

A REVIEW OF CONCENTRATION TECHNIQUES FOR TRACE CHEMICALS IN THE ENVIRONMENT



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OFFICE OF TOXIC SUBSTANCES
U.S. ENVIRONMENTAL PROTECTION AGENCY
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ABSTRACT

The purpose of this report is to present a survey of the methods which have been used for the concentration of trace substances from the environment prior to analysis.

Methods for the preconcentration of organic and inorganic (particularly heavy metal) compounds from water, air and solids (soil and tissue) are discussed. The general characteristics of each method are presented, and they are discussed in terms of their applicability to a large-scale monitoring effort.

The recent literature on the bioaccumulation of trace substances has also been reviewed. While most bioaccumulation is non-quantitative in nature, plant and animal studies may serve as useful indicators of long-term contamination of the environment.

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SECTION ONE

INTRODUCTION

1.0 Accumulation Systems

Many toxic materials are present in environmental media in concentrations (generally in the parts per million to parts per trillion range) which are large enough to have adverse effects on human health but too small for direct detection and measurement. In such cases a preliminary accumulation, or preconcentration step is necessary before accurate quantitative analyses can be made. Such preconcentrations can be carried out using a wide variety of mechanical, chemical, and biological systems, which will be considered here under the general title of accumulation systems.

This report summarizes current research into the application of accumulation systems to the collection of toxic chemicals from a number of environmental media, including air, fresh and salt water, and soil. The systems considered exploit a wide variety of chemical, physical, and biological principles, including mechanical filtration (for the separation of dispersed particulate phases), chelation, ion exchange, solvent extraction, adsorption, and active transport.

1.1 Parameters Which Affect Accumulation

Accumulation systems can work on chemical, physical, or biological principles. Where the accumulant exists as a separate, dispersed phase within a particular medium, a physical separation of phases will often suffice for its concentration. An example of this is high-volume sampling for particulate air pollutants. When the accumulant is dissolved in or adsorbed onto the medium, however, some kind of chemical separation is necessary. Chemical separations include such processes as extraction, adsorption, and complex formation. In biological accumulation, a living system processes the medium and incorporates the chemical into its own structure.

The efficiency of accumulation can be determined by the collection parameters which describe both the medium and the concentration technique. Some of these parameters, such as temperature and sample size are important for all accumulation methods. Other parameters are only relevant for selected systems. For an accumulation from aqueous media the pH and ionic strength of the water are often important. For the concentration of trace substances from air, the sampling rate or pressure are important parameters. Variation of any of these parameters may be used to vary the types of compounds which are accumulated by a given system.

The method of concentration which is the most sensitive to the widest variety of parameters is bioaccumulation. Any factor which affects the growth and metabolic rate of an organism (i.e. temperature, food supply, light) will affect the rate of uptake of a trace element. This makes it very difficult to obtain accurate quantitative information on trace contaminants from biological systems, although they can be excellent qualitative indicators of contamination.

1.2 Criteria for Comparing Concentration Techniques

For most samples containing a trace chemical in an environmental medium more than one accumulation method can be used to concentrate the sample for analysis. A number of criteria are used to choose the best method, the most important of which are:

1. Quality of Results

- a) reproducibility of accumulation
- b) stability of samples during collection or storage
- c) ease of preventing extraneous results due to impurities in the sampling system
- d) range of useability (i.e. pH, temperature, salt concentration, accumulant concentration)
- e) concentration factor

2. Costs

- a) monitoring time requirements and sample sizes
- b) adaptability for field monitoring
- c) degree of technical ability needed for accurate and reproducible use of the accumulator system
- d) ability to recover expensive reagents and materials

3. Relationship to Subsequent Procedures

- a) ease of interface with chosen method of analysis
- b) degree of specificity of collection

Point three relates to the means of analysis and the additional processing that must be done to prepare the concentrated sample for analysis. Since the ultimate choice of an accumulation system should be made with the analytical method in mind, an additional consideration is the quality of the results obtainable from the chosen method of analysis.

1.3 Comments on Analytical Procedures

Currently, the most widely used methods for the quantitative analysis of environmental samples are gas-liquid chromatography (GLC) for organic substances and atomic absorption spectroscopy (AAS) for trace metals.

Advantages shared by both methods include:

- a) excellent interfacing with some accumulation systems
- b) mixtures can generally be analyzed without time consuming pre-separations
- c) high sensitivity and reproducibility of results
- d) ease of operation

Infrared spectroscopy (IR) and nuclear magnetic resonance spectroscopy (NMR) are not suitable for organic trace analysis in most substances because of low sensitivity and lack of selectivity. "Wet" analysis and colorimetric analysis are sometimes used for inorganic trace analysis, but these methods usually require tedious separations. In certain cases, however, an effective interface can be made between an accumulation method with high specificity and a colorimetric assay.

New methods are emerging which combine the attractive features of GLC and AAS with increased abilities for qualitative analysis. For organic samples, the combination of gas chromatography with mass spectrometry (GC/MS) is very effective for both identification and quantitative analysis of the components of complex mixtures. Liquid-liquid chromatography will undoubtedly soon emerge as an important method for the separation and analysis of organic compounds, particularly for water-soluble or non-volatile species.

Similarly, the method of x-ray fluorescence (XRF) is capable of identifying and assaying mixtures of trace metals. It is, moreover, a nondestructive technique which is suitable for the direct analysis of particulate matter on a filter. Other methods that are used to identify and analyze inorganic samples include spark source mass spectrometry, emission spectroscopy, and neutron activation analysis. Cost is the most serious drawback to the use of these newer methods. Equipment for XRF costs 5-6 times as much as equipment for AAS; GC/MS can cost 20 times as much as GLC.

1.4 Format of the Report

The discussion of accumulation systems in this report has been organized by the medium from which the trace element is accumulated: air, water, or a solid phase (soil and tissue). Bioaccumulation has been treated separately because, in this case, the parameters which affect the accumulation are more dependent upon the technique and species used than on the medium from which the accumulation takes place.

Each section consists of a general description of the concentration methods which are used for analysis from the given medium. This includes a discussion of the

physical or chemical basis for the method, along with a description of the experimental techniques which are currently being used.

A chart is included at the end of each section or subsection which lists the accumulation procedures which have been found in the literature along with the chemicals which were obtained and the analytical details. A complete bibliography of the relevant literature also follows each section or subsection.

Section Two will discuss concentration techniques which are used for the isolation of chemicals from aqueous solutions. Organic and inorganic accumulants are treated in separate subsections. Section Three describes accumulation techniques for air samples. Its two major subsections deal with gaseous materials and particulate matter. Section Four treats accumulation from solid media such as soils and living tissues. Bioaccumulation from all media is discussed in Section Five. Biological analysis is a very important part of any environmental program. Naturally occurring systems serve as valuable indicators of long term problems with toxic materials.

SECTION TWO

ACCUMULATION FROM WATER

2.0 Introduction

Aqueous systems contain a large number of elements and compounds in trace quantities. These can enter a body of water from the waste streams of factories or dwellings, from run-off from the soil, or from impurities picked up by rainwater from the air. In order to concentrate such substances for analysis they must first be separated from the water and then from any other major constituents of the system. With sea water, for example, this requires a separation from the salt which is present in a concentration of about 3%.

There are many problems with obtaining a "characteristic" sample from an aqueous system since the concentrations of trace chemicals can vary with the temperature and flow rate of the water. The actual quantitative measurement of a sample is fairly simple since the volume is insensitive to most collection parameters. Usually a grab sample is taken using a glass or polyethylene bottle. Otherwise, water can be passed through an adsorption column for the collection of trace contaminants. The most important sampling parameters are pH, ionic strength, and in some cases, temperature.

In general, methods for the accumulation of trace substances from aqueous systems do not vary significantly between fresh and salt water, since the properties of the accumulant are the primary consideration in choosing a concentration technique. Therefore this section deals with both media.

2.1 Accumulation of Organic Compounds

2.1.0 Introduction

Organic compounds can be roughly divided into two classes based upon the water solubility of the neutral molecules. The compounds which are of interest as pollutants and toxicants are almost invariably among the lipophilic, or non-water soluble organics. The possibility of toxic effects of such compounds is increased by the fact that they may easily be taken up by lipophilic tissues and accumulated in animals. Such accumulation is much less likely for water-soluble organic molecules which may remain in solution while passing through the body. Water-soluble organics such as humic acids, sugars, and proteins are non-volatile and therefore are not amenable to measurement by gas chromatography, currently the most common technique for organic separation and analysis. Methods for accumulating these

water-soluble organic molecules usually involve isolation and purification for a single desired substance. Because of this lack of generality in accumulation methods as well as the limited number of references to such compounds they will be mentioned only briefly in this report.

Immediate polarity compounds such as glycols, phenols and small organic acids can usually be separated from water into an organic phase with the proper choice of pH and extraction solvent. pH control is important since a compound in an ionized form will tend to be much more water-soluble. Very volatile organic compounds such as chloroform or benzene also require care in their accumulation and analysis to avoid loss by evaporation. A number of special methods have been devised to measure these compounds.

As mentioned above, gas chromatography (GC) is currently the method of choice for the separation of organic mixtures. A flame-ionization detector is used for the analysis of most organic compounds. Not only does this detector give sub-nanogram sensitivity, it greatly simplifies sample preparation since it is insensitive to air and water. The electron-capture detector and micrometer are even more sensitive for halogenated compounds than the flame detector.

The two methods which are most often used for the accumulation of organic molecules from water are liquid-liquid extraction and adsorption onto a lipophilic surface.

Other methods, such as reverse osmosis and distillation, will be discussed only briefly since they have been applied only to a very limited extent.

2.1.1 Extraction

Extraction is probably the most widely-used method for the accumulation of organic compounds from various environmental media. The accumulation of organic compounds from water by extraction with various solvents is discussed below. The variables involved include the nature of the solvent, the ratio of solvent to water, the pH of the water, and the ionic strength of the water.

The single most important parameter to consider when selecting an organic solvent for extraction is its polarity. It is convenient to measure polarity in terms of ϵ^0 , a value derived from the use of the solvent in liquid chromatography on alumina columns. Table 2-1 gives the ϵ^0 values for a large number of the most common solvents. These values range from 0 for pentane (and even negative values for fluoroalkanes) through about 1 for solvents such as ethylene glycol. Inspection of this increasing series of polarities shows that the more polar solvents (above approximately $\epsilon^0=0.5$) are water-soluble, whereas those below $\epsilon^0=0.5$ are not. In order to extract lipophilic organic compounds from water one needs to select a water

TABLE 2-1

SOLVENT PROPERTIES OF LIQUID CHROMATOGRAPHIC INTEREST

SOLVENT	$\epsilon^0(\text{Al}_2\text{O}_3)$
Fluoroalkanes	0.25
n-Pentane	0.00
Isooctane	0.01
Petroleum ether	0.01
n-Decane	0.04
Cyclohexane	0.04
Cyclopentane	0.05
1-Pentene	0.08
Carbon disulfide	0.15
Carbon tetrachloride	0.18
Xylene	0.26
i-Propyl ether	0.28
Toluene	0.29
Chlorobenzene	0.30
Benzene	0.32
Ethyl bromide	0.37
Ethyl ether	0.38
Ethyl sulfide	0.38
Chloroform	0.40
Methylene chloride	0.42
Methyl-i-butylketone	0.43
Tetrahydrofuran	0.45
Ethylene dichloride	0.49
Methylethylketone	0.51
1-Nitropropane	0.53
Acetone	0.56
Dioxane	0.56
Ethyl acetate	0.58
Methyl acetate	0.60
Amyl alcohol	0.61
Dimethyl sulfoxide	0.62
Aniline	0.62
Diethyl amine	0.63
Nitromethane	0.64
Acetonitrile	0.65
Pyridine	0.71
Butyl cellosolve	0.74
i-propanol, n-propanol	0.82
Ethanol	0.88
Methanol	0.95
Ethylene glycol	1.11

insoluble extraction solvent, thus limiting the use of the more polar solvents. The exact selection of a solvent from among this nonpolar, water-immiscible group depends on the range of organic compounds which is to be accumulated from the water. Pentane, for example, is ideal for extracting alkanes, but not very efficient for extracting fatty acids. Methylene chloride, on the other hand, is particularly effective for a wide range of solutes. It will efficiently extract alkanes and fatty acids, for example. Other more aromatic solvents may be suitable for extracting polycyclic aromatic hydrocarbons from water.

Although the more polar extraction solvents cannot be used for direct extraction of water, they are particularly important for fractionating extracts obtained with nonpolar solvents. For example, certain pesticide analyses are based on an initial extraction with methylene chloride. This methylene chloride extract is in turn extracted with a more polar solvent such as acetonitrile in order to remove the more polar components from the methylene chloride extract. This type of dual extraction can have wide applicability and excellent specificity when dealing with a known class of solutes.

A review of the literature indicates that methylene chloride has been used for the accumulation of organic

compounds from the Charles River in Boston;¹ that pentane has been used for the accumulation of dissolved organic material in sea water;² and that benzene has been effectively used for the accumulation of organochlorine and organophosphate insecticides from lake waters.³

The second important parameter, the solvent-to-water volume ratio, can be selected from considerations such as the known solubility of the organic solvent in water and the concentration effect to be achieved. With most of the nonpolar solvents the use of about 50 to 100 milliliters of solvent per liter of water seems to be suitable.

The pH of the water in an extraction influences the chemical nature of the compounds which are extracted. For example, to extract both neutral and acidic compounds from water one would adjust the pH to 1 or 2. To extract both neutral and basic compounds, the pH would be adjusted to 13 or 14. Extracts obtained under these conditions can frequently be further separated into neutral and, for example, acidic components by another extraction at suitable pH.

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1. Hites, R.A., and Biemann, K., Science, 178: 158-69 (1972).
 2. Blumer, M., in Proc. Symp. Organic Matter in Natural Waters, Hood, D.W., ed., Univ. of Alaska, 1970.
 3. Konrad, J.G., Pionke, H.B. and Chesters, G., Analyst, 94: 490-492 (1969).

Ionic strength is a parameter which is seldom adjusted, although it is important for the efficient extraction of material from fresh water. Solutions of high ionic strength dissolve less organic solute than solutions of low ionic strength. Therefore, when extracting fresh water, it is advisable to increase its ionic strength with, for example, sodium chloride before proceeding with the extraction.

Large concentration factors can be achieved with solvent extraction. The primary concentration factor is due to the differential volume ratio of water to organic solvent (a factor of 10 to 20). Subsequent concentrations can be achieved by evaporating the organic solvent to a very low volume. For example, if one liter of water is extracted with 200 milliliters of methylene chloride and then evaporated to 100 microliters, a concentration factor of 10^4 is achieved. This concentration by evaporation also places some constraints on the solvent selection. Thus, if one wants to evaporate the solvent subsequent to extraction, a volatile solvent must be selected. Again, pentane and methylene chloride recommend themselves in this regard.

The efficiency of solvent extraction is highly dependent on the polarity of the solvent and the polarity of the solute. For example, with nonpolar solvents and

nonpolar solutes, extractions exceeding 90% in efficiency are common. These values are normally achieved by repetitive extractions with small volumes of solvents; three extractions are usually sufficient. As the polarity of the solute increases, the efficiency of extraction usually decreases. For example, phenols can be extracted with medium polarity solvents such as chloroform or benzene (after the proper pH adjustment) with efficiencies of 30 to 70 percent. Fatty acids can usually be extracted with medium polarity solvents such as methylene chloride to the extent of 75 to 90 percent.

As a method of accumulation, extraction has the advantage of being very straightforward and easy to carry out. Grab samples of water are usually collected for this procedure. Since only a few reagents are used, there is little chance of sample contamination, and of catalyzing reactions of the organic compounds. An extraction is inconvenient to carry out in the field, however. Therefore the necessity of transporting the sample to a laboratory introduces the problem of sample preservation. There is also a disadvantage in working with solvents which are usually both toxic and flammable. The disposal of such solvents could become a problem in the field.

If the extracting solvent is evaporated before analysis, the most volatile organic compounds are lost. This does not pose a great problem if a low-boiling solvent such as methylene chloride is used and a careful evaporation step is carried out (a Kuderna-Danish evaporator has been found most successful in this regard).

Extraction is very appropriate for accumulation from small volumes of water (up to 4 liters), but it is inappropriate for very large samples. With this sample size a G.C. with a flame-ionization detector can give analyses on concentrations down to a part per billion; larger samples should only be needed for analyses in the part per trillion range or when a less sensitive analytical technique is used.

2.1.2 Adsorption

Adsorption of organic compounds on lipophilic surfaces is another method for accumulating organic compounds from both air and water. The pertinent operational parameters are the chemical nature of the adsorbent, factors influencing the mass transfer between the fluid and the adsorbent surface (such as particle diameter and fluid flow rate), and the means of removing the materials of interest from the adsorbent.

Most of the adsorbents used for environmental samples

have lipophilic, that is, nonpolar surfaces. These adsorbents include activated charcoal and a variety of polymeric materials. The organic compounds which may be accumulated on these adsorbents are usually of low to intermediate polarity. Although some adsorbents have been used in batch processes (i.e. 50 mg of activated carbon is added to 100 cc of water and stirred), the usual procedure is to pack a column with the adsorbent of choice and run the aqueous sample through it. This is essentially a chromatographic system in which the water (or air) acts as a mobile phase to elute the adsorbed substances. Thus it is usually the volume of water (or air) passing through an adsorbent, rather than the amount of contaminant, which determines the extent of an accumulation. Once the "break-through" volume of a substrate (the volume at which it starts to elute from the column) has been reached, its concentration in the column is not proportional to its concentration in the medium being measured. Thus it is desirable to insure that the break-through volume of the earliest eluting component of interest is not exceeded. This factor may be controlled by changing the length of the column used or the total volume of water which passes through it.

When lipophilic adsorbents are used there is very seldom any alteration of the aqueous sample before adsorp-

tion. For the collection of ionizable organics, however, it is often necessary to control the pH in the same manner as for liquid-liquid extraction.

