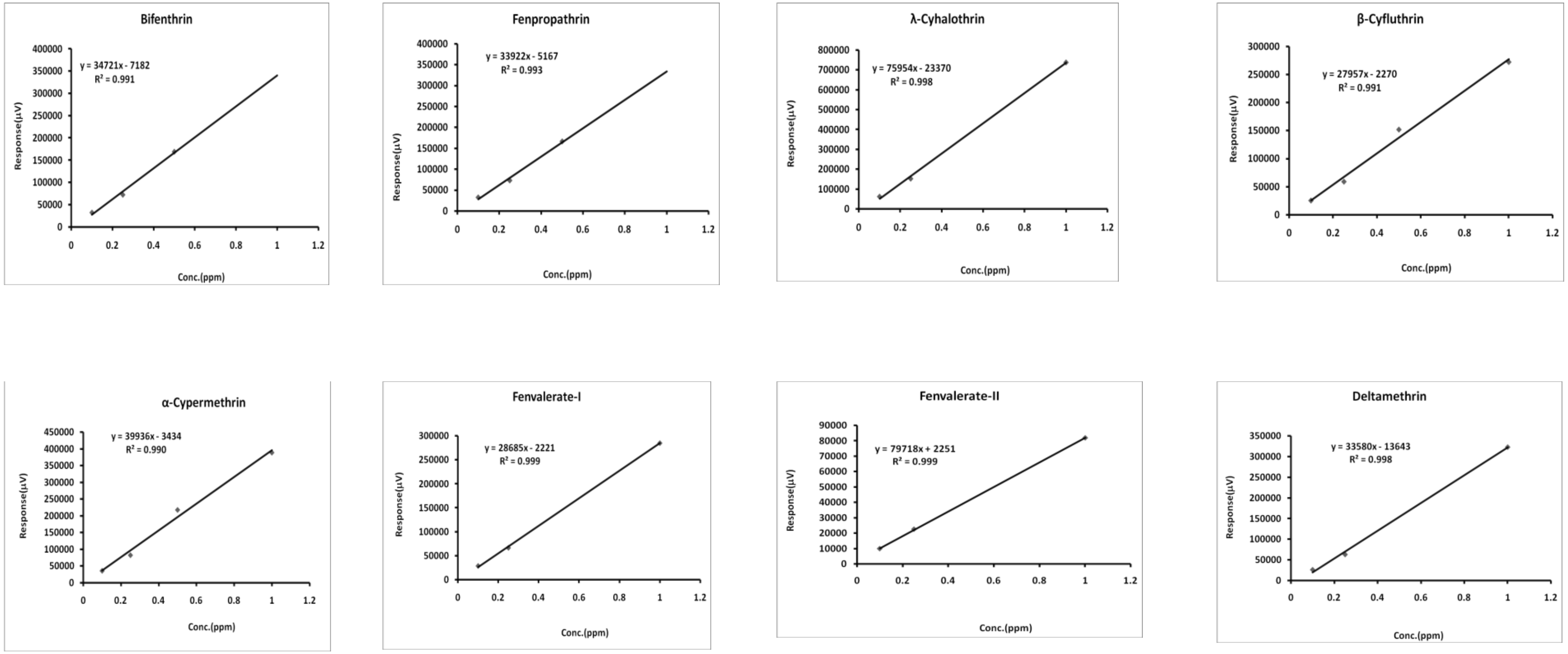


Fig:3 Linearity graph : Senthetic pyrthroids

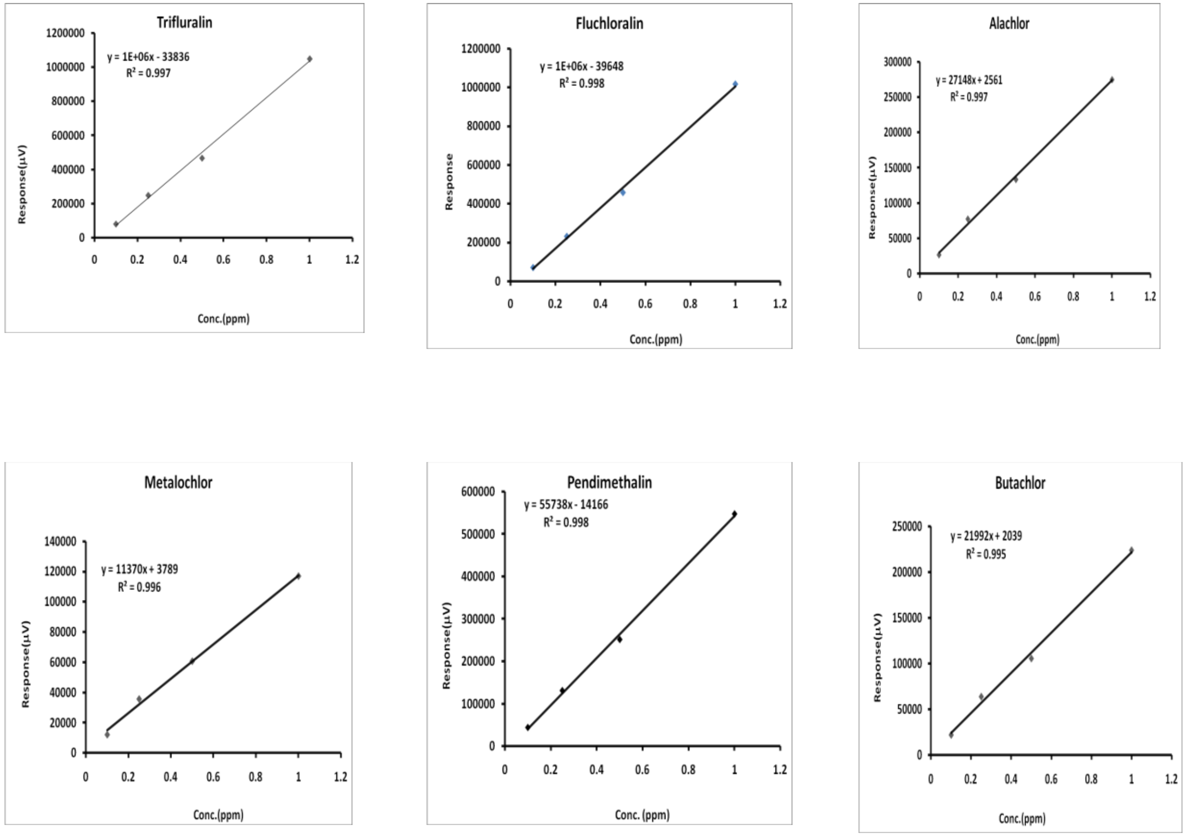


