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ENVIRONMENTAL ANALYSES UTILIZING GAS AND LIQUID CHROMATOGRAPHY

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ABSTRACT

An overview of the various methods of analysis, in environmental studies, which utilize gas chromatography(GC), liquid chromatography(HPLC), and ion chromatography(IC) is presented. In many cases the samples must be prepared for final measurement by employing other analytical techniques; eg., extraction(liquid-liquid, both micro and macro scale; solid-phase(SPE); supercritical fluid(SFE); headspace equilibration(HSGE); etc.). Details of the various methods will not be given but descriptive explanations and pertinent references will be furnished. Theoretical aspects of the various techniques will be referenced adequately for those not familiar with the methodologies. Since most of these methods are validated by the United States Environmental Protection Agency (USEPA) addresses where the methods may be obtained will also be given.

INTRODUCTION

The environment is easily categorized into discrete areas; air, drinking water, wastewater, solid waste, and soil. When discussing the environment we

could be referring to the great out-of-doors or a confined area such as our home or workplace.

Pollution studies of air and water samples have been greatly enhanced by the use of chromatographic techniques. The selection of the proper detectors coupled with the various types of columns and substrates have broadened the capabilities of environmental analyses. Proper care in sampling protocols and sample treatment has steadily improved the sensitivity of these measurements. The recommended methods for air, drinking water, wastewater and solid waste samples all utilize some type of chromatographic analysis for the determination of organic contaminants. Three complimentary techniques: i.e., headspace sampling(dynamic and static methods), microextractions (liquid-liquid and/or liquid-solid) and solid-phase extractions have expanded the capabilities of such studies to permit the analytical chemist to perform fast in-the-field monitoring, concentrate low levels of analytes and determine solubilities of toxic substances.

Why should any of us be concerned about the environment? In the past we allowed Mother Nature to keep it in balance. This was fine because the unbalance did not become too one-sided; i.e., in regards to contamination and pollution. Pollution refers to the state in which substances are present in above-normal natural amounts as a result of human activity. Contamination of the environment refers to the state in which substances are present which are not normally found in the environment.

By employing analytical chemical techniques we can understand and even minimize our ecological problems. However, environmental chemistry deals with

natural systems and matrix effects have a very profound effect on all resulting types of analyses.

To put the situation in perspective, let us consider what is occurring in the world today. Crude oil production is increasing and should reach a plateau, adequate food supplies will level off or even decline, food production must show a significant increase, and there will be an increasing demand for drinking water.

This is a very exciting time for analytical chemists; we have the chance to develop or increase the sensitivity of analytical techniques. Areas of analytical chemistry which are attracting much attention are the techniques of separation. For we "older" analytical chemists this certainly is a time to reflect. Just think of the time we are saving with our present-day separation techniques. If we had all these techniques available forty years ago, we would now routinely be determining parts-per-trillion of analytes in complicated matrix samples. Let me assure you, the new generation of analytical chemists will be doing this exact type of measurement. The quality and choice of analyses we perform are much more important than the number of analyses completed. It is very probable that too many analyses are performed; because fewer, more carefully planned analyses will yield the same or more useful information.

In this paper we will look at the chromatographic techniques used for environmental chemistry. This will be an overview-but adequate references will be furnished.

One may speculate why chromatography became such an important analytical technique for environmental studies. If one considers the number of organic compounds which could be in the environment, either as a pollutant or contaminant, it becomes very confusing.

TABLE 1

REPRESENTATIVE SAMPLING DATA OF EARLY WORK OF BRAUS,
MIDDLETON AND WALTON(1)

SAMPLING TUBE: IRON PIPE, 3.0 FEET IN LENGTH

ADSORBENT: 1200-1500 g GRANULAR ACTIVATED CARBON

SAMPLING RATE: 0.1-0.6 GALLONS/MINUTE

SAMPLE VOLUME: 5000-75000 GALLONS WATER

CARBON ADSORBENT EXTRACTANT: DIETHYL ETHER

A MODIFIED SHRINER & FUSON SEPARATION SCHEME WAS EMPLOYED TO SEPARATE THE ANALYTES INTO AMPHOTERIC AND WATER-SOLUBLE GROUPS; PHENOLIC, ACIDIC, BASIC AND NEUTRAL COMPOUNDS.