Once a compound has been adsorbed onto a solid substrate, it must be desorbed in a form that can be used for further analysis. For gas chromatographic analysis this requires a solution or gaseous state for the accumulant. This is accomplished either by eluting the column with an organic solvent or by heating the adsorbent to desorb the desired substances. Heat desorption may be directly into a G.C. or into a cold trap or other adsorbent column. In choosing a resin as an accumulator, its ability to desorb compounds under fairly mild conditions is just as important a consideration as its adsorptive capabilities. In general, there is a molecular weight above which compounds cannot be desorbed from a given adsorbent.

Until four years ago the most commonly used lipophilic absorbant was activated charcoal. This is a material still favored by many workers within the Environmental Protection Agency. A classic use of charcoal involved the collection of water pollutants by passing several hundred thousand gallons of river water through large-scale carbon filters.⁴ These filters were then extracted with chloroform in a Soxhlet apparatus. Approxi-

4. Rosen, A.A., Skeel, R.T., and Ettinger, M.B., Journal WPCF, 35: 777-782 (1963).

mately 1600 grams of extract were obtained and subsequently fractionated by large-scale chromatographic and distillation techniques. Identification of the specific compounds was carried out with infrared and ultraviolet spectrometry. Materials such as naphthalene, tetralin, bis(chloroethyl)-ether, diisobutyl carbinol, and phenylmethyl carbinol were found in this river. More recently, using G.C. and G.C.-M.S. analyses, hydrocarbons, chlorinated hydrocarbons, pesticides, and many other organic compounds have been isolated from water.⁵

The use of activated carbon creates certain problems. First, considerable variability is observed in collection efficiency apparently due to variations in the moisture content of the carbon. Second, it is well-known that activated carbon can act as a catalyst for oxidation and other reactions, so that compounds which are adsorbed may be chemically transformed before or during the desorption step. A third problem is irreversible adsorption: some compounds, particularly those of intermediate to high polarity, can be irreversibly adsorbed, thus preventing a measurement of their true abundance in the environment.

The current method of drying the carbon and extracting the organics from it with either chloroform, benzene, or tetralin introduce further limitations, since large

5. Kleopfer, R.D., Fairless, B.J., Environmental Science & Technology, 6: 1036 (1972).

amounts of volatile compounds may be lost during both the long drying period and the solvent concentration step.

Most of these problems with activated carbon have been overcome by the use of a wide variety of polymeric adsorbents such as XAD, Porapak, Tenax, and Chromosorb 100.

These materials are all lipophilic. They accumulate non-polar compounds with excellent efficiency and intermediate polarity compounds with moderate efficiency.

Work by Burnham⁶ used macroreticular resins manufactured by Rohm and Haas called XAD-2 and XAD-7 for the adsorption of organic compounds from potable water. This resin is a highly porous material (average pore size=90Å) formed from polystyrene. In operation the resin was ground to 100-150 mesh size and water was passed through it at a flow rate of four bed-volumes per minute. The organic compounds of interest were removed from the adsorbent by elution with ethyl ether. Using this technique, compounds such as acenaphthalene, methylnaphthalene, and methylindene were found in well water from Ames, Iowa. These authors also tested the efficiency of retention of various compounds on these resins. Compounds such as ketones, esters, aromatic hydrocarbons and alkyl phenols were retained to 100%. Phenol itself was retained to 45% and various sulfonic acids were retained only to approximately 20%. (See Table 2-2).

6. Burnham, A.K., Calder, G.V., Fritz, J.S., Junk, G.A., Svec, H.J., and Willis, R., Analytical Chemistry, 44: 139-142 (1972).

TABLE 2-2*

COMPOUNDS TESTED FOR RETENTION
ON XAD-2 AND XAD-7 RESINS

<u>Compound</u>	<u>Resin (7)</u>	<u>Concentration, ppm</u>	<u>Retention, %</u>
Methyl isobutyl ketone	XAD-2	100	100
n-Hexanol	XAD-2	200	85
Ethyl butyrate	XAD-2	100	100
Benzene	XAD-2	100	100
Naphthalene	XAD-2	0.05	100
Benzene sulfonic acid	XAD-2	3.0	31
p-Toluene sulfonic acid	XAD-2	9.0	23
Benzoic acid	XAD-2	1.0	23
Benzoic acid (pH 3.2)	XAD-2	1.0	100
Phenylenediamine	XAD-2	0.9	98
2-Hydroxy-3-naphthoic acid	XAD-2	0.6	39
Phenol	XAD-2	0.4	45
Phenol	XAD-7	0.4	86
2,4-Dimethylphenol	XAD-2	0.4	100
p-Nitrophenol	XAD-2	0.2	100
2-Methylphenol	XAD-2	0.3	100
4,6-Dinitro-2-aminophenol	XAD-2	0.4	43
Aniline	XAD-7	4.0	100
o-Cresol	XAD-2	0.3	100

* A. K. Burnham, G. V. Calder, J. S. Fritz, G. A. Junk, H. J. Svec, and R. Willis, Anal. Chem., 44: 140 (1972). (Used with permission)

They have since developed a detailed analytical procedure for the use of these resins.⁷

Other research using XAD resins has concentrated on the use of these resins for the accumulation of organic compounds from sea water. Riley and Taylor⁸ demonstrate that alkyl carboxylic acids, various steroids, Vitamin B-12, lindane, DDT, and methylene blue can be collected and recovered with efficiencies in excess of 95%. Humic acids can also be collected, although the recovery is difficult to determine due to the lack of characterization of these materials.

Rohm and Haas have published a study of these resins, particularly of XAD-4 which suggests that these resins might be economically useful for the treatment of organic wastes due to their strong adsorptive properties.⁹

Numerous other chromatographic phases are used for the accumulation of environmental compounds, primarily for air analysis or for analysis of the head-space above a liquid or solid sample. Head space analysis of a liquid sample consists of blowing a stream of gas (helium, argon, nitrogen or air) over or through the liquid in a container. In the outlet of this container is placed a trap into which

7. Junk, G.A., et. al., J. Chrom., 99: 745 (1974).

8. Riley, J.P. and Taylor, D., Analytica Chimica Acta, 46: 307-309 (1969).

9. Kennedy, D.C., Environmental Science & Technology, 7: 138 (1973).

the volatile compounds are carried by the gas stream (see Fig. 2-1). When this trap consists of a cooled tube, the operation is akin to a distillation. Usually a packed column is used as the trapping agent, and the liquid may be heated to increase the number of compounds volatilized and collected. The analytical procedure followed is the same as that used for air analysis by adsorption, and, as for air samples, the rate and volume of gas collection as well as the operating temperature are important parameters. The method may be used for organic compounds with boiling points up to 250°C.

Work by Zlatkis at the University of Houston¹⁰ on the concentration and analysis of volatile trace organic compounds in gases and biological fluids has centered around the use of Tenax as a lipophilic adsorbent. The organic compounds were removed from the adsorbent by re-volatilization. In practice the adsorbent trap was inserted into the gas chromatograph in front of the analytical chromatographic column and heated, thus transferring the trapped organic compounds to the analytical column for direct analysis. Adsorbents such as Porapak and XAD-2 were not suitable for this sort of desorption because they lacked thermal stability. In the case of Porapak, for example, the devolatilization temperature was sufficient

10. Zlatkis, A., Lichtenstein, H.A., and Tishbee, A., Chromatographia, 6: 67-70 (1973).

to also decompose the adsorbent. Carbosieve, produced by Supelco, proved even worse in this regard, since temperatures of 400°C were needed to desorb organic compounds. Such high temperatures caused decomposition of the adsorbent resulting in a considerable number of artifacts in the resulting chromatographic analysis.

Tenax, on the other hand, can sustain relatively high temperatures (350°C). This material, distributed by Applied Science Laboratories in State College, Pennsylvania, is a porous polymer of 2,6-diphenyl-p-phenylene oxide, and it will trap a wide range of volatile organic compounds with excellent efficiency. Traps consisted of glass tubes (11 centimeters long by 10 millimeters O.D.) containing 2 ml of Tenax packing and were constructed to fit in the injector port of a Perkin-Elmer 3920 gas chromatograph. (See Fig. 2-2). Aqueous samples were studied by head space analyses in which the sample was heated to 100°C and stirred vigorously. The vaporized organic compounds were then swept onto the Tenax trap as described above. Sample collection with these systems was rapid (an hour per sample), and the trapped samples could be stored conveniently for at least a week. Zlatkis reported several chromatograms of organic compounds trapped from human urine, human breath, and the Houston atmosphere, without any identification of most of the trapped compounds.

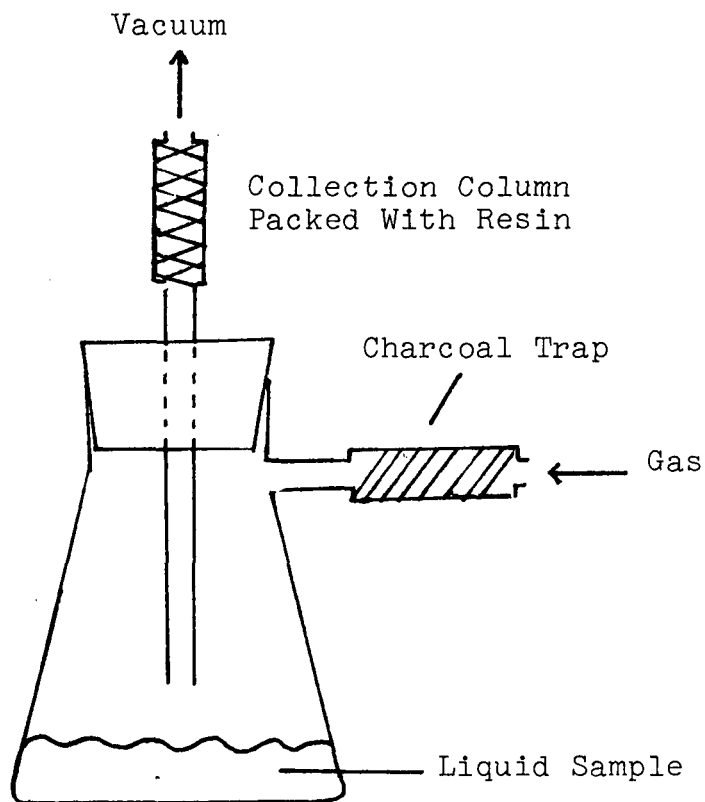


Figure 2-1. Schematic diagram of a cell used to collect headspace vapors from solids and liquids.¹
(Used with permission)

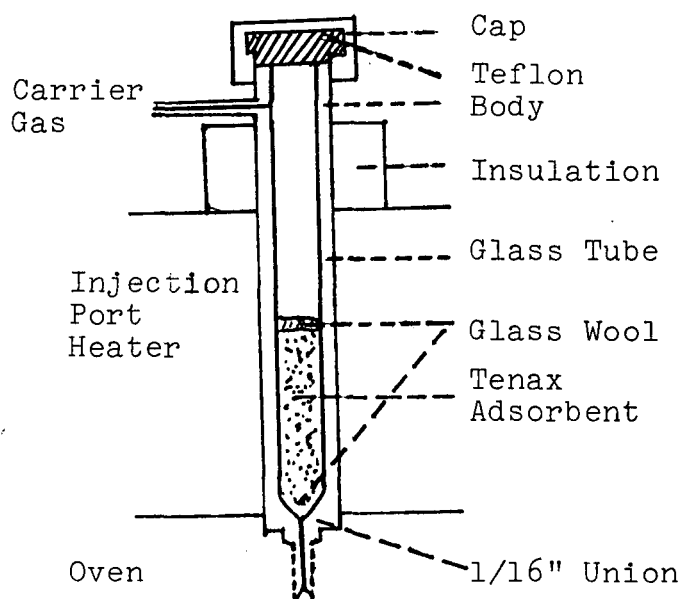


Figure 2-2. Tenax injection port.²
(Used with permission)

¹Mieure, J. P., J. Chrom. Sci. 11: 568 (1973).

²Zlatkis, A., J. Chrom. Sci. 12: 177 (1974).

Tenax is the most widely used resin for this type of analysis although other adsorbents such as Chromosorb and Porapak, which are used for air, can also be used for head space analysis. The Environmental Protection Agency is investigating the use of Tenax for head space analysis in Cincinnati, and has applied it to the study of New Orleans drinking water.¹¹

In summary, adsorption is probably the single most valuable technique for the analysis of organic compounds of intermediate to low polarity which are found in air, water and, to some extent, solids. The primary limitations of adsorption are in the occurrence of artifacts resulting from the decomposition of the organic adsorbent. Most of these artifacts, however, can be avoided by the proper cleaning of the adsorbent before use. XAD resins, for example, can be cleaned of the residual monomer and oligomer species by Soxhlet extraction for several hours with acetonitrile followed by extraction with acetone and methylene chloride.¹² Other lipophilic adsorbents can be cleaned in a similar manner. In most cases, where analysis by devolatilization is used, suitable cleaning of the resin can be achieved by heating the adsorbent in a stream of helium or pure nitrogen for several hours at its maximum tolerable temperature.

11. U.S. Environmental Protection Agency, Draft Analytical Report--New Orleans Water Supply, Nov., 1974.

12. Junk, G.A., et. al., J. Chrom., 99: 745 (1974).

High concentration factors can be achieved by the use of these lipophilic adsorbents as accumulators for organic compounds. The final limitation on concentration enhancement is due to the sample capacity of the adsorbent. The total volume of air or water which can be passed through the lipophilic adsorbent sets a practical limit to the concentration factor and fixes the duration of sample collection. When samples are taken from a polluted body of water, for example, considerable debris is collected on the column, which eventually decreases the flow through the column to impractical levels. In general, concentration factors of 4 to 6 orders of magnitude can easily be achieved using lipophilic adsorbents.

A great advantage in the use of adsorbents is that the solid systems can easily be handled once a sample has been collected in the field. Sealed collection columns can often be stored for long periods of time prior to analysis with no sample decomposition. This is not the case, however, when there is enough adsorbed organic matter to support bacterial growth¹³ (this has occurred on carbon traps). Those systems which allow for direct heat desorption into the G.C. require very little manipulation and the adsorbent is the only chemical in the system which requires purification. This method of desorption does

13. Personal communication: A.A. Rosen, National Field Investigation Center, Cincinnati, December 1974.

have the disadvantage of total sample loss, if G.C. conditions should fail to give a good separation. Extraction methods require more manipulation, but result in the ability to run a number of analyses on one field sample. Unlike extraction, adsorption can accomodate large volumes of water when necessary.

2.1.3 Other Methods

Freeze concentration is a method which has been used for the accumulation of organic compounds from water when the compounds of interest are not necessarily lipophilic. The water sample is frozen slowly, while being stirred, allowing only pure water to be crystallized and leaving all of the impurities in the remaining solution. The choice of technique is critical in order to keep impurities from becoming embedded in the ice as it forms. This method leads to concentrations of only a factor of 10, however, before the recovery of organic molecules drops off drastically. P.A. Kammerer, Jr. and G.F. Lee¹⁴ have accumulated glucose, glycerine, phenylalanine, and citric acid with 88-100% recovery; the average concentration factor was only five in this procedure.

There does not appear to be any advantage to the use of freeze concentration. There are much more efficient

14. Kammerer, P.A., Jr., Lee, G.F., Environmental Science & Technology, 3: 276 (1969).

methods for the accumulation of lipophilic organic molecules. While this form of concentration might be useful for the analysis of water-soluble organic molecules (perhaps by liquid-liquid chromatography), for low-temperature concentration freeze-drying would appear to be a much more efficient method of removing water.

Reverse osmosis is another concentration method which is under investigation.¹⁵ This technique is expected to yield concentration factors of 10 or 20. Since membranes may be chosen with pores of varying sizes, this method of collection may prove to be advantageous for a preliminary separation of organic molecules largely (but not totally) on the basis of molecular size. However, at the present time this technique is not well developed.

Flocculation with FeCl_3 and floatation using surfactants have also been used occasionally to concentrate organic compounds from water, but recoveries from these processes are not quantitative (43-63%).¹⁶

In the past fractional distillation of water was used to separate compounds for further classification and analysis. The use of the G.C. seems to have superceded this method.

15. Deinzer, et. al., "Trace Organic Contaminants in Drinking Water: Their Concentration by Reverse Osmosis", presented before the Division of Environmental Chemists ACS, Los Angeles, 1974.

16. Sridharan, N., Lee, G.F., Environmental Science & Technology, 6: 1030 (1972).

2.2 Accumulation of Inorganic Solutes From Water

2.2.0 Introduction

The accumulation and subsequent analysis of the dissolved inorganic contents of waters are based on the chemical properties of the solutes. This situation contrasts with that of particulates found in air, where physical properties (size distribution, density, mean diameter, etc.) determine the means of accumulation.

Natural waters can be classed according to their total content of dissolved solids:

- Sea water: ca. 3% dissolved solids by weight
- Fresh water: traces of dissolved solids
- Brine: more concentrated than sea water
- Brackish water: intermediate between sea water and fresh water.

Most accumulator systems will work on any of these four classes of water; therefore, no distinction will be made among them in the following discussion, unless specifically noted.

The solutes found in natural and in polluted waters span the periodic table and have widely varying chemical properties. Those occurring in the highest concentrations are the ions of the common salts: Na^+ , Mg^{++} , Ca^{++} , Cl^- ,

$\text{SO}_4^{=}$, etc. and analyses can normally be performed without preconcentration. Large concentrations of other components are encountered occasionally, but they generally are found near their sources, such as the outfall from an industrial plant. Because these components are usually found in trace amounts, some degree of preconcentration or accumulation is convenient, if not actually necessary, as a prelude to quantitative analysis.