Fig:4 Linearity graph : Herbicides

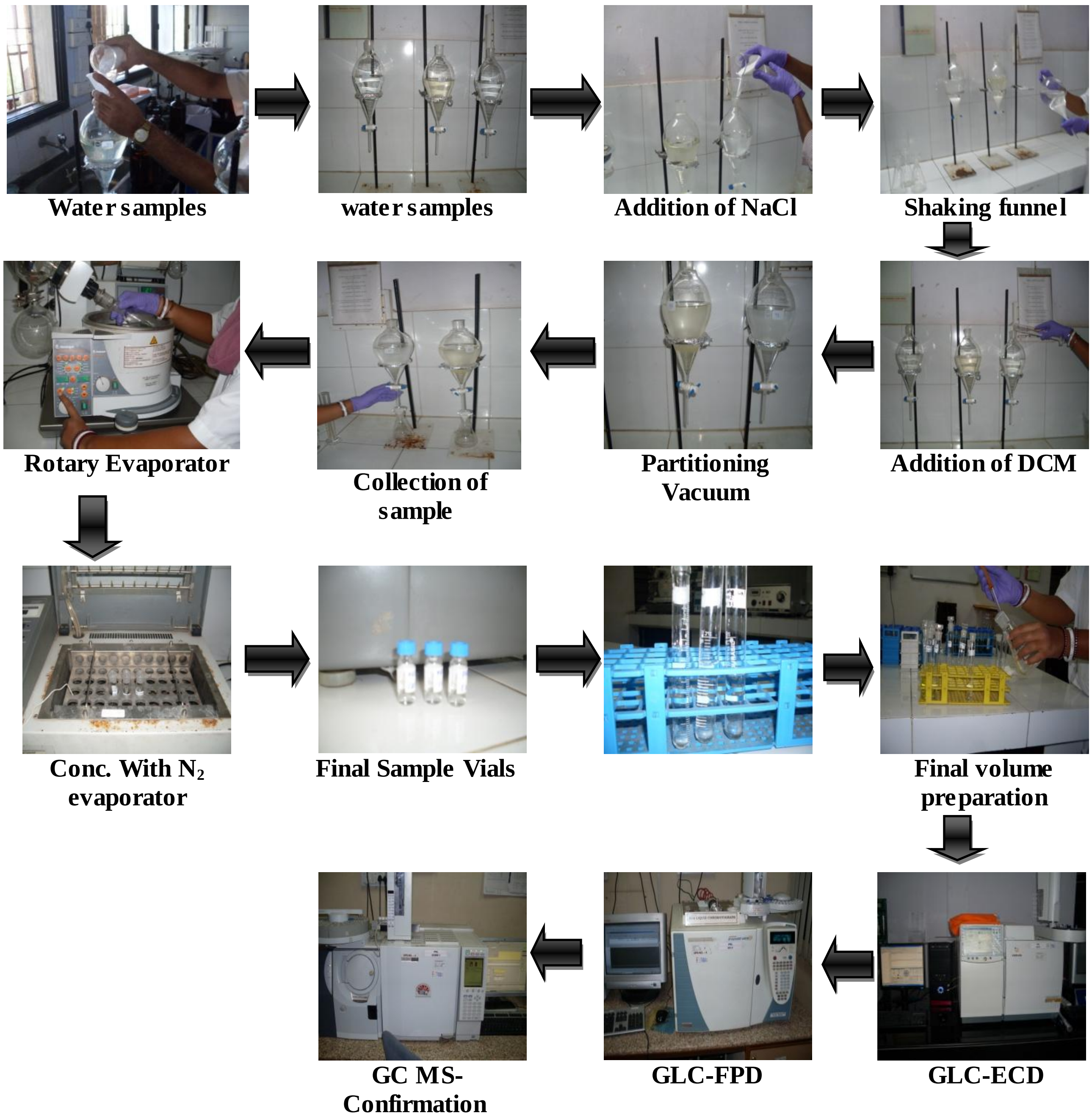


Plate : 1 Extraction of pesticide