Also, one must take into account the fact that many of these compounds are homologues, isomers, and/or possess similar functional groups. If chromatography (gas and liquid) had not become developed and so popular in the early 1950's we would be attempting to solve these problems by classical methods; i.e., collecting very large volumes of water or air, adsorbing the organics on charcoal, dissolving them from the adsorbent, concentrating the resulting solution, and then carrying out some classical qualitative organic analysis scheme and attempting to quantify the analytes. Needless to say this would have been very time consuming. Happily we were spared this ordeal; however, this does not mean it was not done. Braus et al¹ published work of this type in 1951. Their average sampling time was four months and the average water sample volume was 27,856 gallons. Table 1 lists some representative data from their work.

METHODS AND DISCUSSION

Legal limits are placed on the amount of a contaminant or pollutant that may be present in an environmental sample. These limits are referred to as environmental standards or threshold values. They are usually expressed in some concentration unit for a period of time(hours,days or months), for a given volume(for air or water), or for a given weight of sample(for soils and waste). Various criteria are used to decide these values;eg., economics, social aspects, technical information available, health standards, toxicity of the analyte, and political information. Thus, these standards are not always decided from well-founded scientific facts. Probability factors are incorporated in the final decision. Some environmentalists would like to see a "Zero Level" for these various analytes. This philosophy is difficult to implement because of the lack of analytical methodologies and/or instrumentation, cost would be prohibitive, uncertainty of the effect of one analyte alone(it could be that analyte in combination with others cause the problem), and availability of laboratory personnel. One additional thought; if we had a routine analytical method capable of quantifying one attogram (10^{-18} g) of lead, there would be present 3,010 atoms of lead!!

In this discussion we will concern ourselves only with gas and liquid chromatography. One should remember that these two techniques are only two, but two very important aspects of environmental analytical chemistry.

Either of these two chromatographic techniques must be coupled with other sampling,concentration and detection techniques so as to furnish reliable analytical data. Tables 2A,B,&C list many of these

TABLE 2A

TECHNIQUES FOR REMOVAL OF INTERFERING SUBSTANCES

1. CHANGE IN TEMPERATURE OF SAMPLE SYSTEM.
 2. ASHING OF RESIDUES.
 3. ION EXCHANGE TO REMOVE IONIC SUBSTANCES.
 4. DISTILLATION TO REMOVE LOW BOILING COMPONENTS.
 5. COMPLEXING AGENTS TO MASK INORGANIC COMPONENTS.
 6. CHANGE IN pH OF SAMPLE SYSTEM.
 7. REACTION KINETICS TO ALTER AMOUNT OF INTERFERENCE.
 8. USE OF VARIOUS CHROMATOGRAPHIC TECHNIQUES.
-

TABLE 2B

SAMPLE PREPARATION TECHNIQUES

LIQUID-LIQUID EXTRACTION

MICRO

MACRO

GAS-LIQUID/GAS-SOLID EXTRACTIONS

HEADSPACE

STATIC SYSTEMS

DYNAMIC SYSTEMS

SOLID-PHASE EXTRACTION(SPE)

PACKED CARTRIDGES

TEFLON DISKS(EMPORER)

SUPERCRITICAL-FLUID EXTRACTION(SFE)

CO₂(PURE OR MODIFIED)NO₂(PURE OR MODIFIED)

TABLE 2C

ANCILLARY DETECTOR TECHNIQUES USED WITH GAS AND LIQUID CHROMATOGRAPHY

SELECTIVE DETECTORSMASS SPECTROMETRY

ATOMIC EMISSION
 ELECTRON CAPTURE
 ELECTROCHEMICAL
 FLAME IONIZATION
 FLUORESCENCE
 DIODE ARRAY
 ULTRAVIOLET

GC/MS
 LC/MS

ancillary techniques which are combined with chromatographic separations. These ancillary techniques aid in the selectivity and sensitivity of the methods. The majority of the methods in environmental organic analyses use gas chromatographic systems. This can be explained by the fact that in the beginning the greater number of environmental laboratories had gas chromatographs and trained personnel but not liquid chromatographs(HPLC was just being perfected) or mass spectrometers.