Among trace inorganics, those of particular interest are the substances which are toxic to human and other life forms. Their chemical properties range from alkali and alkaline earth metals, such as Cs, Be, and Ba, to transition metals, such as Zn, Cd, Hg, and Pb, to metalloids, such as As and Se. Because of the wide range in chemical properties, no single accumulation system will quantitatively collect all of them. Indeed, in some cases, a given accumulator will not be capable of collecting all the different complexes and oxidation states of a single element present in a sample.

Since the concentration methods discussed below share the characteristic that some part of the system undergoes a change of phase, it is useful to describe these systems by the kind of phase change that occurs. The classification to be used in this report is as follows:

- Chelation/Extraction: A second liquid phase is placed in contact with the aqueous system and the accumulant is extracted into it. Formation of a chelation complex is an essential aspect of this type of system.
- Ion Exchange: A solid phase is placed in contact with the aqueous system and the accumulant is adsorbed onto it.
- Coprecipitation and Cocrystallization: A solid phase is formed which incorporates the accumulant.
- Headspace Analysis: The accumulant is chemically converted (usually by reduction) to a volatile species which is driven out of solution and collected or directly analyzed.
- Flotation: A floating foam is formed which incorporates the accumulant.
- Evaporation, Freeze Drying, and Freeze Concentration: The water is partially or totally removed through a phase change. The accumulants are left as solids or in a more concentrated solution.
- Electrolytic Reduction: The accumulants are collected as metals on the anode of an

electrolytic cell. (Subsequent analysis is performed by anodic stripping voltammetry.)

The three most useful methods are Chelation/Extraction, Ion Exchange, and Coprecipitation and Cocrystallization. The fourth method, head space analysis, is limited to the analysis of those elements which can be reduced to volatile forms, such as Hg, As, and Se, but it is a good method for these elements. The discussion which follows will be limited to these four methods, since the others have been put to relatively little use in the analysis of environmental samples.

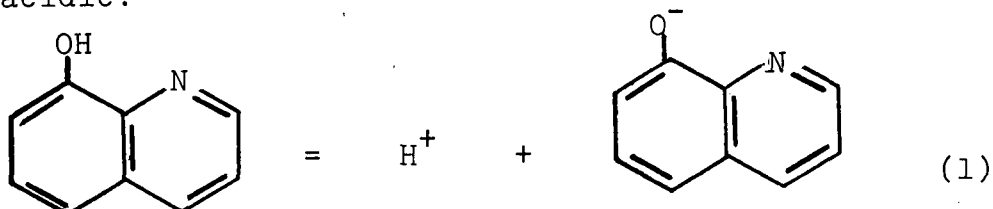
2.2.1 Chelation/Extraction

Solvent extraction, an accumulator method of major importance for organic substrates, is not directly applicable to metal ion accumulants since charged species are generally not soluble in solvents which are immiscible with water. However, most metals can be made to form stable unchanged complexes with organic chelating agents. These complexes can be extracted into an organic phase without difficulty, given a proper choice of solvent and pH.

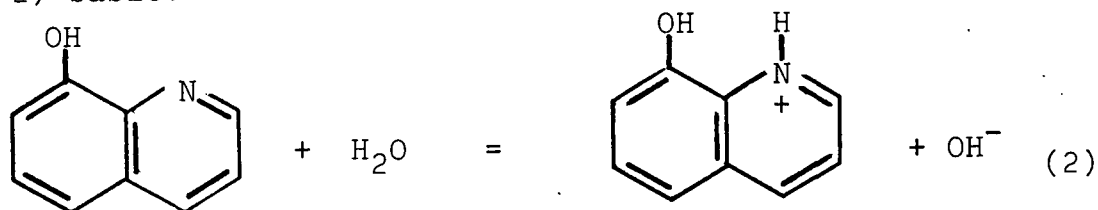
There are several important chemical equilibria that govern chelation/extraction: The acid-base equilibria of

the chelating agent, the formation equilibrium of the chelate complex, and the distribution equilibrium of the complex between the aqueous and organic solvents. For example, the dissociations of 8-hydroxyquinoline are

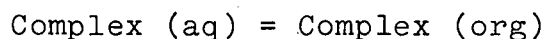
1) acidic:



and 2) basic:



Hence the chemical form of 8-hydroxyquinoline, like all chelating agents, depends on the pH. The basic requirements for a successful chelation-extraction procedure are that a stable complex must be formed between the chelating agent and the metal; the pH must allow the right number of acidic or basic dissociations to yield a neutral complex; and the distribution equilibrium:



must favor extraction into the organic (org) phase. The solvent must be capable of forming a two-phase system with water, and the less water solubility it has, the better

the extraction efficiency. In some chelation/extraction systems, the chelating agent is placed in the aqueous phase prior to extraction, while in others the agent is introduced in the organic phase, the complex forming during extraction.

Examples of chelating agents that have been used in various chelation/extraction systems are 8-hydroxyquinoline, acetyl acetone, diphenyl thiacarbazone (dithizone), and ammonium pyrrolidine dithiocarbamate (APDC). Metals collected with these agents are shown in Figure 2-3. Like all chelating agents, each of these possesses at least two groups that can simultaneously form coordinate bonds to metal ions. The extraction solvents include methylisobutyl ketone (MIBK), ethyl propionate, chloroform and benzene.

The basic equipment for chelation/extraction is the same as that used for organic solvent/solvent extraction, and the procedure is similar. The use of chelating agents and careful control of pH constitute the main procedural differences.

Stability of the aqueous sample can be a problem for some metals, for example Ag and Hg. Immediate extraction in the field is sufficient to stabilize them, but if the water sample is to be taken to the laboratory, it should be acidified in the field. This will prevent, for example,¹⁷

17. F.K. West, P.W. West, and F.A. Iddings, Anal. Chem. 38, 1566 (1966), F.K. West, P.W. West, and F.A. Iddings, Anal. Chim. Acta, 37, 112 (1967).

* Sachdev says Hg collectable with DQA if solution aged 5-6 hrs. with MnO_4^- before extraction.

Figure 2-3: Chelation/Extraction by DQA and APDC

the adsorption of Ag^+ on the walls of the sample vessel. Treatment with 5% HNO_3 + 0.05% K_2CrO_7 is sufficient to stabilize dilute Hg solutions.¹⁸ However, since such acidification increases the possibility of metal contamination, it is advisable to perform the chelation/extraction in the field shortly after the sample is collected, since the organic solution of metal chelates is quite stable.

With a proper choice of chelating agents, most trace metals can be accumulated by chelation/extraction procedures. Although some chelating agents are specific to only one or two metals, others form complexes with a wide range of elements. One especially useful system¹⁹ combines three agents (dithizone, 8-hydroxyquinoline, and acetyl acetone) and quantitatively collects Al, Be, Cd, Co, Cu, Fe, Pb, Ni, Ag, and Zn, all in one extraction at pH = 6. Other documented systems account for at least an equivalent number of elements.

One major class of elements is not generally collected by chelation/extraction procedures, namely the alkali and alkaline earth metals. In addition, mercury is difficult to handle because only some of its compounds can be easily collected. However, other methods do exist for these

18. C. Feldman, Anal. Chem. 46, 99 (1974).

19. S. L. Sachdev and P. W. West, Envir. Sci. Tech. 4, 749-51 (1970).

elements, notably headspace analysis for mercury and ion exchange for the alkali and alkaline earth metals.

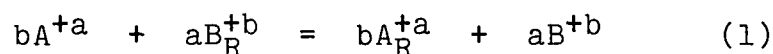
The concentration factor attainable for a given solvent is limited by its water solubility. For example, ethyl propionate forms an approximately 3% aqueous solution by volume. Thus the maximum concentration factor for a single extraction is in the range of 10 to 50. Benzene, with a water solubility of about 0.1% allows for concentration factors up to 500. In any case, concentration factors much in excess of 1,000 are probably inaccessible. Water samples of more than 2 liters are difficult to handle in separatory funnels, and the minimum volume of organic solvent is at least 1-2 ml.

One major advantage of the chelation/extraction method is that the organic extract is an ideal sample for atomic adsorption spectroscopic analysis. In addition to providing a concentration factor, the method also separates out the interfering ions of the common salts (Na^+ , Mg^{++} , Ca^{++} , Cl^-), and the organic solvent enhances the instrument sensitivity by a factor of 2 or 3 over water. Another major advantage is the simplicity of both equipment and procedure. A technician using simple equipment can rapidly produce, from a given water sample, a single extract that is suitable for atomic absorption analysis of a broad range of elements.

This is possible because elements do not usually interfere with each other in atomic absorption spectroscopy.

2.2.2 Ion Exchange

The fundamental chemistry of ion exchange is shown in Equation (1):



where A^{+a} and B^{+b} represent ions in aqueous solution, and A_R^{+a} and B_R^{+b} represent ions bound to resin. The process of ion exchange consists of one kind of ion, originally in aqueous solution, displacing another ion from its binding site on the resin. The result is that the first ion becomes bound to the resin while the second is released to the solution.

If the ions are both univalent and if equilibrium is established, the following equilibrium expression holds:

$$\frac{[A_R^+] (B^+)}{(A^+) [B_R^+]} = K \quad (2)$$

where (A^+) and (B^+) are the concentrations of the ions in solution and $[A_R^+]$ and $[B_R^+]$ are the activities of the ions bound to the resin. It is convenient to tabulate a series of equilibrium constants, $K_1, K_2 \dots$ for a series of ions

A_1^+ , A_2^+ ... all relative to a single ion B^+ . The conventional standard for cationic exchange resins is H^+ , since such resins are normally charged with strong acid before use. Constants in such a series are known as selectivity coefficients, and a typical set is displayed in Figure 2-4. In general, the larger the selectivity coefficient for a given ion, the more strongly it is held by the resin, and the ion with a larger coefficient will tend to displace the ion with a smaller coefficient. However, given a high enough concentration of even a low-valued ion like H^+ (1.0), most ions will be displaced, hence the usefulness of strong concentrated acid such as eluent.

Most ion exchange materials are manufactured in the form of polymeric beads. For example, in the manufacture of DOWEX resins, styrene and divinyl benzene are copolymerized to form a polystyrene type resin with a degree of crosslinking governed by the amount of divinyl benzene in the formulation. Ion exchange sites are then chemically attached to the phenyl rings. According to the chemical nature of the exchange site, the resin can be classed as:

- Strongly acidic (Sulfonic acid)
- Weakly acidic (Carboxylic acid)
- Strongly basic (Quarternary ammonium)
- Weakly basic (Ternary ammonium)

Figure 2-4: Selectivity Coefficients for Uni- and Di-Valent Ions on DOWEX 50-X8 Ion Exchange Resins

- Chelating (Imino diacetic acid)
- Zwitterionic (Weak acid and weak base groups on adjoining polymer strands).

Ion exchange resins are normally used chromatographically. The resin is held in a column, and the aqueous solution is allowed to flow through the column at a slow rate. Much higher collection efficiencies are attainable with columns than can be achieved using batch methods, since a column can be considered a series of "plates". As the aqueous solution passes each plate equilibrium is established, and some of the accumulant binds to the ion exchange sites; the remainder passes to the next plate where still more of the accumulant binds. Given enough plates, collection efficiencies of 100% are possible. Batch methods have been used because of their speed and ease of use, but collection efficiencies are generally not as high as with column methods.

When the entire aqueous sample has passed through the column, and the accumulation is complete, the column is washed with distilled water. Following this, the column is usually eluted in order to remove the accumulant. The eluant solution must contain an ion or combination of ions which can effectively and efficiently displace the accumulant, and in some cases it may also contain a chelating

agent that will complex the accumulator to ease its removal from the column.

As with other types of accumulator systems for inorganic solutes, control of pH is important in the use of ion exchange. This is especially true for resins that are weakly acidic, weakly basic, or chelating, and which are therefore chemically quite sensitive to pH. A weakly acidic resin, for example, has a relatively high affinity for protons. The control of the pH of the eluant is even more important, and the key to efficient separation of the components of a mixture of ions. This is true whether the eluant is a concentrated strong acid or a buffered chelating agent such as EDTA.

The amount of resin and the length of the column are also important. There must be enough binding sites for the accumulators as well as enough plates for effective collection. In addition, the flow rate must be low enough so that near-equilibrium is attained at each plate.

Analytical applications of ion exchange are oriented toward the separation of ions within a mixture. Although much of the early work on ion exchange was performed as part of the Manhattan Project, which demanded accurate separations and analyses of mixtures of lanthanide and actinide rare earths, its potential as an accumulation

system is high. The collection equilibrium (Equation 2) favors the retention of ions at trace levels, making it possible to pass large volumes of sample through an ion exchange column and still have 100% collection of the trace metals. An ion exchange method has been used to concentrate a 40 liter sample.²⁰ It is then possible to use a relatively small amount of eluant to remove 100% of the desired material from the column. In this way concentration factors of several thousand are attainable.

Ion exchange methods, in principle, can be used to collect elements from all parts of the periodic table. This is made possible by the wide choice of resins and eluting solutions. Strong acid resins such as DOWEX 50 can collect nearly any cation, but they are optimum only for the alkaline earths. Polyphosphate type materials are favored for alkali metals, and chelating resins for transition and main group metals. For anions, the basic resins are indicated. (This includes the anionic chloro complexes of Zn, Cd, Bi, Au (III) and Tl (III).)

One significant disadvantage in the use of ion exchange accumulators is the possibility of sample contamination. This arises largely from the use of concentrated, strong

20. T. Joyner, M. L. Healy, D. Chakravarti, and T. Koyanagi, Environ. Sci. Tech., 1, 417-24 (1967).

mineral acids for the conditioning of columns and for elution. Such acids often contain traces of heavy metals which may interfere with accurate analysis.

Another disadvantage lies in the time necessary to process a water sample. Typical flow rates are in the range of milliliters per minute, and in the case of liter size samples, this means hours per sample. On the other hand, one person can run many samples at once since columns can be set up with constant flow reservoirs.

2.2.3 Coprecipitation and Cocrystallization

One of the fundamental procedures in wet analytical chemistry is precipitation of a solid containing the ion or ions to be analyzed. The formation of analytically useful precipitates, however, depends on the solubility product of the solid being exceeded by at least several orders of magnitude. Therefore direct precipitation, in which the desired ion is a major component of the solid, cannot be applied to materials found in solution in trace amounts only.

However, solid formation is still useful as an accumulation technique for trace analysis, if the major component of the solid acts as a carrier which quantitatively collects the desired trace ions. The process is known as

coprecipitation if the carrier is a slightly soluble inorganic base or salt. In general, the solid form of the carrier has the same crystalline structure as the solid form of the accumulant; all accumulants whose solid form matches that of the carrier will be collected.

One commonly-used carrier for coprecipitation is $\text{Fe}(\text{OH})_3$. The formation of this carrier involves addition of ferric ions to an aqueous sample, followed by strong base. The resultant $\text{Fe}(\text{OH})_3$ precipitate collects the hydroxides of the other transition metals.

The use of $\text{Fe}(\text{OH})_3$ presents two problems: the iron that is introduced may interfere with subsequent analysis, and it also may contain its own traces of other metals which will render the analysis invalid. Another coprecipitation method, especially developed for sea water analyses, avoids both problems: the addition of strong base generates a precipitate of $\text{Mg}(\text{OH})_2$ from the natural magnesium ion content of the sea; the hydroxides of Pb and six transition elements are quantitatively collected, and the interference and contamination problems of iron are avoided.²¹

Another type of coprecipitation is involved in the accumulation of radioactive ions from solution. With radioactive barium, for example, BaCl_2 is added,

21. T. Joyner, et al, op. cit.

followed by H_2SO_4 . The resulting precipitate of BaSO_4 is largely unradioactive, but it will contain essentially all the radioactive barium and can be analyzed by radio-assay.²²

When an organic chelating agent is used as a carrier, the process is called cocrystallization. A suitable cocrystallization reagent is relatively insoluble in water, and it forms complexes with the desired metal ions that have even less water solubility than the reagent itself. Their chemical form is quite similar to that of the agents used in chelation/extraction, and it is possible that some chelating agents could be used for both types of concentration.

In practice, the process begins with an aqueous sample. The chelating agent is added in an organic solvent (such as acetone) that is miscible with water. The system is then heated to remove the solvent, and as the solvent is stripped off, crystals of the chelating agent form. If the agent is well-chosen the crystals will contain the desired metal ions as chelation complexes. Examples of chelation agents used in this manner are 5,7-di-bromo-8-hydroxyquinoline, thionalid and 2-mercaptobenzimidazole.

22. D. N. Kelkar and P. V. Joshi, Health Phys., 17, 253-7 (1969).

Elements collected by these agents are shown in Figure 2-5.

As an alternative method of crystal formation, a water soluble chelating agent might be rendered insoluble by the addition of ethanol to the system. In this manner potassium rhodizonate can be used to accumulate Sr and Ba from aqueous solution.²³

Cocrystallization has been tested on approximately 25-30 elements, mostly transition and main group metals. Quantitative collections have been reported for most of them. The alkali metals and the metalloids are not generally collected. As with other accumulators of metal ions, pH is an important collection parameter.

Both coprecipitation and cocrystallization yield solids which can readily be taken up in solution for further processing. Solids produced by cocrystallization can readily be taken up in an organic solvent, or directly inserted into an AA spectrophotometer for analysis. Coprecipitated hydroxides can be taken up in a small amount of acid for further analysis, and at least one investigator has treated such a solution by chelation/extraction.²⁴ Very large concentration factors are

23. H. V. Weiss and M. G. Lai, Anal. Chem., 32, 475-8 (1960)

24. T. Joyner et al, op. cit.

					Cr TB	Mn TB	Fe B	Co TB		Cu B	Zn TB						
							Ru T			Ag TM		In T	Sn TM				
			Hf T	Ta TM	W T		Os T	Ir T		Au TM	Hg TM	Tl T					

T = Thionalid
M = 2-Mercaptobenzimidazole
B = 5,7-Di-Bromo-8-Hydroxyquinoline

Figure 2-5: Cocrystallization by Thionalid, 2-Mercaptobenzimidazole, and 5,7-Dibromo-8-Hydroxyquinoline

possible, especially if the weight of the solid phase is measured against the weight of the aqueous sample.