residues from water samples

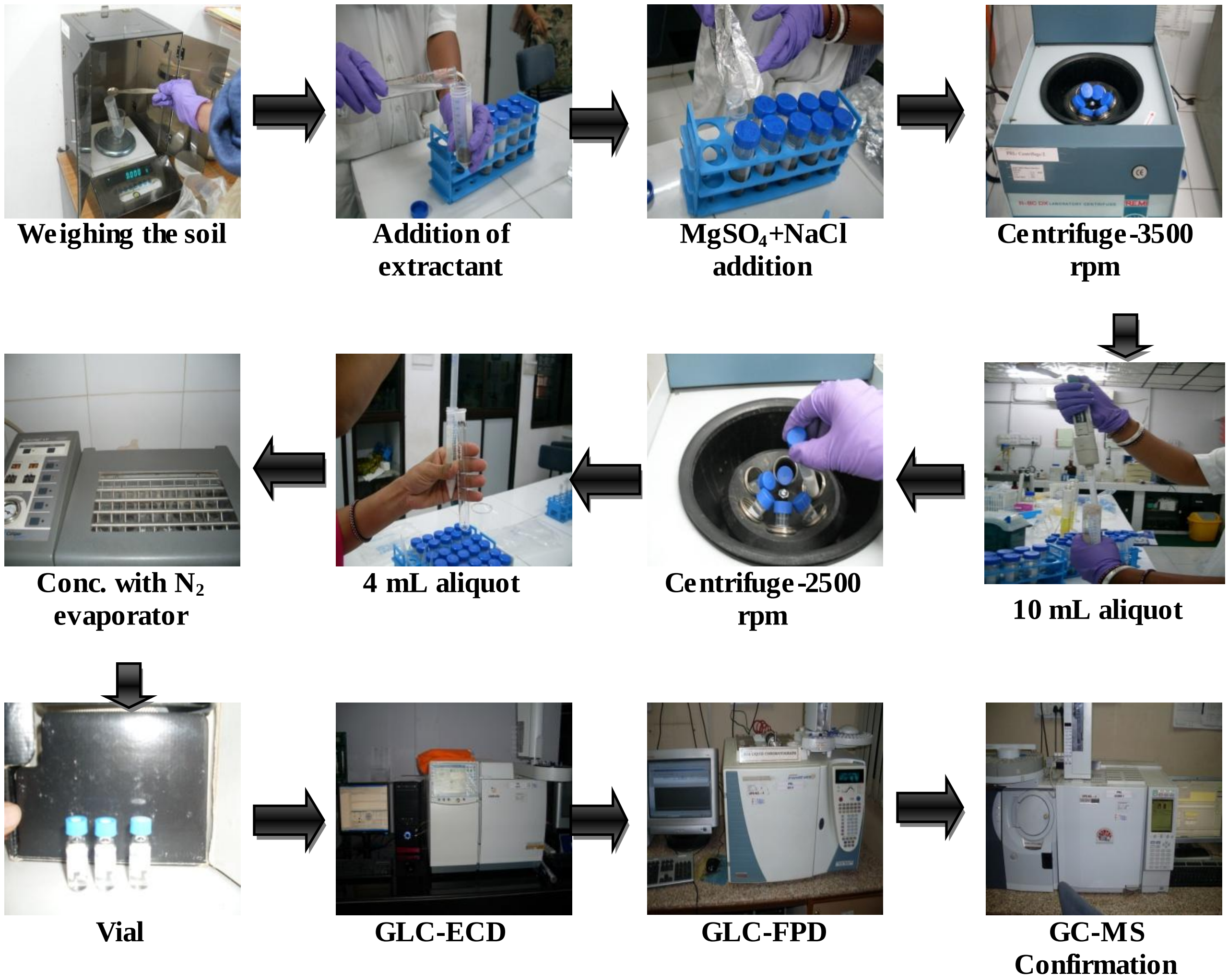


Plate : 2 Extraction of pesticide residues from soil samples