Gas Chromatographic Techniques

Gas chromatography has been one of the most extensively used separation techniques for organic environmental analyses. There are numerous gas chromatographic methods for both drinking water and municipal/industrial discharge water. Additionally, methods are also available for solid-waste samples.

In this paper we will discuss predominately the methods published by the United States Environmental Protection Agency(USEPA). There are numerous methods available for the various analytes we will cover. Some of these are published by individuals in the chemical literature and others appear in the compilation of methods published by the American Public Health Association(APHA), the American Water Works Association(AWWA) and the Water Pollution Control Federation(WPCF)². The methods appearing in this latter publication are acceptable by the USEPA and state environmental agencies(because they have been validated by these organizations and/or the USEPA). One should keep in mind that there are many procedures which have appeared in the chemical literature which are just as reliable(and in many cases require less time) but are not acceptable by the USEPA simply

because they(USEPA) have not validated them. Good examples of this are field and laboratory methods utilizing Headspace Gas Chromatography and Micro-Extractions.

The reader who wishes to obtain copies of the USEPA Methods may purchase them from one of the sources given below.

1. 500 Methods(Drinking Water Samples)
National Technical Information Service
5285 Port Royal Road
Springfield,VA 22161
1-703-487-4600
Document Numbers:
PB89-220461(Volume 1)
PB90-215039(Volume 2)
2. 600 Methods(Municipal & Industrial Discharge
Water)
Environmental Monitoring & Support Laboratory
USEPA
Cincinnati, OH 45268
1-513-569-7562
3. 8000 Methods(Municipal & Industrial Soils &
Waste)
U.S. Government Printing Office
Washington,DC 20402
1-202-783-3238
Document Number: 955-001-000001
4. TO(Total Organics)Methods for Ambient Air
National Technical Information Service
5285 Port Royal Road
Springfield,VA 22161
1-703-487-4600
Document Number: PB90-127374

One may find background information on the theoretical aspects of gas chromatography³, liquid chromatography⁴ and ion chromatography^{4,5} in several good publications. Applications of these techniques may be found in other excellent publications^{6,7}.

Tables 3,4,6 and 7 summarize the methodologies for the determination of organic analytes in drinking wa-

TABLE 3

DRINKING WATER TEST METHODS(USEPA)

<u>METHOD</u>	<u>ANALYTES</u>
501.3	Trihalomethanes. GC/MS with Selected Ion Monitoring.
502.1	Volatile Halogenated Organic Cpds. by P&T, GC/Halide Specific Detector.
502.2	Volatile Halogenated Organic Cpds. by P&T, Capillary GC/Halide Specific Detector & PID.
503.1	Volatile Aromatics and Unsaturated Organic Cpds. by P&T, GC/PID.
504	1,2-Dibromoethane(EDB) and 1,2-Dibromo-3-Chloropropane(DBCP) by Microextraction and GC/ECD.
505	Organohalide Pesticides and Aroclors by Microextraction and GC/ECD.
507	Nitrogen & Phosphorus Containing Pesticides, GC/NPD.
508	Chlorinated Pesticides. GC/ECD.
508A	Screening for PCBs by Perchlorination, Extn. and GC/ECD.
513	2,3,7,8-TCDD. Liquid/Liquid Extn. GC/ECD.
515	Chlorinated Herbicides. Liquid/liquid Extn. GC/ECD.
515.1	Chlorinated Acids. Derivatization to Esters. Capillary GC/ECD. Dalapon, Picloram, Cl ₅ Phenol.
515.2	
524.1	Volatile Organic Cpds. by P&T. Packed & Capillary Column GC/MS.
524.2	
525	Organic Cpds. SPE, Capillary Column GC/MS.
531	Carbamate Pesticides. Direct Aqueous Injection by Reversed-Phase HPLC. Post-Column Derivatization & Fluorescence(230nm Excitation, 419nm Emission)
531.1	
547	Glyphosate. Direct Aq. Injection, HPLC & Fluor.
548	Endothall. Derivatization, Extn. and GC/ECD.
549	Diquat. Extn. with HPLC & UV Detection.
550	PAHs. GC & HPLC. FID & UV or Fluorescence.
550.1	
551	Halogenated Volatiles from Chlorination. GC/ECD, FSOT Column.
552	Haloacetic Acids from Chlorination. GC/ECD, FSOT Column.