For either method, in addition to the equipment normally used for the collection of aqueous samples, either a filtration setup or a setup for centrifugation and decantation is needed. Filtration is necessary for large samples; sea water samples of up to 100 liters have been treated by $\text{Mg}(\text{OH})_2$ coprecipitation.²⁵

As in the case of ion exchange, the process times may be quite long (overnight in at least one case) but many samples can be treated at once.

Both methods have been used in oceanographic work. Both have the advantage of reducing a huge aqueous sample of low stability to a small solid sample that is easily handled and stable enough to be preserved until the ship has returned to its home port.

2.2.4 Head Space Analysis

In head space analysis, an aqueous sample is treated with reagents that convert the accumulants into volatile chemical forms. The volatile species are swept from solution by aeration (if necessary), cryogenically trapped

25. T. Joyner (telephone interview).

(if necessary), and analyzed, usually by atomic absorption spectroscopy.

Head space analysis is limited to a small portion of the periodic table: Hg, As, Sb, Bi, and Se. It may be possible to apply this method to such elements as Ge, Sn, and Te in addition, but there seems little likelihood of using it on any other large family of elements. It is nonetheless a useful method since it is applicable to elements which are difficult to accumulate by other methods. The equipment is necessarily more complex for head space analysis than for the other methods discussed above for accumulating trace metals from water. In a procedure²⁶ for As, Sb, Bi, and Se, the gaseous hydrides are collected in the balloon of a gas generator and subsequently released into the burner of an atomic absorption spectrophotometer. In the standard head space procedure²⁷ for Hg, the solution is aerated to sweep out elemental Hg, and the air stream is dried and then passed into a quartz-windowed atomic absorption cell. A reaction flask, a drying flask, a pump, and tubing complete the apparatus.

The chemistry of head space analysis for inorganic

26. F. J. Schmidt and J. L. Royer, Anal. Lett., 46, 489-92 (1974).

27. W. R. Hatch and W. L. Ott, Anal. Chem., 40, 2085 (1968).

accumulants usually consists of reduction to the metal (Hg) or the hydride (AsH_3 , SeH_2 , SbH_3 , BiH_3). The reducing agent for Hg is SnSO_4 in hydroxylamine sulfate, sulfuric acid and sodium chloride. For As, Se, Sb and Bi the reducing agent is NaBH_4 added to the sample solution previously acidified with 6M HCl. For both methods, with a sample of 50 ml, detection limits of 1 ppb are attainable for Se, Hg and Bi; for As and Sb the limit is 0.1 ppb.

If it is assumed that the accumulant is removed from the aqueous solution into the same volume of vapor, a concentration factor of about 1000 by weight is obtained, based on the relative densities of water and air. For aqueous concentrations in the ppb range this is sufficient. If additional accumulation is necessary, cryogenic trapping of the vapors can provide the additional concentration factor.

This method is fairly simple to apply to the analysis of environmental samples, at least after the apparatus is set up and tested. Grab samples are collected in the field and brought back to the laboratory, and assay by head space analysis is rapid and routine enough to be run by a technician.

TABLE 2-3

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER BY COMPOUND

This table lists those organic compounds or classes of compounds which have been concentrated from water using one of the accumulation techniques which have been described.

The accumulator column lists either the solid adsorbent or liquid extractant that was used for preconcentration. The desorption or extraction medium in most cases is the solvent or temperature which was used to remove the organic compounds from a solid adsorbent.

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Alkanes and Alkenes</u>							
sym-Tetra-chloroethane	XAD-2	Chloroform Extraction	22cc/min	Distilled water	70%		Webb (1975)
sym-Tetra-chloroethane	XAD-4+8				90	GC	
n-Hexadecane					11		
sym-Tetra-chloroethane	XAD-4				90		
bis-Chloro isopropyl ether					80		
n-Hexadecane	XAD-2				90		
sym-Tetra-chloroethane	XAD-7				90		
n-Hexadecane					3		
sym-Tetra-chloroethane					90		
CHCl ₃	Chromosorb 102	Heat	5cc/min		85-93		Mieure, Dietrich (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
BrCl ₂ CH	Activated Carbon	CHCl ₃	2320 l 3 days	1ppb	22%	GC-MS SE 30 on Chromosorb W 40°-210°C	Kleopfer, Fairless (1972)
ClBr ₂ CH							
Cl ₂ CCCl ₂							
Br ₃ CH							
C ₂ Cl ₆							
<u>Alcohols and amines</u>							
Hexyl alcohol	XAD-2	Ether	30-50cc/min 1 l	pH=8	93		Junk, et al. (1974)
2-Ethyl- hexanol					99		
2-Octanol					100		
Decyl alcohol					91		
Dodecyl alcohol					93		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Hexanol	XAD-2	Ether	50cc/min 150l	pH=8	85%	GC-MS 15% Carbowax 20M on Chromosorb P 200° C	Burnham, et al. (1972)
Hexadecylamine			50cc/min 1l		94	GC-FID	Junk, et al. (1974)
α -Terpineol	XAD-4+8	Chloroform Extraction	22cc/min	Distilled water	90	GC	Webb (1975)
α -Terpineol	XAD-4				80		
2-Ethylhexanol					91		
α -Terpineol	XAD-2				81		
2-Ethylhexanol					85		
α -Terpineol	XAD-7				36		
2-Ethylhexanol					74		
α -Terpineol	XAD-8				62		
2-Ethylhexanol					79		
Methanol	Chromosorb 102	Heat	5cc/min		<5%		Mieure, Dietrich (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Ethers</u>							
bis-Chloro isopropyl ether	XAD-2	Chloroform Extraction	22cc/min		70	GC/MS	Webb (1975)
Hexyl ether		Ether	50cc/min 1L	pH=3.2 neutral	75	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
bis-Chloro isopropyl ether	XAD-4+8	Chloroform Extraction	22cc/min	Distilled Water	77	GC	Webb (1975)
bis-Chloro isopropyl ether	XAD-8				77		
Bis(2-chloro- isopropyl) ether	Activated Carbon	CHCl ₃	2320L	1ppb		GC-MS SE-30 on Chromosorb W 40°-210°C	Kleopfer, Fairless (1972)
Bis(2-chloro- isopropyl) ether							
<u>Acids</u>							
n-Heptanoic acid	XAD-1	2N NH ₄ OH	5cc/min	Seawater pH=2	90	¹⁴ C-counting	Riley, Taylor (1969)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
n-Heptadecanoic acid	XAD-1	1N KOH	5cc/min	Seawater pH=2	100	¹⁴ C-counting	Riley, Taylor (1969)
4-Ketoglutaric acid		EtOH		pH=7.6	100		
Octanoic Acid	XAD-4	Ether	50cc/min 1l	.5% HCl - H ₂ O	108	GC-FID 5% OV-1 Chromosorb W	Junk, et al. (1974)
Decanoic Acid					90		
Palmitic Acid					101		
Oleic Acid					100		
Heptanoic Acid		NH ₄ OH		pH=2	100	¹⁴ C	Riley, Taylor (1969)
Heptadecanoic Acid		NH ₄ OH	1l	pH=2	100		
4-Ketoglutaric Acid		KOH	5cc/min	pH=7.6	100		
Palmitic acid	XAD-4	Chloroform Extraction	22cc/min	Distilled water	79	GC	Webb (1975)
Palmitic acid	XAD-2				67		
Dehydroabiatic acid					94		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Palmitic acid	XAD-7	Chloroform extraction	22cc/min	Distilled water		GC	Webb (1975)
Palmitic acid	XAD-8				16		
Dehydroabietic acid	XAD-7				90		
Dehydroabietic acid	XAD-8				90		
<u>Esters</u>							
Acetophenone	XAD-2	Ether	50cc/min 1l	pH=8	92	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
Diethyl fumarate	XAD-2			pH=3.2 neutral	86		
Dibutyl fumarate					92		
Di-2-ethyl hexyl fumarate					84		
Diethyl malonate					103		
Methyl benzoate					101		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Methyl decanoate	XAD-2	Ether	50cc/min 12	pH=3.2 neutral	95%	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
Methyl octanoate					98		
Methyl palmitate					70		
Methyl salicylate					96		
Methyl methacrylate					35		
Ethyl butyrate	Extraction with CH ₂ Cl ₃	Extraction in n-heptane	2cc/min 50cc	1ppb	100	GC-MS 15% Carbowax 20M on Chromosorb P 200°C	Burnham, et al. (1972)
Di-2-ethyl hexyl adipate			23202			GC-MS .05% OV-17 on glass beads 70°-250°C	Hites (1973)
<u>Ketones and Aldehydes</u>							
2,6-Dimethyl -4-heptanone	XAD-2	Ether	50cc/min 12	pH=8	93	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
2-Undecanone	XAD-2	Ether	50cc/min	pH=8	92%	GC-FID 5% OV-1 on Chromosorb W GC	Junk, et al. (1974)
Isophorone	XAD-4	Chloroform Extraction	22cc/min	Distilled Water	86		Webb (1975)
Isophorone	XAD-2				76		
Isophorone	XAD-7				46		
Isophorone	XAD-8				47		
Methyl isobutyl ketone	Chromosorb 102	Heat	5cc/min		100	GC-FID acid-washed DMCS-treated Chromosorb-W 5% OV-1	Mieure and Dietrich (1973)
Acetone <u>Unsubstituted</u> <u>Aromatics</u>					21-42		
Naphthalene	XAD-2	Ether	50cc/min 12	pH=8	98		Junk, et al. (1974)
Biphenyl					101		
Fluorene					84		
Anthracene					83		
Acenaphthene					92		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference		
Tetrahydro- naphthalene	XAD-2	Ether	30-50cc/min 1	pH=8	62%	GC-FID acid-washed DMCS-treated Chromosorb W	Junk, et al. (1974)		
Benzene Naphthalene				100	GC-MS 5% OV-1	Burnham, et al. (1972)			
					15% Carbowax 20M on Chromosorb P 200° C				
Acenaphthylene		Ether Extraction		pH=3 Iowa wellwater	10.3 ± 2ppb		GC/MS		
Indene					18.8 ± 8ppb				
Acenaphthene					18.0 ± 2ppb				
2,2-Benzothio- phene					1.7 ± 2ppb				
Naphthalene					15ppb				
2,3-Dihydro- indene					15ppb				
sym-Tetra- chloroethane					16ppb				
Indole					pH=3.2 neutral		89%	GC-FID	Junk, et al. (1974)
Benzothiazole							100		
Quinoline							84		
Benzoxazole							92		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Naphthalene	XAD-4+8	Chloroform Extraction	22cc/min	Distilled water	90%	GC	Webb (1975)
Benzothiazole					82		
Dibenzofuran					84		
Naphthalene	XAD-4				80		
Benzothiazole					82		
Dibenzofuran					82		
Acenaphthene					81		
Naphthalene	XAD-2				79		
Benzothiazole					74		
Acenaphthene					99		
Dibenzofuran					93		
Naphthalene	XAD-7				64		
Benzothiazole					40		
Acenaphthene					72		
Dibenzofuran					73		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Naphthalene	XAD-8	Chloroform Extraction	22cc/min	Distilled water	78%	GC	Webb (1975)
Benzothiazole					53		
Acenaphthene					20		
Dibenzofuran					95		
Benzene pyridine	Chromosorb 102	Heat	5cc/min		90 46-79		Mieure, Dietrich(1973)
Anthracene	Tenax GC	Ether Extraction	3l/hr	pH 6.8-7.2 lug/l standard	71-104	GC, Electron capture, and Phosphorus detectors	Leoni, Puccetti, Grella (1975)
Perylene					95-86		
Idene					95-86		
Pyrene					98		
Fluoranthene					96		
3,4 Benzopyrene					97		
Toluene	Activated Carbon	CHCl ₃	2320l	1ppb		GC-MS SE-30 on Chromosorb W 40°-210°C	Kleopfer, Fairless (1972)
Biphenyl	Extraction with CH ₂ Cl ₃	Extraction c̄ n-heptane	2320l		46-79	GC-MS	Hites (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Substituted- Non-polar</u>							
1-Me naphthalene	XAD-2	Ether	30-50cc/min 100%	pH=8	87-93%	GC-FID acid-washed DMCS-treated ChromosorbW 5%OV-1	Junk, et al. (1974)
2-Me naphthalene			50cc/min 1%		95		
Ethyl benzene			30-50cc/min		81		
Cumene					93		
p-Cymene					92		
1-Methylnaph- thalene		Ether Extraction	50cc/min 150%	pH=3 Iowa wellwater	19.3 ± 2ppb	GC/MS	Burnham, et al. (1972)
Isopropyl Benzene					.4 ± 2ppb		
Ethyl Benzene					15ppb		
Benzyl chloride		Ether	50cc/min 1%		88%	GC-FID	Junk, et al. (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chlorobenzene	XAD-2	Ether	50cc/min 1l	pH=3.2 neutral	90%	GC-FID	Junk, et al. (1974)
Iodobenzene					81		
o-Dichloro- benzene					88		
m-Dichloro- benzene					93		
1,2,4,5-Tetra- chlorobenzene					74		
2,4-Dichloro- toluene					71		
α-o-Dichloro- toluene					96		
m-Chlorotoluene					80		
1,2,4-Tri- chlorobenzene					99		
2Methyl naphthalene	XAD-4+8	Chloroform Extraction	22cc/min	Distilled water	77	GC	Webb (1975)
2-Methyl naphthalene	XAD-4				77		
1-Methyl naphthalene					77		
2-Methyl naphthalene	XAD-2				75		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1 Methyl naphthalene	XAD-2	Chloroform Extraction	22cc/min	Distilled water	76%	GC	Webb (1975)
2 Methyl naphthalene	XAD-7				63		
1 Methyl naphthalene					64		
2 Methyl naphthalene	XAD-8				77		
1 Methyl naphthalene					80		
Xylene	Activated Carbon	CHCl ₃	23204	1ppb		GC-MS SE-30 on Chromosorb W 40°-210°C	Kleopfer, Fairless (1972)
Styrene							
Ethylbenzene							
Hexachloro- benzene							
<u>Substituted- Polar</u>							
Benzyl alcohol	XAD-2	Ether	30-50cc/min 14	pH=8	91		
Cinnamyl alcohol					85		
2-Phenoxy- ethanol					102		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Nitrobenzene	XAD-2	Ether	50cc/min 1L	pH=3.2 neutral	91%	GC-FID	Junk, et al. (1974)
o-Nitro- toluene					80		
N-Methyl- aniline					84		
Phenylene diamine			2 cc/min 50cc		98	GC-MS 15% Carbowax 20M on Chromosorb P 200°C	Burnham, et al. (1972)
TNT		Acetone Toluene	250cc/min		80 95	LC- C ₁₈ /Corasil acetonitrile- H ₂ O	Walsh, Chalk: Merritt, Jr. (1973)
Aniline	XAD-2	KOH	5cc/min	pH=7.6	100	¹⁴ C	Riley, Taylor (1969)
o-Nitrotoluene	XAD-4+8	Chloroform Extraction	22cc/min	Distilled water	83	GC	Webb (1975)
o-Nitrotoluene	XAD-4				83		
o-Nitrotoluene	XAD-2				82		
o-Nitrotoluene	XAD-7				64		
o-Nitrotoluene	XAD-8				77		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
TNT	Activated carbon	Acetone	2320L	1ppb	22%	LC-C ₁₈ / Corasil Acetonitrile H ₂ O	Walsh, Chalk, Merritt, Jr. (1973)
<u>Phenols</u>							
Phenols	XAD-1	2N KOH	5cc/min	pH=2-9	0 (not accum)	Photometric	Riley, Taylor (1969)
2,4-Dimethyl phenol	XAD-2	Ether	50cc/min 150L	pH=8	100	GC-MS 15% Carbowax 20M on Chromosorb P 200°C	Burnham, et al. (1972)
Phenol					45		
p-Nitro phenol					100		
2-Methyl phenol					100		
o-Cresol					100		
4,6-Dinitro-2- amino phenol					43		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
o-Cl phenol	XAD-4	Ether	30-50cc/min 12	.5% HCL	96%	GC-FID 5% OV-17 ChromosorbW	Junk, et al. (1974)
Phenol					40		
p-Cl phenol					95		
o-Cresol					73		
2,4,6-Cl ₃ phenol ³					99		
3,5 Xylenol					79		
1-Naphthol					91		
Phenol	XAD-7	KOH	5cc/min	pH=7.6	86	GC-MS 15% Carbowax 20 M on ChromosorbP 200°C	Burnham, et al. (1972)
Phenol	XAD-4+8	Chloroform Extraction	22cc/min	Distilled Water	46	GC	Webb (1975)
p-Cresol					68		
Phenol	XAD-2				14		
p-Cresol					44		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Pentachloro-phenol	XAD-2	Chloroform Extraction	22cc/min	Distilled Water	84%	GC	Webb (1975)
Phenol	XAD-7				19		
p-Cresol					33		
Phenol	XAD-8				29		
p-Cresol					47		
Pentachloro-phenol					77		
Phenol	Chromosorb 102	Heat	5cc/min		25-61		Mieure, Dietrich (1973)
m-Cresol					75		
o-Ethyl phenol					97		
p-Ethyl phenol					89		
Phenol	A-26 Anion Exchange Resin	4M HCl elution followed by CH ₂ Cl ₂ extraction	10-15cc/min	pH=12.0-12.5	93-95	GC-OV-17	Chriswell, et al. (1975)
o-Cresol					90-94		
p-Cresol					80-96		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
p-Chloro-phenol	A-26 Anion Exchange Resin	4M HCl elution followed by CH ₂ Cl ₂ extraction	10-15cc/min	pH=12.0-12.5	95-100%	GC-OV-17	Chriswell, et al. (1975)
4-Chloro-3-methyl phenol					95-100		
2,4,6-Tri-chloro phenol					95-102		
Pentachloro-phenol					80-89		
3,5-Dimethyl-phenol					90-95		
2 Naphthol	Activated Carbon	CHCl ₃ followed by Florisil column	2320L	1ppb	95	GC-FID 10% Carbowax 2M on Chromosorb W 210°-240°C	Eichelberger, Dresser, Longbottom (1970)
Cresols					95-100		
Dimethyl phenols					60-88		
Trimethyl phenols					88-94		
2,3,5,6-Tetra methyl phenol					90		
Chlorophenols					80-82		
Dichloro-phenol					86-100		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Trichloro-phenols	Activated Carbon	CHCl ₃ followed by Florisil column	2320l	1ppb	83-100%	GC-FID 10% Carbowax 2M on Chromosorb W 210°-240°C	Eichelberger, Dresser, Longbottom (1970)
Phenol					104		
1-Naphthol					77		
2-Naphthol					111		
o-Nitro-phenol					102		
Pentachloro-phenol	Extraction with n-hexane/ isopropanol	Extraction c̄ n-heptane	100cc	2cc conc. H ₂ SO ₄		GC-ECD QF-1 on Varaport 150°C	Rudling (1970)
<u>Ketones and Aldehydes</u>							
Benzil	XAD-1	Ether	50cc/min 1l	pH=8	91-97	GC-FID acid-washed DMCS-treated Chromosorb W 5% OV-1	Junk, et al. (1974)
Acetophenone	XAD-2				92		
Benzophenone				pH=3.2 neutral	93		
Benzil					97		
Benzaldehyde					101		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Salicyl- aldehyde	XAD-2	Ether	50cc/min 1l	pH=3.2 (neutral)	100%	GC-FID acid-washed DMCS-treated Chromosorb W	Junk, et al. (1974)
Chlorohydroxy- benzophenone	Activated carbon	Acetone	2320l	1ppb		GC-MS SE-30 on Chromosorb W 40°-210°C	Kleopfer, Fairless (1972)
<u>Ethers</u>							
Benzyl ether	XAD-2	Ether	50cc/min 1l	pH=3.2 neutral	99	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
Anisole					87		
2-Methoxy- naphthalene					97		
Phenyl ether					91		
<u>Acids</u>							
Benzene Sulfonic Acid			2cc/min	Distilled water	31	GC or UV	Webb (1975)
p-Toluene Sulfonic Acid			50cc		23		Burnham, et al. (1972)
Benzoic				pH=3.2 (neutral)	100		
Benzoic					23		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
2 Hydroxy- naphthoic	XAD-2	Ether	50cc	pH=3.2 (neutral)	39%	GC or UV	Burnham, et al. (1972)
Benzoic			50cc/min 12	.5% HCl-H ₂ O	107	GC-FID 5% OV-IM Chromosorb W	Junk, et al. (1974)
<u>Esters</u>							
Benzyl acetate				neutral	100		
Dimethoxyethyl phthalate					94		
Dimethyl phthalate					63-91		
Diethyl phthalate					92		
Dibutyl phthalate					90-101		
Di-2-ethyl- hexyl phthal- ate					88		
2-Ethylhexyl phthalate		Chloroform Extraction	22cc/min	Distilled Water	33	GC	Webb (1975)
2-Ethylhexyl phthalate	XAD-7				22		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
2-Ethylhexyl phthalate	XAD-4	Chloroform Extraction	22cc/min	Distilled Water	11%	GC	Webb (1975)
2-Ethylhexyl phthalate					13		
Diethyl phthalate	Extraction with CH_2Cl_3	Extraction \bar{c} n-heptane	2320L	1ppb		GC-MS .05% OV-17 on glass beads 70°-250°C	Hites (1973)
Di-n-butyl phthalate							
Di-octyl phthalate							
Butyl benzoate							
Diisodecyl phthalate							

