

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE NO.
1.1	Schematic diagram of focused (open) Microwave extractor	27
1.2	(A) Conventional Soxhlet extractor (B) Focused microwave-assisted Soxhlet extractor from Prolabo	35
2.1	Influence of varying carbonate strength from 0.001-5.0% (shown) on the recovery of PCP extracted from leather	66
2.2	The correlation of % MW power, duration of extraction and the recovery of PCP present originally in a solid sample (leather of 2g was used)	68
2.3	HPLC-UV chromatogram of a leather sample spiked (1 $\mu\text{g/g}$ of each of the species) with standard mixture of chlorophenols except PCP that is originally present (50 μl was injected; Detection at 280 nm): (1) 2-MCP, (2) 4-MCP, (3) 3- MCP (4) 2,6-DCP (5) 2,5-DCP, (6) 2,4-DCP, (7) 3,4-DCP, (8) 2,3,6 -TCP, (9) 3,5-DCP, (10) 2,4,6-TCP, (11) 2,3,4-TCP, (12) 2,4,5-TCP, (13) 2,3,5-TCP, (14) 3,4,5-TCP, (15) 2,3,5,6-TeCP, (16) 2,3,4,6-TeCP, (17) 2,3,4,5-TeCP, (18) PCP	77

FIGURE NO.	TITLE	PAGE NO.
2.4	GC-ECD chromatogram of a wood sample spiked (1 µg/g of each of the species) with standard mixture of chlorophenols except PCP that is originally present : (1) 2-MCP, (2) 3- MCP, (3) 4-MCP (4) 3,5-DCP, (5) 2,5-DCP, (6) 2,4-DCP, (7) 2,6-DCP (8) 3,4-DCP, (9) 2,4,6-TCP, (10) 2,3,5-TCP, (11) 2,4,5-TCP, (12) 2,3,6 -TCP, (13) 3,4,5-TCP, (14) 2,3,4-TCP, (15) 2,3,4,6-TeCP, (16) 2,3,5,6- TeCP, (17) 2,3,4,5-TeCP (18) PCP (19) Tetrachloroguaiacol. (Internal standard)	78
2.5	GC-ECD chromatogram of PCP extracted from a real (leather) sample using 0.01% carbonate and optimised MAE conditions, followed by ENVI-18 SPE enrichment of acetyl derivatised analyte	79
2.6	Electrophoregram of chlorophenols detected at 214 nm Peaks: (1) 2,3,4-TCP, (2) 2,3,4,6-TeCP, (3) 2,4,5-TCP, (4) 3,4,5-TCP, (5) 2,3,4,5-TeCP, (6) 2,4,6-TCP, (7) PCP, (8) 2,3,6-TCP, (9) 2,3,5,6-TeCP, (10) 2,3,5-TCP. Conditions: 100mM Ammonium Carbonate, 50mM β-CD, pH 8.5, voltage +15kV, current 143.6 µA.	82

FIGURE NO.	TITLE	PAGE NO.
3.8	The influence of variation of n-octane on the recovery of a volatile analyte, DCB and a semi volatile analyte, Aroclor 1248 in correlation to MWP. Conditions:- volume of aqueous phase: 25 ml; duration:3 min	106
3.9	The Influence of weight of sample is shown with Aroclor 1260 extraction from soil sample, weight varied from 1-5 g @ MWP 10%	106
3.10	The GC-ECD chromatograms of spiked Aroclor 1260 to a soil A. using 10 ml n-octane; B. using 1 ml n-octane, showing the enrichment. Sample extracted (under identical conditions except for n-octane)	111
3.11	The GC-ECD chromatograms of a real water sample found to contain γ -HCH (under identical conditions except for n-octane). A. using 10 ml n-octane; B. using 1 ml n-octane, showing enrichment	
3.12	GC-ECD chromatogram of a soil sample spiked with DCB, HCB and HCH extracted (trap) using 2 ml n-octane	112 113
3.13	GC-ECD chromatogram of spiked recovery Aroclor1248 from 100ml water using 1 ml n-octane	113