TABLE 4
EFFLUENT DISCHARGE TEST METHODS(USEPA)

<u>METHOD</u>	<u>ANALYTES</u>
601	Purgeable Halocarbons; P&T, GC/Halide Specific Detector(Hall).
602	Purgeable Aromatics; P&T, GC/PID.
603	Acrolein & Acrylonitrile; GC/PID, P&T.
604	Phenols; Extraction and GC/FID.
604.1	Hexachlorophene & Dichlorophen. HPLC, 254nm.
605	Benzidines; HPLC and Electrochemical Detector
606	Phthalates Esters; Extraction and GC/ECD.
607	Nitrosamines; Extr ction and GC/NPD.
608	Organochlorine Pesticides & PCBs; Extraction and GC/ECD.
609	Nitroaromatics and Isophorone; Extraction and GC/ECD, FID.
610	PAHs; HPLC with UV(254nm and Fluorescence Detection; 280nm Excitation, 389nm Emission) Varian uses Diode Array Detection. Also Extraction and GC/FID.
611	Haloethers; Extraction and GC/Hall Detector
612	Chlorinated Hydrocarbons; Extraction & GC/ECD.
613	2,3,7,8-Tetrachlorodibenzo-p-Dioxin Extn. & 2,3,7,8-Tetrachlorodibenzofuran GC/MS.
615	Chlorinated Herbicides; GC/ECD.
619	Triazine Pesticides; GC/NPD.
624	Purgeables; P&T and GC/MS.
625	Base/Neutrals & Acids; Extn. and GC/MS
631	Benomyls & Carbendazim; HPLC at 254nm.
632	Carbamate & Urea Pesticides; HPLC at Variable UV Wavelength.
632.1	Same Analytes; HPLC at 640-644nm.
635	Rotenone; HPLC at 254nm.
636	Bensulide; HPLC at 270nm.
637	MBTS & TCMTB; HPLC & UV detection.
638	Oryzalin; HPLC at 254nm.
639	Bendiocarb; HPLC at 254nm.
640	Mercaptobenzothiazole; HPLC + UV Detection.
641	Thiabendazole; HPLC with Fluorescence: 300nm Ex. 360nm Em.
642	Biphenyl & Orthophenylphenol; HPLC at 254nm.
643	Bentazon(Basagran); HPLC at 340nm.
644	Picloram; HPLC at 225nm.
680	Pesticides & PCBs in Water & Soil/Sediments by GC/MS.
1624	Volatile Organic Cpds.; Isotopic Dilution GC/MS
1625	Semivolatile Organic Cpds; Isotopic Dil. GC/MS.

ter, effluent discharge(waste) water, hazardous wastes and air. All of these methods are promulgated by the USEPA. Many of these methods are similar to those used in many countries throughout the world. In addition to these methods for organic analytes, one has an extensive compilation of wet chemical and physical methods for metals, inorganics and physical properties available. These methods employ such techniques as atomic absorption spectroscopy(AAS), graphite furnace atomic absorption spectroscopy(GFAAS) and inductively-coupled plasma spectroscopy(ICPS).