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Pesticides</u>							
Lindane	XAD-1	EtOH	5cc/min 1l	pH=2 sea water	100%	¹⁴ C	Riley, Taylor (1969)
DDT					100	GC	
Endrin					75	GC	
Malathion					100	GC	
Atrazine	XAD-2	Ether	50cc/min. 1l		83	GC-FID	Junk, et al.(1974)
Lindan					95		
Aldrin					47		
Dieldrin					93		
DDT					96		
DDE	XAD-2	Ether			81		Junk, et al.(1974)
Hexachloro- benzene	Tenax GC	Ether	3l/hr	pH 6.8 - 7.2 1ug/l standard	71-91	GC, Electron Capture, and Phosphorus detectors	Leoni, Puccetti, Grella (1975)
Aldrin					42-92		
Dieldrin					94-97		
Heptachlor					88-102		
Heptachlor epoxide					96		
α -Chlordane					99		
DDT					91-109		
DDE					60-96		
TDE					93-96		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
DCBP BHC Methoxychlor Ronnell Dursban Diazinon Malathion Parathion Summition	Tenax GC	Ether	3l/hr	pH 6.8 - 7.2 1 µg/l standard	80-84% 62-105 75 88-104 89 80	GC, Electron Capture, and Phosphorus detectors	Leoni, Puccetti, Grella (1975)
Lindane Heptachlor Aldrin Heptachlor- epoxide Endrin p,p'-DDE Dieldrin o,p'-DDT p,p'-TDE p,p'-DDT PCB's	Polyurethane	hexane	30-250l/min 4l	lppb	55 50 45 68 78 80 73 68 80 84	GC-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
Lindane Heptachlor Aldrin	Polyurethane Coated c SE-30	hexane	30-250l/min 4l	lppb	84 95 80 71	GC GC-ECD 3% SE-30	Webb (1975) Uthe, Reinke, Gesser (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Heptachlor- epoxide	Polyurethane Coated \bar{c} SE-30	hexane	30-250 μ /min 4 μ	1ppb	95%	GC-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
Endrin					90		
p,p'-DDE					92		
Dieldrin					60		
o,p'-DDT					50		
p,p'-TDE					72		
p,p'-DDT					50		
Lindane	Polyurethane Coated \bar{c} DEG 5	hexane	30-250 μ /min 4 μ	1ppb	100	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
Heptachlor					87		
Aldrin					73		
Heptachlor- epoxide					89		
Endrin					95		
p,p'-DDE					88		
Dieldrin					51		
o,p'-DDT	Polyurethane coated \bar{c} QF-1	hexane	30-250 μ /min 4 μ	1ppb	47	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
p,p'-TDE					74		
p,p'-DDT					34		
Lindane					100		
Heptachlor					87		
Aldrin					80		
Heptachlor- epoxide					95		
Endrin					100		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
p,p'-DDE Dieldrin o,p'-DDT p,p'-TDE p,p'-DDT	Polyurethane coated c SE-30	hexane	30-250 l/min 4l	lppb	96% 73 72 87 69	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
Lindane Heptachlor Aldrin Heptachlor- epoxide Endrin p,p'-DDE Dieldrin o,p'-DDT p,p'-TDE p,p'-DDT	Polyurethane Coated c OV-25	hexane	30-250 l/min 4l	lppb	97 58 47 77 70 72 48 50 60 45	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)
Lindane Heptachlor Aldrin Heptachlor- epoxide Endrin p,p'-DDE	Polyurethane Coated c OV-225	hexane	30-250 l/min 4l	lppb	91 58 45 76 74 72	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Dieldrin o,p'-DDT p,p'-TDE p,p'-DDT DDT γ-BHC Heptachlor Aldrin Heptachlor epoxide Dieldrin Endrin	Polyurethane coated c OV-225 Humic Acid- Fe colloids Extraction with benzene	hexane Extraction c n-heptane	30-250 l/min 42 500cc	lppb	44% 50 64 45 80.5 x 15,000 conc 94 89 95 97 98 96	G-C-ECD 3% SE-30 14C GC	Uthe, Reinke, Gesser (1972) Poirrier, Bordelon, Laseter (1972) Konrad, Pionke, Chesters (1969)
<u>Surfactants</u> Teepol Hyamine 2839 Triton-X-100 Nonidet P80	XAD-1	EtOH	5cc/min	pH=2	100 100 100 100	Photometric	Riley, Taylor (1969)
<u>Dyes</u> Rhodamine B Methylene Blue	XAD-1	EtOH 2N HNO ₃		pH=7.6	100 100	Photometric	Riley, Taylor (1969)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Other							
Cholesterol	XAD-1	EtOH	5cc/min	pH=2	100%	Fluorimetric	Riley, Taylor (1969)
Pregnenalone				pH=2	100	¹⁴ C-counting	
Vitamin B ₂				pH=2	100	Fluorimetric	
Vitamin B ₁₂				pH=2	100	⁵⁷ C-counting	

TABLE 2-4

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER BY ACCUMULATOR

This table lists those organic compounds or classes of compounds which have been concentrated from water using one of the accumulation techniques which have been described.

The accumulator column lists either the solid adsorbent or liquid extractant that was used for preconcentration. The desorption or extraction medium in most cases is the solvent or temperature which was used to remove the organic compounds from a solid adsorbent.

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-1	n-Heptanoic acid	2N NH ₄ OH	5cc/min	Seawater pH = 2	100%	¹⁴ C-counting	Riley, Taylor (1969)
	n-Heptadecanoic acid	1N KOH		pH = 2	100		
	4-Ketoglutaric acid	EtOH		pH = 7.6	100		
	Cholesterol	EtOH		pH = 2	100	Fluorimetric	
	Pregnenalone	EtOH		pH = 2	100	¹⁴ C-counting	
	Vitamin B ₂	EtOH		2	100	Fluorimetric	
	Vitamin B ₁₂	EtOH		pH = 7.6	100	⁵⁷ Co counting	
	<u>Surfactants</u>						
	Teepol	EtOH		pH = 2	100	Photometric	
	Hyamine 2389				100		
	Triton-X-100				100		
	Nonidet P80				100		
	<u>Dyes</u>						
	Rhodamine B	EtOH		pH = 7.6	100	Photometric	
	Methylene Blue	2N HNO ₃					
	Humic Acids	2N KOH			100		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-1	Carbohydrates	2N KOH	5cc/min	pH = 2 - 9	0 (not accum)	Photometric	Riley, Taylor (1969)
	Amino acids						
	Phenols						
	Herbicides and Pesticides						
XAD-2	Lindane	EtOH	5cc/min 1%	pH = 2 sea water	100%	C ¹⁴	Riley, Taylor (1969)
	DDT				100	GC	
	Endrin				75	GC	
	Malathion				100	GC	
XAD-2	<u>Aromatics</u>	Ether	30-50cc/min	pH = 8	87-93	GC-FID acid-washed DMCS-treated Chromosorb-W 5% OV-1	Junk, et al. (1974)
	1-Me naphthalene						
	Benzil						
	Napthalene						
	2-Me Napthalene						
	Biphenyl						
	Fluorene						
	Anthracene						
	Acenaphthene						

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Hexanol	Ether	50cc/min 150 μ l		85%	GC-MS 15% Carbowax 20M on Chromosorb P 200° C	Burnham, et al. (1972)
	<u>Phenols</u>						
	2,4-Dimethyl phenol	Ether	50cc/min 150 μ l		100	GC-MS 15% Carbowax 20M on Chromosorb P 200° C	Burnham, et al. (1972)
	Phenol				45		
	p-Nitro phenol				100		
	2-Methyl phenol				100		
	o-Cresol				100		
	4,6-Dinitro-2- amino phenol				43		
	<u>Aldehydes & Ketones & Ethers</u>						
	2,6-Dimethyl -4-heptanone	Ether	50cc/min 1 μ l		93	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
	2-Undecanone				88		
	Acetophenone				92		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	<u>Aromatics</u>						
	Acenaphthylene	Ether Extraction	50cc/min 150l	pH=3 Iowa wellwater	19.3% \pm 2ppb	GC/MS	Burnham, et al. (1972)
	1-Methylnaphthalene				11.0 \pm .6ppb		
	Indene				18.8 \pm .8ppb		
	Acenaphthene				18.0 \pm 2ppb		
	2,2-Benzothio- phene				1.7 \pm .2ppb		
	Isopropyl Benzene				.4 \pm .1ppb		
	Ethyl Benzene				15ppb		
	Naphthalene				15ppb		
	2,3-Dihydro- indene				15ppb		
	Alkyl Naphthalenes				15ppb		
	bis-Chloro isopropyl ether	Chloroform Extraction	22cc/min	Distilled water	70		Webb (1975)
	sym-Tetra- chloroethane				61		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	n-Hexadecane	Chloroform Extraction	22cc/min	Distilled water	90%	GC	Webb (1975)
	α-Terpineol				81		
	Naphthalene				79		
	o-Nitrotoluene				82		
	2-Methyl naphthalene				75		
	1-Methyl naphthalene				76		
	Benzothiazole				74		
	Phenol				14		
	p-Cresol				44		
	Acenaphthene				99		
	Dibenzofuran				93		
	2-Ethylhexanol				85		
	Isophorone				76		
	Pentachloro- phenol				84		
	Palmitic acid				67		
	Dehydroabietic acid				94		
	2-Ethylhexyl phthalate				33		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Benzene Sulfonic acid	Ether	2cc/min	pH=3.2 neutral	31%	GC or UV	Burnham, et al. (1972)
	p-Toluene Sulfonic acid		50cc		23		
	Benzoic				100		
	Benzoic (-3-)				23		
	2-Hydroxynaphthoic Esters	Ether	50cc/min 12		39	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
	Benzyl acetate				100		
	Dimethoxyethyl phthalate				94		
	Dimethyl phthalate				63-91		
	Diethyl phthalate				92		
	Dibutyl phthalate				90-101		
	Di-2-ethyl-hexyl phthalate				88		
	Diethyl fumarate				86		
	Dibutyl fumarate				92		
	Di-2-ethyl-hexyl fumarate				84		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Diethyl malonate	Ether	50cc/min 1l		103%	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
	Methyl benzoate				101		
	Methyl decanoate				95		
	Methyl octanoate				98		
	Methyl palmitate				70		
	Methyl sulicylate	Ether	2cc/min 50cc		96	GC-MS 15% Carbowax 20M on Chromosorb P 200° C	Burnham, et al. (1972)
	Methyl methacrylate				35		
	Ethyl butyrate				100		
	<u>Nitrogen Compounds</u>	Ether	50cc/min 1l			GC-FID	Junk, et al. (1974)
	Hexadecylamine				94		
	Nitrobenzene				91		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Indole	Ether	50cc/min 1l		89%	GC-FID	Junk, et al. (1974)
	o-Nitro- toluene				80		
	N-Methyl- aniline				84		
	Benzothiazole				100		
	Quinoline				84		
	Isoquinoline				83		
	Benzonitrile				88		
	Benzoxazole				92		
	Phenylene diamine	Ether	2cc/min 50cc		98	GC-MS 15% Carbowax 20M on Chromosorb P 200° C.	Burnham, et al. (1972)
	TNT	Acetone Toluene	250cc/min		80 95	LC- C ₁₈ /Corasil acetonitrile- H ₂ O	Walsh, Chalk, Merritt, Jr. (1973)
	<u>Halogenated Aromatics</u>						
	Benzyl chloride	Ether	50cc/min 1l		88	GC-FID	Junk, et al. (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Chlorobenzene	Ether	50cc/min 14		90%	GC-FID	Junk, et al. (1974)
	Iodobenzene				81		
	O-Dichloro- benzene				88		
	m-Dichloro- benzene				93		
	1,2,4,5-Tetra- chlorobenzene				74		
	2,4-Dichloro- toluene				71		
	α-o Dichloro- toluene				96		
	m-Chlorotoluene				80		
	1,2,4-Tri- chlorobenzene				99		
	<u>Herbicides & Pesticides</u>	Ether					Junk, et al. (1974)
	Atrazine				83		
	Lindan				95		
	Aldrin				47		
	Dieldrin				93		
	DDT				96		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Benzophenone	Ether	50cc/min 1 $\frac{1}{2}$		93%	GC-FID 5% OV-1 on Chromosorb W	Junk, et al. (1974)
	Benzil				97		
	Benzaldehyde				101		
	Salicyl- aldehyde				100		
	Hexyl ether				75		
	Benzyl ether				99		
	Anisole				87		
	2-Methoxy- naphthalene				97		
	Phenyl ether				91		
	Methyl iso- butyl ketone	Ether	2cc/min 50 $\frac{1}{2}$		100	GC-MS 15% Carbowax 20M on Chromosorb P 200° C.	Burnham, et al. (1972)
XAD-2	DDE	Ether			81		Junk, et al. (1974)
	<u>Acids</u>						
	Octanoic	Ether	50cc/min 1 $\frac{1}{2}$.5% HCl - H ₂ O	108	GC-FID 5% OV-1M Chromosorb W	Junk, et al. (1974)
	Decanoic				90		
	Palmitic				101		
	Oleic				100		
	Benzoic				107		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-4	sym-Tetra- chloroethane	Chloroform Extraction	22cc/min	Distilled water	90%	GC	Webb(1975)
	bis-Chloro isopropyl ether				80		
	α -Terpineol				80		
	Naphthalene				80		
	o-Nitrotoluene				83		
	2-Methyl naphthalene				77		
	1-Methyl naphthalene				77		
	Benzothiazole				82		
	Phenol				38		
	p-Cresol				69		
	Acenaphthene				81		
	Dibenzofuran				82		
	2-Ethylhexanol				91		
	Isophorone				86		
	Pentachloro- phenol				84		
	Palmitic acid				79		