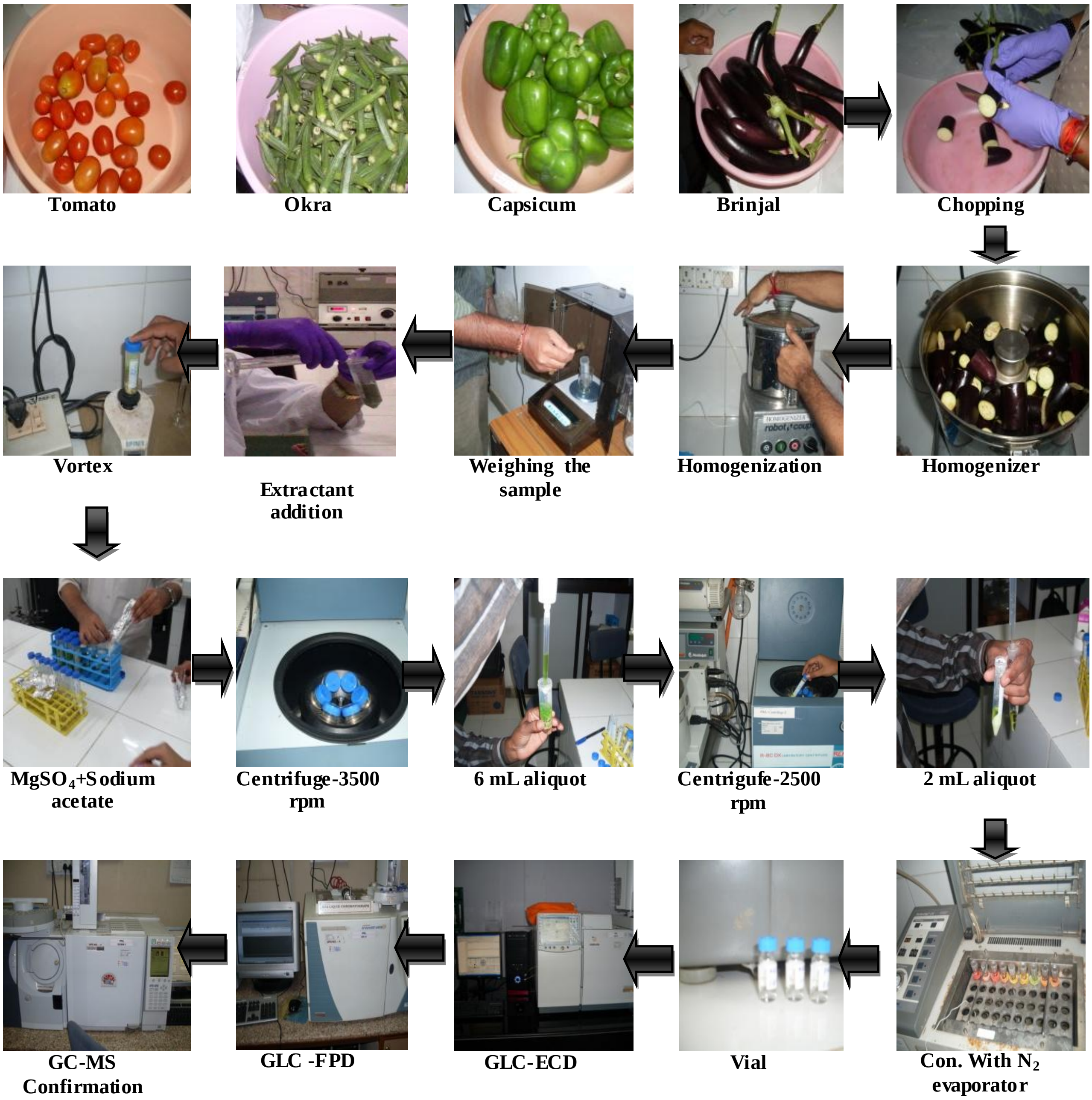


Plate : 3 Extraction of pesticide residues from vegetable samples