ION AND LIQUID CHROMATOGRAPHIC TECHNIQUES

Liquid chromatographic methods are employed when the analytes of interest lacked sufficient vapor pressure to be volatilized without decomposition. An exception to this criterion is the group of organic compounds known as the polyaromatic hydrocarbons; they may be determined by either gas or liquid chromatography(see Methods 550, 550.1, 610, 8100, 3640, 8310 and T0-13).

Methods using HPLC for the separation employ two types of detection; ultraviolet radiation at 254nm or fluorescence radiation(excitation at one wavelength and emission at another wavelength(see Methods 531.1, 547, 549, 550, 550.1, 605, 610, 632, 632.1, 635, 636, 637, 638, 639, 640, 641, 642, 643, 644, 8310, T0-5, T0-6, T0-8, T0-11, and T0-13).

The methods utilizing Ion Chromatography all use conductivity as the means of detection with the exception of Method 218.6, which is a colorimetric method using the absorption at 530nm.

Each of the above mentioned methods are listed in Tables 3-7 inclusive.

TABLE 5
ION CHROMATOGRAPHIC METHODS FOR VARIOUS ANALYTES

<u>METHOD NUMBER(USEPA)</u>	<u>TECHNIQUE</u>	<u>ANALYTES</u>	<u>DETECTOR</u>
300.0(A) Approved Dec.1989	CHEMICAL SUPPRESSION	F ⁻ , Cl ⁻ , NO ₂ ⁻ , Br ⁻ , NO ₃ ⁻ , HPO ₄ ²⁻ , SO ₄ ²⁻	CONDUCTIVITY
300.0(B) Approved Dec.1989	CHEMICAL SUPPRESSION	F ⁻ , Cl ⁻ , Br ⁻ , ClO ⁻ , BrO ₃ ⁻ , NO ₂ ⁻ , ClO ₃ ⁻ , NO ₃ ⁻ , HPO ₄ ²⁻ , SO ₄ ²⁻	CONDUCTIVITY
300.6	CHEMICAL SUPPRESSION	ACID RAIN SAMPLES Cl ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻	CONDUCTIVITY

300.7	CHEMICAL SUPPRESSION	ACID RAIN SAMPLES	CONDUCTIVITY
		$\text{Na}^+, \text{K}^+, \text{NH}_4^+, \text{Mg}^{2+}, \text{Ca}^{2+}$	
<u>DIONEX</u>	MICRO MEMBRANE SUPPRESSOR	$\text{F}^-, \text{Cl}^-, \text{SO}_4^{2-}$	CONDUCTIVITY
<u>DIONEX</u>	CHEMICAL SUPPRESSION & GRADIENT IC	35 ANIONS	CONDUCTIVITY
218.6	POST-COLUMN COLORIMETRIC RXN. & PHOTOMETRIC DETECTION	$\text{Cr}^{6+} (\text{CrO}_4^{2-})$	PHOTOMETRIC 530nm
BIOLL WATERS	ION CHROMATOGRAPHY	$\text{NO}_2^-, \text{NO}_3^-$	CONDUCTIVITY
ASTM STP 786	CHEMICAL SUPPRESSION	HCN(alkali absorption-hydrolysis to sodium formate	CONDUCTIVITY

TABLE 6
HAZARDOUS WASTE-SOLID WASTE TEST METHODS(USEPA)