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Accumulator	Accumulant	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-4	Dehydroabietic acid	Chloroform Extraction	22cc/min	Distilled Water	90%	GC	Webb (1975)
	2-Ethylhexyl phthalate				11		
	<u>Phenols</u>						
XAD-4	o-Cl phenol	Ether	30-50cc/min	5% HCL	96		Junk, et al. (1974)
	Phenol		1l		40		
	p-Cl phenol				95		
	o-Cresol				73		
	2,4,6-Cl ₃ phenol				99		
	3,5 Xylenol				79		
	1-Naphthol				91		
	<u>Acids</u>						
	Heptanoic	NH ₄ OH		pH=2	100	¹⁴ C	Riley, Taylor (1969)
	Heptadecanoic	NH ₄ OH	1l	pH=2	100		
	4-Ketoglutaric	KOH	5cc/min	pH=7.6	100		
	<u>Nitrogen Compounds</u>						
XAD-7	Aniline				100	GC-MS	Burnham, et al.
	<u>Phenols</u>					15% Carbowax	(1972)
	Phenol				86	20 M on Chromosorb P	
	sym-Tetra-chloroethane	Chloroform	22cc/min	Distilled water	90	GC	Webb (1975)
	n-Hexadecane				3		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-7	α -Terpineol	Chloroform Extraction	22cc/min	Distilled water	36%	GC	Webb (1975)
	Naphthalene				64		
	o-Nitrotoluene				53		
	2-Methyl naphthalene				63		
	1-Methyl naphthalene				64		
	Benzothiazole				40		
	Phenol				19		
	p-Cresol				33		
	Acenaphthene				72		
	Dibenzofuran				73		
	2-Ethylhexanol				74		
	Isophorone				46		
	Pentachloro- phenol				83		
	Palmitic acid				12		
	Dehydroabietic acid				90		
	2-Ethylhexyl phthalate				22		
XAD-8	sym-Tetra- chloroethane	Chloroform	22cc/min	Distilled water	90	GC	Webb (1975)
	bis-Chloro isopropyl ether				77		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-8	α -Terpineol	Chloroform Extraction	22cc/min	Distilled water	62	GC	Webb (1975)
	Naphthalene				78		
	o-Nitrotoluene				77		
	2-Methyl naphthalene				77		
	1-Methyl naphthalene				80		
	Benzothiazole				53		
	Phenol				29		
	p-Cresol				47		
	Acenaphthene				20		
	Dibenzofuran				95		
	2-Ethylhexanol				79		
	Isophorone				47		
	Pentachloro- phenol				77		
	Palmitic acid				16		
	Dehydroabiatic acid				90		
	2-Ethylhexyl phthalate				13		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD- 4+8	sym-Tetra- chloroethane	Chloroform Extraction	22cc/min	Distilled water	90%	GC	Webb (1975)
	n-Hexadecane				11		
	α -Terpineol				80		
	Naphthalene				80		
	o-Nitrotoluene				83		
	2-Methyl naphthalene				77		
	1-Methyl naphthalene				79		
	Benzothiazole				82		
	Phenol				46		
	p-Cresol				68		
	Acenaphthene				81		
	Dibenzofuran				84		
	bis-Chloro isopropyl ether				77		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 102	<u>Aromatics</u>	Heat	5cc/min		90% 46-79	GC	Mieure,Dietrich (1973)
	Benzene pyridine						
	<u>Phenols</u>						
	Phenol						
	m-Cresol						
	o-Ethyl phenol						
	p-Ethyl phenol						
	<u>Ketones</u>						
	Methyl isobutyl ketone						
	Acetone						
	<u>Halogenated Aliphatics</u>						
	CHCl ₃						
	<u>Alcohols</u>						
Methanol	<5%						

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	<u>Pesticides</u>	Ether Extraction	3l/hr	pH 6.8 - 7.2 1µg/l standard	71-91%	GC, Electron Capture, and Phosphorus detectors	Leoni, Puccetti, Grella (1975)
	Hexachloro- benzene						
	Aldrin				42-92		
	Dieldrin				94-97		
	Heptachlor				88-102		
	Heptachlor epoxide				96		
	α-Chlorodene				99		
	DDT				91-109		
	DDE				60-96		
	TDE				93-96		
	DCBP				80-84		
	BHC				62-105		
	Methoxychlor				75		
	Ronnel				88-104		
	Dursban				89		
	Diazinon				80		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	Malathion	Ether Extraction	3L/hr	pH 6.8 - 7.2 1µg/L standard	71-104%	GC, Electron Capture, and Phosphorus detectors	Leoni, Puccetti, Grella (1975)
	Parathion				90-112		
	Summition				108		
	<u>Aromatics</u>						
	Anthracene				97-100		
	Perylene				86-92		
	Ideno				95-86		
	Pyrene				98		
	Fluoranthene				96		
	3,4 Benzopyrene				97		
A-26 Anion Exchange Resin	Phenol	4M HCl elution followed by CH ₂ Cl ₂ extrañtion	10-15cc/min	pH 12.0 - 12.5	93-95	GC-OV-17	Chriswell, et al.(1975)
	σ-Cresol				90-94		
	p-Cresol				80-96		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
A-26 Anion Exchange Resin	p-Chlorophenol	4M HCL elution followed by CH ₂ Cl ₂ extraction	10-15cc/min	pH 12.0 - 12.5	95-100%	GC-OV-17	Chriswell, et al. (1975)
	4-Chloro-3- methyl phenol				95-100		
	2,4,6-Tri- chloro phenol				95-102		
	Pentachloro- phenol				80-89		
	3,5-Dimethyl- phenol				90-95		
	2 Naphthol				95		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
XAD-2	Tetrahydro- naphthalene	Ether	30-50cc/min	pH=8	62%	GC-FID acid-washed DMCS-treated Chromosorb-W 5% OV-1	Junk, et al. (1974)
	Ethyl benzene				81		
	Cumene				93		
	p-Cymene				92		
	Benzene	Ether	2cc/min 50 l		100	GC-MS 15% Carbowax 20M on Chromosorb P 200° C	Burnham, et al. (1972)
	naphthalene						
	<u>Alcohols</u>	Ether	30-50cc/min 1 l				Junk, et al. (1974)
	Hexyl				93		
	2-Ethylhexanol				99		
	2-Octanol				100		
	Decyl				91		
	Dodecyl				93		
	Benzyl				91		
	Cinnamyl				85		
	2-Phenoxy- ethanol				102		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane		hexane	30-250 l/min 40	1 ppb		G-C-ECD 3% SE-30.	Uthe, Reinke, Gesser (1972)
	Lindane				55%		
	Heptachlor				50		
	Aldrin				45		
	Heptachlor- epoxide				68		
	Endrin				78		
	p,p'-DDE				80		
	Dieldrin				73		
	o,p'-DDT				68		
	p,p'-TDE				80		
	p,p'-DDT				84		
	PCB's				84	GC	Webb (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated \bar{c} DC-200	Lindane	hexane	30-250 l/min 4l.	1 ppb	97%	G-C-ECD 3% SE-30	Uthe, Reinke, & Gesser (1972)
	Heptachlor				92		
	Aldrin				92		
	Heptachlor- epoxide				100		
	Endrin				100		
	p,p'-DDE				99		
	Dieldrin				100		
	o,p'-DDT				93		
	p,p'-TDE				100		
	p,p'-DDT				98		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated C SE-30	Lindane Heptachlor Aldrin Heptachlor- epoxide Endrin p,p'-DDE Dieldrin o,p'-DDT p,p'-TDE p,p'-DDT	hexane	30-250 μ /min 40	1 ppb	95% 80 71 95 90 92 60 50 72 50	G-C-ECD 3% SE-30	Uthe, Reinke, Gesser (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated \bar{C} DEG 5	Lindane Heptachlor Aldrin Heptachlor- epoxide Endrin p,p' -DDE Dieldrin o,p' -DDT p,p' -TDE p,p' -DDT	hexane	30-250 μ /min 4l	1 ppb	100% 87 73 89 95 88 51 47 74 34	G-C-ECD 3% SE-30	Uthe, Rainke, & Gesser (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated C QF-1	Lindane	hexane	30-250 l/min 4l	1 ppb	100%	G-C-ECD 3% SE-30	Uthe, Rainke, Gesser (1972)
	Heptachlor				87		
	Aldrin				80		
	Heptachlor- epoxide				95		
	Endrin				100		
	p,p'-DDE				96		
	Dieldrin				73		
	o,p'-DDT				72		
	p,p'-TDE				87		
	p,p'-DDT				69		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated C OV-25		hexane	30-250 μ /min 4%	1 ppb		G-C-ECD 3% SE-30	Uthe, Reinke, & Gesser (1972)
	Lindane				97%		
	Heptachlor				58		
	Aldrin				47		
	Heptachlor- epoxide				77		
	Endrin				70		
	p,p'-DDE				72		
	Dieldrin				48		
	o,p'-DDT				50		
	p,p'-TDE				60		
	p,p'-DDT				45		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Polyurethane Coated \bar{c} OV-225	Lindane	hexane	30-250 l/min 4 l	1 ppb	91%	G-C-ECD 3% SE-30	Utha, Reinke & Gesser (1972)
	Heptachlor				58		
	Aldrin				45		
	Heptachlor- epoxide				76		
	Endrin				74		
	p,p'-DDE				72		
	Dieldrin				44		
	o,p'-DDT				50		
	p,p'-TDE				64		
	p,p'-DDT				45		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Activated Carbon	<u>Aromatics</u>						
	Toluene	CHCl ₃	2320 L			GC-MS SE-30 on Chromosorb-W 40°-210° C	Kleopfer, Fairless (1972)
	Xylene						
	Styrene						
	Ethylbenzene						
	<u>Phenols</u>						
	Cresols	CHCl ₃ followed by Florisil column			95-100%	GC-FID 10% Carbowax 2M on Chromosorb W 210°-240° C	Eichelberger, Dresser, Longbottom (1970)
	Dimethyl phenols				60-88		
	Trimethyl phenols				88-94		
	2,3,5,6-Tetra- methyl phenol				90		
	Chlorophenols				80-92		
	Dichloro- phenols				86-100		

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference		
Activated carbon	Trichloro- phenols	CHCl ₃ followed by Florisil column	2320 ±		83-100%	GC-FID 10% Carbowax 2M on Chromosorb W 210°-240°C	Eichelberger, Dresser, Longbottom (1970)		
	Phenol				104				
	1-Naphthol				77				
	2-Naphthol				111				
	o-Nitro- phenol				102				
	<u>Ketones & Ethers</u>	CHCl ₃						GC-MS SE-30 on Chromosorb W 40°-210° C.	Kleopfer, Fairless (1972)
	Bis(2-chloro- ethyl) ether								
	Bis(2-chloro- isopropyl) ether								
	Chlorohydroxy- benzophenone								
	<u>Nitrogen Compounds</u>								
TNT	Acetone			22	LC-C ₁₈ / Corasil Acetonitrile- H ₂ O	Walsh, Chalk, Merritt, Jr. (1973)			

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Activated Carbon	<u>Halogenated Aliphatics</u> BrCl_2CH ClBr_2CH Cl_2CCCl_2 Br_3CH C_2Cl_6	CHCl_3	2320 \pm 3 days			GC-MS SE 30 on Chromosorb W 40°-210° C	Kleopfer, Fairless (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Activated Carbon	<u>Halogenated Aromatics</u> Hexachloro- benzene	CHCl ₃	2320 l			GC-MS SE-30 on Chromosorb W 40°-210° C	Kleopfer, Fairless (1972)
Humic Acid- Fe colloids	<u>Herbicides & Pesticides</u> DDT	Extraction ♂ n-heptane			80.5 x 15,000 conc	¹⁴ C	Poirrier, Bordelon, Laseter (1972)
Extraction with CH ₂ Cl ₃	<u>Aromatics</u> Biphenyl <u>Esters</u> Diethyl phthalate Di-n-butyl phthalate Di-2-ethyl- hexyl adipate				46-79%	GC-MS GC-MS .05% OV-17 on glass beads 70°-250° C	Hites (1973) Hites (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER
ARRANGED BY ACCUMULATOR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Extraction with CH ₂ Cl ₂	Di-octyl phthalates Butyl benzoate Diisodecyl phthalate					GC-MS .05% OV-17 on glass beads 70°-250°C	Hites (1973)
Extraction with n-hexane/ isopropanol	<u>Halogenated Aromatics</u> Pentachloro- phenol		100cc	2cc conc. H ₂ SO ₄		GC-ECD QF-1 on Varaport 150° C.	Rudling (1970)
Extraction with benzene	<u>Herbicides & Pesticides</u> γ-BHC Heptachlor Aldrin Heptachlor epoxide Dieldrin Endrin		500cc		94 % 89 95 97 98 96	GC	Konrad, Pionke, Chesters (1969)

ACCUMULATION OF ORGANIC SUBSTANCES FROM WATER

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TABLE 2-5

ACCUMULATION OF METAL IONS FROM WATER

The following pages contain tables that function as a guide to the accumulator literature. The entries are arranged in alphabetical order by atomic symbol with each entry referring to a specific accumulation procedure for that metal.

Each entry contains the literature reference and identifies the procedure by type of accumulator and specific accumulator formulation (chelating agent and solvent for chelation/extraction, resin type for ion exchange, carrier for coprecipitation and cocrystallization, and reducing agent for head space analysis).