FIGURE NO.	TITLE	PAGE NO.
4.1	Dependency of recovery of 2-MCP from soil sample on MW heating duration	126
4.2	Dependency of PCP recovery from soil sample on MW heating duration	126
4.3	Dependency of recovery of 2,4,6 TCP in textile on MW heating duration	127
4.4	Dependency of recovery of PCP from leather on MW heating duration	127
4.5	Dependency of recovery of DCP on sample volume in water sample in correlation to MW heating duration (MWP 10%)	131
4.6	Dependency of recovery of PCP on sample volume in water sample in correlation to MW heating duration (MWP 10%)	131
4.7	Recovery data of MCPs from soil on varying MW heating	134
4.8	Recovery data of DCPs from soil on varying MW heating	134
4.9	Recovery data of TCPs from soil on varying MW heating	135
4.10	Recovery data of TeCPs and PCP from soil on varying MW heating	135
4.11	Recovery data of TeCPs and PCP from leather on varying MW heating	136

FIGURE NO.	TITLE	PAGE NO.
4.12	The influence of variation of aqueous phase volume on the recovery of spiked PCP using n-octane 1 ml and duration of 3 min	138
4.13	Influence of variation of solvent used for trapping in correlation to the variation of MWP from 10-40%. The Other conditions: 25 ml aqueous phase and duration of extraction is 3 min	140
4.14	Influence of weight of soil sample on Microwave extraction. Conditions:- 10-40% MWP was varied for a fixed duration of 8 min (as that is suitable for volatiles) 25 ml of aqueous phase was used with 1 ml n-octane as trap	141
4.15	Overlay of chromatograms of PCP extracted (as derivative) from a leather sample (native) A. extracted using 1ml n-octane; B. extracted using 5ml n-octane.	144
4.16	GC chromatogram of a textile sample extracted by insitu derivatisation method using 1 ml of n-octane. PCP originally present in the sample is detected. TCG shown is an internal standard added while HCB is added as an injection marker	145
5.1	Focused Microwave Assisted Steam distillation set-up with on-line SPE (solid) Enrichment arrangement	153
5.2	Recovery of derivatised MCP by MASD from solids	156

FIGURE NO.	TITLE	PAGE NO.
5.3	Recovery of derivatised DCP by MASD from solids	156
5.4	Recovery of derivatised TCP by MASD from solids	157
5.5	Recovery of derivatised TeCP by MASD from solids	157
5.6	Recovery of derivatised PCP by MASD from solids	158
5.7	Recovery of Aroclor 1248 by MASD from solids	158
5.8	Recovery of Aroclor 1260 by MASD from solids	159
5.9	Recovery of DCB by MASD from solids	159
5.10	Recovery of HCB by MASD from solids	160
5.11	Recovery of HCH by MASD from solids	160
5.12	Dwell time kept during MASD of various chloro organics	163
5.13	Dwell time kept during MASD of DCB	163
5.14	Dwell time kept for derivatised MASD for chlorophenols	164
5.15	Derivatisation duration for various chlorophenols studied using standard mixture, containing 2-MCP; 2,4-DCP; 2,4,6-TCP; 2,3,5,6-TeCP and PCP. MWP-20% standards added to 25 ml carbonate solution , 0.1% along with acetylating agents of 0.5 ml each of acetic anhydride and triethylamine	167
5.16	The dependency of recoveries on solid sample weight (taken a constant, maximum weight of 5 g of soil sample)	170

FIGURE NO.	TITLE	PAGE NO.
5.17	The dependency of recoveries on solid sample weight (taken a constant, maximum weight of 5 g of soil sample) dispersed in aqueous phase varied from 15-30 ml	171
5.18	Over lay of chromatograms obtained for (A) Standard mixture of Aroclor 1260 injected directly with out MASD (B) Recovery by MASD (50% MWP for 10 min with initial dwelling period of 8 min at 20% MWP) of the same concentration of A (200 ng) and recovered from soil	176
5.19	GC-ECD chromatogram of MASD recovery of chloro organic compounds (DCB, HCB and HCH). This is spiked recovery from a soil. The sample originally contains HCH level (208 ng/g) along with spiked level (50 ng/g). This has resulted from on-line SPE (ENVI-18) enrichment	177
5.20	To show the matrix cleaning by MASD, overlay of GC chromatograms for a leather sample found to have PCP originally were given (A) MASD insitu acetyl derivatisation (Conditions: 40% MWP for 10 min with dwell time 5 min at 20% MWP) using 1g leather sample and liquid trapped (2.5ml n-octane volume) (B) Soxhlet extracted using (hexane/acetone 1:1) for 6 hrs, derivatisation done externally, and volume is 2 ml n-octane	178