<u>METHOD</u>	<u>ANALYTES</u>
8010	Halogenated Volatile Organics; P&T,GC/Hall Det.
8011	EDP & DBCP; GC/ECD.
8015	Nonhalogenated Volatile Organics;P&T,GC/FID.
8020	Aromatic Volatile Organics; P&T, GC/PID.
8021	Aromatic Volatile Organics; P&T,GC/FID.
8030	Acrolein,Acrylonitrile,Acetonitrile;P&T, GC/
8040	Phenols; Extraction and GC/FID or GC/ECD.FID.
8060	Phthalate Esters; Extraction & GC/FID or GC/ECD.
8070	Nitrosamines; Extraction & GC/FID or GC/ECD.
8080	Organochlorine Pesticides & PCBs;GC/ECD;GC/Hall
8090	Nitroaromatics & Cyclic Ketones;Extraction & GC/FID or GC/ECD.
8100	PAHs;Extraction and GC/FID.
8110	Haloethers; Extraction & GC/Hall
8120	Chlorinated Hydrocarbons;Extraction & GC/ECD.
8140	Organophosphorus Pesticides;Extraction and GC/FPD Or GC/Thermionic Detector.
8141	Same with Capillary Columns.
8150	Chlorinated Herbicides; Extraction and GC/ECD or GC/Hall Detector.
8240/60	Volatile Organics; P&T and GC/MS.
8250/60	Semivolatile Organics; P&T,GC/MS,FSOT Column.
8270	Semivolatile Organics;Extraction,FSOT Column, GC/MS.
8280	Polychlorinated Dibenzo-p-Dioxins & Polychlor- inated Dibenzofurans; FSOT Column,GC/MS.
8310	PAHs;HPLC and UV at 254nm or Fluorescence at 280nm Excited & 389nm Emission.
3640	A GPC Clean-Up Method to separate Pesticides, Phenols and PAHs. PAHs then separated by HPLC Method 8310.

ANCILLARY TECHNIQUES

As stated above, we have a variety of ancillary techniques, which are employed with the chromatographic methods, to simplify the overall procedures, increase the selectivity and/or sensitivity, or aid in the concentrating of the sample analytes. These various techniques are summarized in Tables 2B and 2C.

TABLE 7

AIR QUALITY MONITORING TEST METHODS(USEPA)

<u>METHOD</u>	<u>ANALYTES</u>
PSD/VC/01	Vinyl Chloride. Collection by Charcoal Adsorption; Analysis by GC/FID.
TO-1	Volatile, nonpolar organics(eg., aromatic hydrocarbons, Chlorinated hydrocarbons) having B.P. in Range 80°-200°C. Tenax-GC Adsorption and GC/MS Analysis.
TO-2	Highly Volatile, Nonpolar Organics(eg., Vinyl Chloride, Vinylidene Chloride, Benzene, Toluene) having B.P. in Range -(15°-(+)120°C. Carbon Molecular Sieve Adsorption and GC/MS Analysis.
TO-3	Volatile, nonpolar Organics having B.P. in Range (-)10°-(+)200°C. Cryogenic Trapping and GC/FID or ECD Analysis.
TO-4	Organochlorine Pesticides & PCBs. High Volume Polyurethane Foam(PUF) Sampling & GC/ECD Analysis.
TO-5	Aldehydes & Ketones. Dinitrophenylhydrazine(DNPH) Liquid Impinger Sampling and HPLC/UV Analysis.
TO-6	Phosgene. HPLC/UV Analysis.
TO-7	N-Nitrosodimethylamine. Thermosorb/N Adsorption.
TO-8	Cresol/Phenol. Sodium Hydroxide Liquid Impinger with HPLC.
TO-9	Dioxin. High Volume Polyurethane Foam (PUF) Sampling with High Resolution GC/High Resolution MS(HRGC/HRMS).
TO-10	Pesticides. Low Volume <u>PUF</u> Sampling with GC/ECD Analysis.
TO-11	Formaldehyde. Adsorbent Cartridge(Silica Gel/DNPH) Followed by HPLC
TO-12	Non-Methane Organic Compounds(NMOC). Cryogenic Pre-Concentration & Direct Flame Ionization Detection(PDFID).
TO-13	Benzo(A)Pyrene(B(a)P) & Other PAHs. Adsorption on XAD-2 or PUF Filters. GC & HPLC Analysis.
TO-14	Volatile Organic Compounds(VOCs). Summa ^R Passivated Canister Sampling & GC.