Where available, the concentration factor, applicable concentration, collection efficiency and optimum collection pH are also given. The collection efficiency is stated in terms of percent recovery and/or the standard deviation in the percent recovery. Lists of abbreviations and references are given at the end.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Ag	Chelation-extraction	APDC/MIBK				pH = 2.8	Brown, Skougstad, Fishman (1970)
Ag	Chelation-extraction	DQA/ethyl propionate				pH = 6	Sachdev and West (1970)
Ag	Chelation-extraction	Dithizone					Takeichi, et al. (1966)
Ag	Chelation-extraction	Oxine/ethyl propionate	10	.002-.25ppm		pH = 6	Sachdev and West (1970)
Ag	Chelation-extraction	Oxine/CHCl ₃	4 x 10 ⁵	.3 ppb	100	pH = 7.2	Brooks (1965)
Ag	Chelation-extraction	APDC/MIBK				pH = 2.0-2.5	Chao, Fishman, and Ball (1969)
Ag	Chelation-extraction	Dithizone/Ethyl propionate	10	.002 ppm		pH = 7.5	Sachdev and West (1969)
Ag	Ion exchange	Anionic resin					Chao, Fishman, and Ball (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Ag	Ion exchange	AG 1-X8 resin		.02-6.0 ppb	90-100	pH = 1	Chao, Fishman, and Ball (1969)
Ag	Ion exchange	Chitosan		.01 ppm	100	pH = 7.8	Muzzarelli, et al. (1969, 1970)
Ag	Chelation-extraction	DEDC/ CHCl ₃					Joyner et al. (1967)
Ag	Evaporation			.05-100 ppt	±11.7	T = 120°C	LeRoy and Lincoln (1974)
Ag	Cocrystallization	Thionalide			100	pH = 3.5-7.0	Lai and Weiss (1962)
Ag	Cocrystallization	2-Mercapto-benzimidazole			99	pH = 1 - 5	Weiss and Lai (1963)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Al	Chelation- extraction	Oxine/ MIBK				pH = 8	Fishman (1972)
Al	Chelation- extraction	DQA/ Ethyl pro- pionate			100	pH = 6	Sachdev and West (1970)
Al	Chelation- extraction	Oxine/ Ethyl pro- pionate	10	.10-10.0 ppm		pH = 6	Sachdev and West (1970)
Al	Chelation- extraction	Trifluoro- acetyl ace- tone/ CHCl ₃					Joyner et al. (1967)
Al	Chelation- extraction	Oxine/ CHCl ₃	4 x 10 ⁵	.01 ppm	100	pH = 7.2	Brooks (1965)
Al	Chelation- extraction	DQA/ benzene		.009 ppm	100	pH = 5	Hsu and Pipes (1972)
Al	Ion exchange	CPG-8-HOQ		270 ppb	100 ±3	pH = 4-9	Sugawara, Weetall, and Schucker (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Al	Filtration	HA Millipore filter; com- plex with ferron and orthophenan- throline					Joyner (1964)
Al	Evaporation			.01ppm-.2ppt	±7.3	T = 120°C	LeRoy and Lincoln (1974)
As	Chelation- Extraction	APDC/ MIBK					Mulford (1966)
As	Chelation- Extraction	DDDC/ CHCl ₃		1 ppm	103±6	pH = 2 (six hours UV photooxida- tion)	Tam (1974)
As	Chelation- Extraction	AMTH/ butanol, ethyl ace- tone and isoamyl acetate		.025 ppm		pH = 0.6-1.2	Ramakrishna, Robinson and West (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
As	Evaporation			.2-1.0 ppm	±19.2	T = 120°C	LeRoy and Lincoln (1974)
As	Head space analysis	Reduce with NaBH ₄		≥0.00015 ppb			Fernandez (1973)
Au	Ion exchange	AG 1-X8 resin		.05 ppb		pH = 1 for storage	Chao (1969)
Au	Ion exchange	Chitin		4 x 10 ⁻⁴ M	100	pH = 6 1-5 hr., distilled water	Muzzarelli and Tubertini (1969)
Au	Chelation- extraction	Dithizone/ CHCl ₃		.000004 ppm	100	pH = 7.5	Brooks (1965)
Au	Evaporation			.02-1.0 ppm	±11.7	T = 120°C	LeRoy and Lincoln (1974)
Au	Cocrystal- lization	Thionalide			98	pH = 0-7	Lai and Weiss (1962)
Au	Cocrystal- lization	2-Mercapto- benzimidazole		.069 ppb	99	pH = 1	Weiss and Lai (1963)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Ba	Chelation- Extraction	HFA/ isoamyl ace- tate		5 ppb	100	1 mg/ml K ⁺ to control ion- ization; high pH	Edelbeck and West (1970)
Ba	Ion exchange	Dowex 50W-X8 resin			91 [±] 14		Szabo and Joensuu (1967)
Ba	Ion exchange	Dowex 50-X12 Ca form		1 ppb	±0.86		Andersen and Hume (1968)
Ba	Cocrystal- lization	Potassium rhodizonate		10 ⁻¹⁴ -10 ⁻¹⁶ M	99-100	pH = 5-8	Weiss and Lai (1960)
Be	Chelation- Extraction	DQA/ ethyl pro- pionate	10	.002-.10 ppm	100	pH = 6	Sachdev and West (1970)
Be	Evaporation			.01-1.0 ppm	±18.6	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Bi	Chelation- Extraction	APDC/ MIBK				pH = 2.8	Mulford (1966)
Bi	Ion exchange	Chelating resin					Riley and Taylor (1968)
Bi	Evaporation			.01-1.0 ppm	±12.4	T = 120°C	LeRoy and Lincoln (1974)
Bi	Head space analysis	Reduce with NaBH ₄					Pollock and West (1973); Schmidt and Royer (1973); Fernandez (1973)
Ca	Ion exchange	Chelex-100= Dowex A-1		1 ppm			Blake, Bryant and Waters . (1969)
Ca	Ion exchange	Dowex 50-X10					Christova and Kruschevska (1966)
Ca	Evaporation			.01-500 ppm	+ -10.4	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Cd	Chelation- Extraction	APDC/ MIBK				pH = 2.8	Brown, Skougstad and Fishman (1970)
Cd	Chelation- Extraction	Dithizone					Takeuchi, Suzuki and Yanagisawa (1966)
Cd	Chelation- Extraction	DQA/ ethyl pro- pionate	10	.001-.1	100	pH = 6	Sachdev and West (1970)
Cd	Ion exchange	Chelating resins					Riley and Taylor (1968); Biechler (1965)
Cd	Evaporation			.01-1.0 ppm	±12.5	T = 120°C	LeRoy and Lincoln (1974)
Cd	Chelation- Extraction	Dithizone/ ethyl pro- pionate	10	.001 ppm		pH = 7.5	Sachdev and West (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Ce	Chelation-Extraction	H(FHD)/DBSO		0.2-15 µg	99±2	pH = 5.5	Burgett and Fritz (1973)
Ce	Chelation-extraction	Bis(2-ethyl hexyl)-hydrogen phosphate/n-Heptane					Joyner et al. (1967)
Ce	Cocrystallization	1-Nitroso-2-naphthol			>95	pH = 7	Joyner et al. (1967)
Co	Chelation-Extraction	APDC/MIBK				pH = 2.8	Brown, Skougstad and Fishman (1970)
Co	Chelation-Extraction	Diethyl/Dithiocarbamate					Nix and Goodwin (1970)
Co	Chelation-Extraction	Dithizone					Sachdev and West (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Co	Chelation- Extraction	Dithizone/ Ethyl pro- pionate	10	4 ppb		pH = 7.5	Sachdev and West (1969)
Co	Chelation- Extraction	DQA/ Ethyl pro- pionate	10	.004-.20 ppm	100	pH = 6	Sachdev and West (1970)
Co	Ion exchange	Chitosan		.0104 µg	100	pH = 7.8 from sea water	Muzzarelli, Raith, and Tubertini (1970)
Co	Chelation- Extraction	APDC/ MIBK					Joyner et al. (1967)
Co	Chelation- Extraction	Dithizone/ CCl ₄ or CHCl ₃		.5 ppb	100	pH = 7.5	Brooks (1965)
Co	Ion exchange	Chelating resin					Riley and Taylor (1968)
Co	Evaporation			.01-10.0ppm	±11.6	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Co	Cocrystallization	1-Nitroso-2-naphthol			100	pH = 1-9	Joyner et al. (1967)
Co	Cocrystallization	Thionalide			96	pH = 10	Lai and Weiss (1962)
Co	Cocrystallization	5,7-Dibromo-8-Hydroxy quinoline		.025 ppm	99	pH = 8	Riley and Topping (1969)
Co	Coprecipitation	KOH			97±8	pH = 6-7 From Sea Water	Joyner et al. (1967)
Co	Coprecipitation	Magnesium hydroxide		.1-100 ppb	93		Geetha and Joseph (1968)
Co	Ion exchange	CPG-8-HOQ		248 ppb	100±3	pH = 5-7	Sugawara, Weetall, and Schucker (1974)
Co	Chelation-Extraction	Dithizone/acetone		2 ppm	80±5	pH = 2-8	Matkovich and Christian (1974)
Co	Chelation Extraction	HHFA and TOPO/cyclohexane		.2 ppt	30-40	pH = 4-9	Mitchell and Ganges (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Co	Chelation-Extraction	DEDC/MIBK			100	pH = 6	Joyner et al. (1967)
Co ⁵⁸ Co ⁶⁰	Chelation-Extraction	Sodium diethyldithiocarbamate/benzene		3 x 10 ⁻¹⁴ g/ml	99.5±1.4	pH = 5.0-5.5	Motojima, Kenji, Bando, and Tamura (1967)
Cr	Chelation-Extraction	Diethyl dithiocarbamate					Nix and Goodwin (1970)
Cr	Chelation-Extraction	Add MnO ₄ ⁻ Extract: APDC/MIBK				pH = 2.8	Mulford (1966)
Cr (VI)	Chelation-Extraction	Dithizone/MIBK					Joyner et al. (1967)
Cr (VI)	Evaporation			.01-500 ppm	±9.8	T = 120°C	LeRoy and Lincoln (1974)
Cr	Cocrystallization	Thionalide			100	pH = 10	Lai and Weiss (1962)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Cr (III)	Cocrystallization	5,7-Dibromo-8-Hydroxyquinoline		.5 ppb	100	pH = 8	Riley and Topping (1969)
Cr ₂ O ₇ ⁼	Chelation-Extraction	APDC/MIBK				pH = 2.8	Mulford (1966)
Cs	Ion exchange	AMP crystals		5-50 ppm	100	pH = 2	Joyner et al. (1967)
Cs	Ion exchange	APMW crystals			100	pH ≤ 7	Krtíl and Krivy (1963)
Cs	Ion exchange	AMP		.006 ppb	97±2	pH = 6-7	Feldman and Rains (1964)
Cs	Ion exchange	Chitosan		.1 ppb	100	pH = 7	Muzzarelli and Tubertini (1969)
Cs	Cocrystallization	Ammonium dipiecryl-amine			≥ 98	pH ≤ 2-5	Joyner et al. (1967)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Cu	Chelation-Extraction	APDC/MIBK	10	2 ppb	100	pH = 2.8	Brown, Skougstad, and Fishman (1970)
Cu	Chelation-Extraction	DQA/Ethyl propionate				pH = 6	Sachdev and West (1970)
Cu	Chelation-Extraction	Diethyldithiocarbamate					Takeuchi, Suzuki, and Yanagisawa (1966)
Cu	Chelation-Extraction	Dithizone/Ethyl propionate				pH = 7.5	Sachdev and West (1969)
Cu	Ion exchange	Chelating resins					Riley and Taylor (1968); Biechler (1965)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Cu	Ion exchange	Lix-64				pH ≥ 2	Cerrai and Ghersini (1969)
Cu	Ion exchange	Chitin		4 x 10 ⁻⁴ M	100	pH = 6 (1 hr dist. water)	Muzzarelli and Tubertini (1969)
Cu	Chelation-extraction	DEDC/MIBK			100	pH = 6	Joyner et al. (1967)
Cu	Chelation-extraction	Dithizone/ CCl ₄ or CHCl ₃ DEDC/CCl ₄ or CHCl ₃ 2,2'-Diquinoline neocuproline/n-Hexanol Trifluoroacetyl acetone/ CHCl ₃ 8-Quinolinol/ CHCl ₃ APDC or Cupferon/MIBK					Joyner et al. (1967)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Cu	Solvent extraction	Dithizone CHCl ₃		.003 ppm	100	pH = 7.5	Brooks (1965)
Cu	Evaporation			.01-500 ppm	±8.0	T = 120°C	LeRoy and Lincoln (1974)
Cu	Coprecipitation	KOH			92-99	pH = 6-7	Joyner et al. (1967)
Cu	Cocrystallization	5,7-Dibromo-8-Hydroxy quinoline		5 ppb	100	pH = 8	Riley and Topping (1969)
Cu	Ion exchange	CPG-8-HOQ		39 ppb	100±3	pH = 4-7	Sugawara, Weetall, and Schucker (1974)
Cu	Chelation-Extraction	Dithizone/acetone		5 ppm	90±10	pH = 1-8	Matkovich and Christian (1974)
Cu	Chelation-Extraction	HHFA and TOPO/cyclohexane		.2 ppt	50	pH = 4-6	Mitchell and Ganges (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Eu	Chelation-Extraction	H(HFD)/DBSO		0.2-15 µg	99 [±] 2	pH = 5.5	Burgett and Fritz (1973)
Eu	Chelation-Extraction	HHFA and TOPO/cyclohexane		.002 M	25-40	pH = 1-5	Mitchell and Ganges (1974)
Fe	Chelation-Extraction	APDC/MIBK				pH = 2.5-3	U.S. Environmental Protection Agency
Fe	Chelation-Extraction	Cupferron or Oxine					Takeuchi, et al. (1966)
Fe	Chelation-Extraction	Diethyl-dithio-carbamate					Platte (1968); Nix and Goodwin (1970)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Fe	Ion exchange	Chelating resins					Biechler (1965); Galle (1971)
Fe	Chelation- extraction	DEDC/ MIBK			100	pH = 6	Joyner et al. (1967)
Fe	Chelation- extraction	Diphenyl- phenanthro- line/Isobu- tyl Alcohol Cupferron/ MIBK APDC or DEDC CHCl ₃ Trifluoro- acetone/ CHCl ₃					Joyner et al. (1967)
Fe	Filtration	HA Millipore filter				Complex with ferron and orthophenan- throline	Joyner (1964)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Fe	Evaporation			.05-500 ppm	±10	T = 120°C	LeRoy and Lincoln (1974)
Fe	Cocrystallization	1-Nitroso-2-naphthol			99	pH = 2-9	Joyner et al. (1967)
Fe	Coprecipitation	KOH			100	pH = 6-7 From Sea Water	Joyner et al. (1967)
Fe (II)	Ion exchange	Chitin		4×10^{-4} M	100	pH = 7, sea water	Muzzarell and Tubertini (1969)
Fe (II)	Chelation-Extraction	Dithizone/Acetone		4 ppm	100	pH = 0-5	Matkovich and Christian (1974)
Fe (II)	Chelation Extraction	HHFA and TOPO/cyclohexane			70±5	pH = 4-5	Mitchell and Ganges (1974)
Fe (III)	Chelation-Extraction	DQA/Ethyl propionate	10	.004-.40 ppm	100	pH = 6	Sachdev and West (1970)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Fe (III)	Cocrystallization	5-7-Dibromo-8-Hydroxyquinoline		5 ppb	98-99	pH = 8	Riley and Topping (1969)
Fe (III)	Ion exchange	CPG-8-HOQ		41 ppb	92±4	pH = 4-5	Sugawara, Weetall, and Schucker (1974)
Fe (III)	Chelation-Extraction	Dithizone/acetone		4 ppm	100	pH = 0-5	Matkovich and Christian (1974)
Fe (III)	Chelation-Extraction	HHFA and TOPO/cyclohexane			20-50	pH = 1-4	Mitchell and Ganges (1974)
Ge	Head space Analysis	Reduce with NaBH ₄					Pollock and West; Schmidt and Royer (1973)
Hf	Cocrystallization	Thionalide			99	pH = 10	Lai and Weiss (1962)
Hg	Ion exchange	Chitosan		.040 µg	100 from sea water	pH = 7.8	Muzzarelli, Raith and Tubertini (1970) Muzzarelli and Tubertini (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Hg	Ion exchange	Diethylamino-ethyl cellulose, thiocyanate form		100 µg	100	SCN Cl conc.	Kuroda, Kiriyama and Ishida (1968)
Hg	Solvent Extraction	High-molecular-weight amines (quaternary)		<1 ng	100	Extracted from brine solutions	Moore (1972)
Hg	Evaporation			.5-100 ppm	±24.6	T = 120°C	LeRoy and Lincoln (1974)
Hg	Cocrystallization	Thionalide			98-100	pH = 3.5-7	Lai and Weiss (1962)
Hg	Cocrystallization	2-Mercaptobenzimidazole			99	pH = 1-5	Weiss and Lai (1963)
Hg	Head space analysis	Helium saturated with water vapor		10 ng-10 µg	100	TiCl ₄ in H ₂ SO ₄ to reduce Hg compounds	April and Hume (1970)
CH ₃ Hg	Extraction	Benzene		<1 µg	90±4		Bisogni and Lawrence (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Hg	Head space analysis	Reduce with SnCl_2		≥ 0.0001 ppm			Hatch and Ott (1968)
In	Ion exchange	Chitosan		2.6 μg	100	pH = 7.8	Muzzarelli, Raith and Tubertini (1970)
In	Cocrystallization	Thionalide			98-100	pH = 3.5-7	Lai and Weiss (1962)
Ir	Evaporation			.07-2.0 ppm	± 14.8	T = 120°C	LeRoy and Lincoln (1974)
Ir	Cocrystallization	Thionalide			95	pH = 10	Lai and Weiss (1962)
K	Evaporation			1.-100. ppm	± 19.8	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
La	Chelation-Extraction	H(HFD)/DBSO	4 x 10 ⁵	0.2-15 µg	99±2	pH = 5.5	Burgett and Fritz (1973)
La	Chelation-extraction	Oxine/CHCl ₃		.0003 ppm	100	pH = 7.2	Brooks (1965)
Lu	Chelation-Extraction	HHFA and TOPO/cyclohexane		.002 M	20-60	pH = 1-5	Mitchell and Ganges (1974)
Mg	Chelation-Extraction	Eriochrome black T/Butyl Alcohol			±5	pH, conc., time of contact	Zolotov and Bagreev (1967)
Mg	Ion exchange	Dowex 50-X10					Christova and Krushevskaya (1966)
Mg	Evaporation			.01-20 ppm	±13.4	T = 120° C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Mn	Chelation-Extraction	APDC/MIBK				pH = 6 immediate analysis	Brown, Skougstad and Fishman (1970)
Mn	Ion exchange	Chelating Resins					Riley and Taylor (1968); Galle (1971)
Mn	Chelation-Extraction	TTA/Acetone-benzene 1-Nitroso-2-naphthol/ CHCl ₃ APDC/MIBK Cupferron/ MIBK APDC/CHCl ₃					Joyner et al. (1967)
Mn	Chelation-Extraction	DEDC/MIBK			99	pH = 6	Joyner et al. (1967)
Mn	Chelation-Extraction	Oxine/ CHCl ₃	4 x 10 ⁵	.002 ppm	100	pH = 7.2	Brooks (1965)
Mn	Evaporation			.01-5 ppm	±11.1	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Mn	Cocrystal- lization	Thionalide			96	pH = 10	Lai and Weiss (1962)
Mn	Cocrystal- lization	5,7-Dibromo-8- -Hydroxy- quinoline		5 ppb	95 (pure water) 85 (sea water)	pH = 8	Riley and Topping (1969)
Mn	Coprecipita- tion	KOH			102 \pm 4	pH = 6-7 From Sea Water	Joyner et al. (1967)
Mn	Chelation- Extraction	Dithizone/ Acetone		2 ppm	85 \pm 5	pH = 6-9	Matkovich and Christian (1974)
Mn	Chelation- Extraction	HHFA and TOPO/cyclo- hexane		.2 ppt	30	pH = 4-9	Mitchell and Ganges (1974)
Mo	Chelation- Extraction	Oxine/ MIBK				pH = 2-2.4	Chau and Lum- Shue-Chan (1969)
Mo	Ion exchange	Dowex 1-X8, SCN- or Cl- form		<10 ppb	100		Kawabuchi and Kuroda (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Mo	Chelation-Extraction	DEDC or APDC/ CHCl ₃ 8-Quinolinol/ CHCl ₃ APDC or Dithiol/MIBK α -Benzoinoxime/ CHCl ₃ 2-Amino-4-chlorobenzene-thiol hydrochloride/ CHCl ₃					Joyner et al. (1967)
Mo	Chelation-Extraction	Oxine/ CHCl ₃	4 x 10 ⁵	.01 ppm	100	pH = 7.2	Brooks (1965)
Mo	Evaporation			.02-20 ppm	\pm 9.8	T = 120°C	LeRoy and Lincoln (1974)
Mo	Cocrystallization	α -Benzoinoxime			99-100	pH = 2-5	Joyner et al. (1967)
Mo	Coprecipitation	Fe(OH) ₃		12 ppb	96.5	pH = 4.0	Kim and Zeitlin (1969)
Mo	Ion exchange	CPG-8-HOQ		212 ppb	100 \pm 3	pH = 2-5	Sugawara, Wee-tall and Schucker (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Na	Evaporation			.001-3.5 ppt	± 13.3	T = 120°C	LeRoy and Lincoln (1974)
Nd	Chelation-Extraction	H(HFD)/DBSO		.2-15 µg	99 \pm 2	pH = 5.5	Burgett and Fritz (1973)
Ni	Chelation-Extraction	APDC/MIBK				pH = 2.8	Brown, Skougstad and Fishman (1970)
Ni	Chelation-Extraction	Diethyldithiocarbamate					Takeuchi, Suzuki and Yanagisawa (1966); Nix and Goodwin (1970)
Ni	Chelation-Extraction	Oxine					Takeuchi, Suzuki and Yanagisawa (1966)
Ni	Chelation-Extraction	Dithizone					Takeuchi et al. (1966); Sachdev and West (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Ni	Chelation- Extraction	furil α -dioxime/ CHCl_3	10	.3-50 ppb	99		Wilson (1968)
Ni	Chelation- Extraction	Dithizone/ Ethyl pro- pionate	10	.004 ppm		pH = 7.5	Sachdev and West (1969)
Ni	Ion exchange	Chelating Resins					Riley and Taylor (1968); Biechler (1965); Galle (1971)
Ni	Chelation- extraction	DEDC/ MIBK			99	pH = 6	Joyner et al. (1967)
Ni	Chelation- extraction	APDC or Cupferron/ MIBK Dithizone or DEDC/ CHCl_3 Dimethylgly- oxime/ CHCl_3					Joyner et al. (1967)
Ni	Chelation- extraction	Oxine/ CHCl_3	4×10^5	.002 ppm	100	pH = 7.2	Brooks (1965)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Ni	Evaporation			.01 -50 ppm	±9.3	T = 120°C	LeRoy and Lincoln (1974)
Ni	Coprecipita- tion	KOH			98	pH = 6-7 From Sea Water	Joyner et al. (1967)
Ni	Chelation- Extraction	DQA/ Ethyl pro- pionate	10	.004-.30 ppm		pH = 6	Sachdev and West (1970)
Ni	Ion exchange	CPG-8-HOQ		248 ppb	100±3	pH = 6-7	Sugawara, Weetall and Schucker (1974)
Ni	Chelation- Extraction	Dithizone/ Acetone		2 ppm	80	pH = 1-6	Matkovich and Christian (1974)
Os	Cocrystal- lization	Thionalide			100	pH = 7-10	Lai and Weiss (1962)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
P	Chelation- Extraction	AMTH/ iso-butyl acetate		.01 ppm		pH = 0.6-1.0	Ramakrishna, Robinson and West (1969)
P	Evaporation			1-100 ppm	+5.9	T = 120°C	LeRoy and Lincoln (1974)
Pb	Chelation- Extraction	APDC/ MIBK				pH = 2.8	Brown, Skougstad and Fishman (1970)
Pb	Chelation- Extraction	Diethyl di- thiocarbamate					Takeuchi, Suzuki and Yanagisawa (1966); Platte (1968); Nix and Goodwin (1970)
Pb	Chelation- Extraction	Dithizone or Oxine					Takeuchi, Suzuki and Yanagisawa (1966)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Pb	Chelation- Extraction	Dithizone/ Ethyl pro- pionate	10	.004 µg/ml		pH = 7.5	Sachdev and West (1969)
Pb	Ion exchange	Chelating Resins					Riley and Taylor (1968); Biechler (1965); Galle (1971)
Pb	Solvent extraction of PbI ₂	KI/ MIPK		.013 ppm		EDTA added to remove inter- ferences	Chakrabarti, Robinson and West (1966)
Pb	Chelation- extraction	Oxine/ CHCl ₃	4 x 10 ⁵	.0001 ppm	100	pH = 7.2	Brooks (1965)
Pb	Chelation- extraction	Dithizone/ CHCl ₃ Diethyl- ammonium diethyl- dithio-carb- amate/CHCl ₃ NaI, dithi- zone/Iso- propyl methyl Ketone					Joyner, et al. (1967)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Pb	Evaporation			.01-5.0 ppm	±14	T = 120°C	LeRoy and Lincoln (1974)
Pb	Coprecipitation	KOH			90-95	pH = 6-7 From Sea Water	Joyner, et al. (1967)
Pb	Chelation-Extraction	DQA/ ethyl propionate	10	.005-.6 µg/ml	100	pH = 6	Sachdev and West (1970)
Pb	Adsorption	Anion-Exchange Membrane		.1 ppb		Ca inter-feres	Lochmuller, Galbraith, and Walter (1974)
Pd	Ion exchange	Chitin		4×10^{-4} M	100	pH = 6, 2hr, dist. water	Muzzarelli and Tubertini (1969)
		Chitosan			100	pH = 7	
Pd	Chelation-Extraction	Dithizone/ CHCl ₃			100	pH = 7.5	Brooks (1965)
Pd	Evaporation			.02-1.0 ppm	±9.8	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Pr	Chelation- Extraction	H(HFD)/ DBSO		.2-15 µg	99±2	pH = 5.5	Burgett and Fritz (1973)
Pt	Chelation- extraction	Dithizone/ CHCl ₃			100	pH = 7.5	Brooks (1965)
Pt	Evaporation			.05-10 ppm	±12.3	T = 120°C	LeRoy and Lincoln (1974)
Pu	Cocrystal- lization	Potassium rhodizonate		10 ⁻¹⁴ - 10 ⁻¹⁶ M	100	pH = 7-8	Weiss and Lai (1960)
Ra	Cocrystal- lization	Potassium rhodizonate		10 ⁻¹⁴ - 10 ⁻¹⁶ M	100	pH = 5-7	Weiss and Lai (1960)
Ra	Coprecipita- tion	ZnS(Ag) scin- tillation powder/Ba carrier		>1 pCi/l	68	pH = 3 T = 50°C	Kelkar and Joshi (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Rb	Ion exchange	AMP crystals		5-50 ppm	100	pH = 2	Brooks (1965)
Rb	Ion exchange	APMW crystals			100	pH \leq 7	Krt11 and Krivy (1963)
Rb	Cocrystallization	Ammonium dipicylamine			\geq 95	pH = 2-8	Joyner, et al. (1967)
Rh	Evaporation			.01-1 ppm	\pm 7.4	T = 120°C	LeRoy and Lincoln (1974)
Rn	Coprecipitation	ZnS(Ag) scintillation powder/ Ba carrier		>1 pCi/l	68	pH = 3 T = 50°C	Kelkar and Joshi (1969)
Ru	Ion exchange	Chitin and Chitosan		1.3 ppb	100	pH = 3.1 HNO ₃ added	Muzzarelli (1970)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Ru	Cocrystal- lization	Thionalide			100	pH = 10	Lai and Weiss (1962)
Ru	Evaporation			.01-5.0 ppm	±12.8	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Sb	Ion exchange	Chitosan			100	pH 7.8, from brine	Muzzarelli, Raith, and Tubertini (1970)
Sb	Evaporation			.04-1.0 ppm	±11.1	T=120°C	LeRoy and Lincoln (1974)
Sb	Head space analysis	Reduce with NaBH ₄					Pollock and West (1973); Schmidt and Royer (1973); Fernandez (1973)
Se	Chelation- extraction	APDC/ MIBK				pH=3-6	Mulford (1966)
Se	Head space analysis	NaBH ₄ as reducing agent		≥0.00015 ppm			Fernandez (1973)
Si	Chelation- extraction	AMTH and citrate/MIBK		0.01 ppm		pH=0.6-1.2	Ramakrishna, Robinson, and West (1969)
Si	Evaporation			.1-10 ppm	±10.0	T=120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Sm	Chelation- extraction	H(FHD)/DBSO	4 x 10 ⁵	.02-15 µg	99±2	pH = 5.5	Burgett and Fritz (1973)
Sn	Chelation- extraction	Oxine/ CHCl ₃		.003 ppm	100	pH = 7.2	Brooks (1965)
Sn	Evaporation			.04-1.0 ppm	±9.2	T = 120°C	LeRoy and Lincoln (1974)
Sn	Cocrystal- lization	Thionalide			96	pH = 10	Lai and Weiss (1962)
Sn	Cocrystal- lization	2-Mercapto- benzimidazole			100	pH = 5	Weiss and Lai (1963)
Sn	Head space analysis	Reduce with NaBH ₄		≥0.0002 ppm		0.2N HCl	Fernandez (1973)
Sr	Ion exchange	Dowex 50-X10					Christova and Kruschevska (1966)
Sr	Ion exchange	Dowex 50-X12 Ca form		1 ppm	±0.15		Andersen and Hume (1968)
Sr	Evaporation			.08-3.0 ppm	±8.8	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Sr	Cocrystal- lization	Potassium rhodizonate		10^{-14} - 10^{-16} M	100	pH = 5-7	Weiss and Lai (1960)
Ta	Cocrystal- lization	Thionalide			98	pH = 3.5	Lai and Weiss (1962)
Ta	Cocrystal- lization	2-Mercapto- benzimidazole			95-98	pH = 1-5	Weiss and Lai (1963)
Te	Evaporation			.1-50 ppm	±13.7	T = 120°C	LeRoy and Lincoln (1974)
Te	Head space analysis	Reduce with NaBH_4					Pollock and West (1973); Schmidt and Royer (1973); Fernandez (1973)
Ti	Ion exchange	CPG-8-HOQ		47 ppb	95±3	pH = 2-4	Sugawara, Weetall and Schucker (1974)
Ti	Evaporation			.01-1.0 ppm	±11.7	T = 120°C	LeRoy and Lincoln (1974)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Tl	Chelation- extraction	Dithizone/ CHCl ₃		.01 ppb	100	pH = 7.5	Brooks (1965)
Tl	Cocrystal- lization	Thionalide			100	pH = 10	Lai and Weiss (1962)
Tl	Chelation- extraction	APDC/MIBK				pH = 3-10	Mulford (1966)
²³³ U	Ion exchange	Chitosan		.1 ppm	94-97	pH = 5.5 (1-18 hour shaking time)	Muzzarelli, Raith, and Tubertini (1970)
UO ₂ ⁺⁺	Ion exchange	Chitosan		4 X 10 ⁻⁴ M	100	pH = 7	Muzzarelli and Tubertini (1969)
U	Cocrystal- lization	1-Nitroso-2- naphthol			>95	pH = 7-8	Joyner et al. (1967)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
V	Chelation- extraction	Cupferron/ MIBK	4 X 10 ⁵			pH = 1	Crump-Wiesner, Feltz, and Purdy (1971)
V	Chelation- extraction	Dichloro- oxine					Chau and Lum-Shue-Chan (1970)
V	Chelation- extraction	Phosphotung- state/Iso- butyl alcohol DEDC/CHCl ₃ APDC/MIBK ³					Joyner et al. (1967)
V	Chelation- extraction	Oxine/ CHCl ₃		.002 ppm	100	pH = 7.2	Brooks (1965)
V	Chelation- extraction	KCN, CyDTA, and PAR/CHCl ₃		.025 ppb	±8 at 1 ppb	pH = 6.5	Nishimura, Matsunaga, Kudo and Obara
V	Evaporation			.05-50 ppm	±8.3	T = 120°C	LeRoy and Lincoln (1974)
V(IV) and V(V)	Chelation- extraction	Cupferron/ MIBK				pH = 3.8	Crump-Wiesner and Purdy (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
V(V)	Ion exchange	CPG-8-HOQ		200 ppb	100±3	pH = 4-7	Sugawara, Weetall and Schucker (1974)
W	Ion exchange	CPG-8-HOQ		250 ppb	100±3	pH = 2-4	Sugawara, Weetall and Schucker (1974)
W	Ion exchange	Dowex 1-X8 SCN-form		.1 ppb	100		Kawabuchi and Kuroda (1969)
W	Chelation- extraction	Oxine/ CHCl ₃					Joyner et al. (1967)
W	Cocrystal- lization	Thionalide			91	pH = 3.5	Lai and Weiss (1962)
Zn	Chelation- extraction	Dithizone/ acetone		2 ppm	100	pH = 6-9	Matkovich and Christian (1974)
Zn	Chelation- extraction	HHFA and TOPO/ cyclohexane		.0028 M	.10-25	pH = 3-6	Mitchell and Ganges (1974)
Zn	Chelation- extraction	DQA/ethyl propionate	10	.001-.10ppm	100	pH = 6	Sachdev and West (1970)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concentration Factor	Applicable Concentration	% Recovery (+)	Collection Parameters	Reference
Zn	Chelation-extraction	Dithizone/ethyl propionate	10	1 ppb		pH = 7.5	Sachdev and West (1969)
Zn	Chelation-extraction	APDC/MIBK				pH = 2-6	Mulford (1966)
Zn	Chelation-extraction	Diethyl dithio carbamate					Platte (1968); Nix and Goodwin (1970)
Zn	Chelation-extraction	Dithizone					Sachdev and West (1969)
Zn	Ion exchange	Chelating resins					Riley and Taylor (1968); Blechler (1965); Galle (1961)
Zn	Ion exchange	Chitosan		0.02 µg	100	pH = 7.8	Muzzarelli, Raith, and Tubertini (1970)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Zn	Ion exchange	Chitin		0.4 ppb	100	pH = 7 distilled water	Muzzarelli and Tubertini (1969)
	Chelation- extraction	DEDC/MIBK			101	pH = 6	Joyner et al. (1967)
Zn	Chelation- extraction	Dithizone/ CHCl ₃ or CCl ₄ APDC/CHCl ₃ Oxine / CHCl ₃					Joyner et al. (1967)
Zn	Chelation- extraction	Oxine/ CHCl ₃	4 X 10 ⁵	0.01 ppm	100	pH = 7.2	Brooks (1965)
Zn	Evaporation			.01-2000ppm	±11.0	T = 120°C	LeRoy and Lincoln (1974)
Zn	Cocrystal- lization	1-Nitroso-2- naphthol			>95	pH = 7	Joyner et al. (1967)
Zn	Cocrystal- lization	Thionalide			98-100	pH = 7-10	Lai and Weiss (1962)
Zn	Cocrystal- lization	5,7-Dibromo- 8-Hydroxy- quinoline		5 ppb	100	pH = 8	Riley and Topping (1969)

