

CONTENTS

	PAGE
PREFACE	iii
I. INTRODUCTION	1
A. <i>BACKGROUND</i>	1
1. Federal Water Pollution Control Administration Water Pollution Surveillance System	1
2. Organic Pollution	1
B. <i>CARBON ADSORPTION SAMPLING</i>	3
C. <i>BOTTLED SAMPLES</i>	3
1. Water Samples	3
2. Bottom Samples	4
II. SAMPLE COLLECTION	5
A. <i>THE CARBON ADSORPTION METHOD (CAM)</i>	5
1. Preparation of the Carbon Adsorption Cartridge	5
2. Precautions Necessary to Prevent Accidental Contamination of Carbon	6
B. <i>DISCRETE BOTTLED SAMPLES</i>	6
1. Water Samples	6
2. Bottom Samples	6
3. Preparation of Container	7
III. PREPARATION OF SAMPLES PRELIMINARY TO GAS CHROMATOGRAPHIC ANALYSIS	8
A. <i>CARBON ADSORPTION SAMPLES</i>	8
1. Treatment of Carbon	8
a. Drying the Carbon	8
b. Extraction of the Carbon	8
2. Preliminary Separation of CCE	12
a. Procedure for General Organic Analysis	12
b. Column Chromatographic Separation of CCE (Alternate Procedure)	15
c. Thin Layer Chromatographic (TLC) Separation of Pesticides from Aromatic Fraction of CCE	17

	PAGE
B. <i>DISCRETE BOTTLED SAMPLES</i>	22
1. Extraction of Pesticides	22
a. Extraction from Water	22
b. Extraction of Bottom Samples ..	25
2. Concentration of Extract	25
a. Water Sample	25
b. Bottom Sample	25
3. Thin Layer Chromatography	27
IV. DETERMINATIVE STEPS	28
A. <i>GAS CHROMATOGRAPHY</i>	28
1. Application of Electron Capture Gas Chromatography	28
2. Application of Microcoulometric Titration Gas Chromatography	30
3. Calculations	30
4. Column Packings	31
5. Column Conditioning	32
B. <i>INFRARED SPECTROPHOTOMETRY</i>	32
V. CONTROL OF INTERFERENCES	33
A. <i>SOLVENT INTERFERENCES</i>	33
1. Chloroform	33
2. Hexane-Benzene	33
3. Hexane-Acetone	34
4. Carbon Tetrachloride and Acetone	34
B. <i>CARBON INTERFERENCES</i>	34
1. Carbon Blank	34
C. <i>OTHER SOURCES OF INTERFERENCE</i>	34
D. <i>INTERPRETATION</i>	35
VI. SENSITIVITY AND SPECIFICITY	36
A. <i>SENSITIVITY</i>	36
1. Carbon Adsorption Extracts Examined by Electron Capture Gas Chromatography	36
2. Carbon Adsorption Extracts Examined by Micro- coulometric Titration Gas Chromatography	37
3. Bottled Sample Extracts Examined by Electron Capture Gas Chromatography	37
4. Bottled Sample Extracts Examined by Microcoulometric Titration Gas Chromatography	37

	PAGE
B. <i>SPECIFICITY</i>	37
1. Carbon Adsorption Samples	37
2. Bottled Samples	37
APPENDIX ONE	38
Engineering Aspects of Sampling by the Carbon Adsorption Method	
APPENDIX TWO	51
Chromatograms, Sample Calibration Curves, Infrared Spectra, and Structural Formulae	
APPENDIX THREE	61
Equipment, Solvents and Reagents	
APPENDIX FOUR	64
General Composition of Carbon Chloroform and Carbon Alcohol Extracts	
APPENDIX FIVE ..	67
Glossary	
REFERENCES	67

LIST OF TABLES

	PAGE
TABLE 1	58
R_f Values of Pesticides Developed with CCl_4 on Silica Gel-G Thin Layer Plate	
TABLE 2	59
Gas Chromatographic Retention Data	
TABLE 3	60
Some Column Packings Used for Gas Chromatographic Analysis of Chlorinated Hydrocarbon Pesticides	