Solid-phase extraction⁸⁻¹⁰ is a technique which utilizes small disposable tubes containing silica-gel-based or alumina-based bonded phase packings to separate analytes of interest from impurities. A more recent development for this technique has been the 3M EMPORE^R disks which are made of Teflon^R embedded with the appropriate packing particles. The extractions may be performed one of three ways:

1. Selective extraction in which the impurities are not retained but the compounds of interest are.
2. Selective washing in which all sample components are retained by the packing; then choosing a solvent system strong enough to remove the impurities but weak enough to leave the components of interest retained.

3. Selective elution in which a solvent system is chosen to elute the components of interest but leaves the strongly retained impurities behind.

The advantage of SPE is that the sample components of interest are in a small volume of solution and free of interferences.

Microextraction^{11,12} refers to the liquid-liquid extraction procedure in which the volume of extracting solvent is much smaller than the volume of the volume containing the sample(usually water). The technique is probably the easiest and fastest and requires the least special apparatus. Because of the low ratio of extracting solvent volume to sample volume, microextractions are only rewarding when the solute partition coefficients are large. In a typical microextraction, the sample volume may range from 10-100mL, whereas the extracting solvent volume may range from 0.2-10.0mL. Often a salt(NaCl or Na₂SO₄) is added to the sample phase to increase the ionic

TABLE 8
CHARACTERISTICS OF STATIC AND DYNAMIC HEADSPACE
ANALYSES

<u>STATIC</u>	<u>DYNAMIC</u>
Equilibrium exists between sample and gas phase.	No equilibrium.
No purging.	Purging of sample phase.
Sample is an aliquot of gas phase.	Sample is concentrated as purge gas passes through adsorbent.
Sample is injected onto chromatographic column by gas-tight syringe.	Sample is thermally desorbed from adsorbent trap directly onto the chromatographic column.
Up to 60 samples per day.	8-10 samples per day.

strength and thus increase the value of the partition coefficient. The advantage of this technique is that the analytes are separated and concentrated in the extracting solvent; thus, analysis can be performed directly without further concentration.

Headspace sampling¹³⁻¹⁶ is an indirect method for the determination of analytes from a liquid or solid sample wherein the vapor phase is analyzed after it has been in equilibrium with the sample or has been purged from sample and trapped onto an adsorbent. If the vapor phase has been equilibrated with the sample it is referred to as a static technique. If vapor phase has been purged from the sample (no equilibrium exists) and then subsequently adsorbed and thermally desorbed it is referred to as a dynamic technique.

Both techniques have advantages and disadvantages. Table 8 lists the more important distinctions between

static and dynamic headspace analysis. A variation of the static technique is the successive equilibration technique of McAuliffe¹⁷. The big advantage of the headspace technique is that the sample solvent or diluent gas does not give a response from the gas chromatographic detector.

A variety of selective detectors¹⁸ are available to the environmental chemist for both GC and LC. One will note that most of the methods given in Tables 3-7 employ GC because of its availability at the time of development. A more specific type of detector which is receiving more attention is the Atomic Emission Detector(AED). This detector system will selectively furnish signals(peaks) from compounds which contain the element(s) which are being monitored by the detector. This detector system has been coupled with the Infrared Detector(IRD) and the Mass Selective Detector(MSD) to furnish more specific interpretation of the resulting chromatograms¹⁹. Another detection technique which is very popular is mass spectrometry²⁰. One has the capability of either gas chromatography-mass spectrometry(GC/MS) or liquid chromatography-mass spectrometry(LC/MS). This latter technique(LC/MS) is acquiring more attention because of the capability of interfacing a Thermospray Module between the chromatograph and the mass spectrometer. This eliminates the older technique of the hot moving wire belt to vaporize the organic solvent(mobile phase). An added feature is that the Thermospray Module is compatible with reversed-phase liquid chromatographic systems.

SUMMARY

It is essential for us to recognize that science and technology must play important roles in solving our environmental problems. Additionally, to combat

threats to our environment we must understand the nature and magnitude of the problems involved. Under the direction of people with a strong environmental consciousness and a basic knowledge of the environmental sciences; and especially analytical chemistry, will mankind survive on the limited resources of this planet.

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