* See list of Abbreviations at end of table.

ACCUMULATION OF METAL IONS FROM WATER

Accumulant	Accumulator Type	Specific* Accumulator Formulation	Concen- tration Factor	Applicable Concen- tration	% Recovery (+)	Collection Parameters	Reference
Zn	Coprecipi- tation	KOH			96±7	pH = 6-7 From Sea Water	Joyner et al. (1967)
Zr	Ion exchange	CPG-8-HOQ		135 ppb	100±5	pH = 4-6	Sugawara, Weetall, and Schucker (1974)
Zr	Evaporation			.01-1.0 ppm	±9.7	T = 120°C	LeRoy and Lincoln (1974)
Zr	Cocrystal- lization	1-Nitroso-2- naphthol			97	pH = 5	Joyner et al. (1967)

* See list of Abbreviations at end of table.

LIST OF ABBREVIATIONS USED IN TABLE 2-5

AMP	Ammonium molybdophosphate
AMTH	Ammonium molybdate tetrahydrate
APOC	Ammonium pyrrolidine dithiocarbamate
APMW	Ammonium phosphomolybdatotungstate
CPG-8-HOQ	Controlled Pore Glass with 8-Hydroxyquinoline as immobilized chelate
DBSO	Di-n-butylsulphoxide
DDDC	Diethylammonium diethyldithiocarbamate
DEDC	Sodium diethyldithiocarbamate
DQA	Dithizone, 8-Hydroxyquinoline, and acetyl acetone
HFA, HHFA	Hexafluoroacetylacetone
H(FOD)	1,1,1,2,2,6,6,7,7,7-decafluoro-3,5-heptanedione
MIBK	Methyl isobutyl ketone
MIPK	Methyl isopropyl ketone
TOPO	Tri-n-octyl-phosphine oxide
TTA	2-Thenoyltrifluoroacetone
Oxine	8-Hydroxyquinoline
Dithizone	Diphenylthiocarbazone

ACCUMULATION OF METAL IONS FROM WATER
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SECTION THREE: ACCUMULATION OF TRACE ELEMENTS
 FROM AIR

3.0 Introduction

The ambient atmosphere is an aerosol composed of nitrogen and oxygen with vapors, particulates and other gases in trace amounts. In order to analyze for most of the atmospheric gases, vapors and particulates, they must be separated from the aerosol and concentrated. The conditions for collection must be carefully controlled and measured in order to relate the mass of the trace substance accumulated to its concentration in the atmosphere. The quantitative collection of substances is complicated by the fact that the aerosol volume changes with variations in temperature and pressure.

The problem of reliable and accurate sampling is exacerbated by the dynamic nature of the atmosphere. Changes in humidity, temperature, and windspeed can also have significant effect on the collection efficiency of sampling devices. If an aqueous solution is used to collect the trace substance, change in this ambient temperature can cause freezing or significant evaporation of the solution, producing questionable results. The reliability of the sampling technique is the most critical

factor in determining the precision and accuracy of the analytical results of a given measurement.

Organic, inorganic and organo-metallic substances are found in trace gases of the atmosphere. The metallic elements are primarily found in the atmospheric particulates along with some inorganic salts such as silicates and sulfates. A significant amount of organic particulates is present in the atmosphere as a result of fossil fuel combustion. The composition of these particulates can be significantly altered by adsorption of organic gases and vapors present in the atmosphere. The atmospheric particulates demonstrate a dichotomous distribution with a separation size of approximately 2μ . The larger particulates generally emanate from mechanical sources such as suspended soil particulates from fields, or particles from rock-crushing for the production of cement. Si, Co, Fe, Cr, and Mg are found primarily in the larger particles. The smaller particulates originate principally from combustion sources, heterogenous and photochemical reaction, and condensation. The highest concentration of V, Mn, Cu, Zn, and S are observed in these particulates. Because these smaller particulates are readily inhaled, they are most significant in determining the health hazard of atmospheric particulates.

The techniques for the accumulation of substances from the atmospheric aerosol discussed in this report are based on physical, chemical, aerodynamical and electrical properties. Some of the more important properties employed in the collection of gases and particulates are absorptivity, solubility, adsorptivity, chemical reactivity, polarity, mass, inertia, boiling point, and electrical resistivity and conductivity. The methods of collection described in the report are (1) absorption; (2) adsorption; (3) condensation; (4) filtration; (5) sedimentation; and (6) electrical precipitation. These methods are best suited for the collection of different trace substances, depending on their chemical and/or physical properties. The efficiency of collection of a sampling system is normally in the range of 90 to 100 percent.

In the next section, the various collection devices for sampling trace substances in ambient air are described. For each method, the sampling device should be designed to minimize interference and contamination and to maximize reliability, precision and accuracy.

3.1 Particulate Sampling

The atmospheric aerosol is composed of particles of diverse physical and chemical composition. The particles

that readily settle out of the atmosphere (greater than 50μ) are referred to as dustfall or settleable particles. Particles in the 0.01μ to 10μ range, which can remain in the atmosphere for several weeks, are defined as suspended particles because these particles have a settling velocity comparable to the velocity of air motion. The particles of the size range 0.01μ to 0.1μ that can act as condensation sites for supersaturated vapor in the atmosphere are known as Aitken nuclei.¹

The sources of particles in the atmosphere may be industrial operations, transportation sources, natural sources, or may be formed from chemical reactions in the atmosphere involving natural or man-made gases.

Atmospheric particulates are generally classified by their equivalent particle diameter based on their aerodynamic characteristics. This method of classification of particles is necessary because of the aggregate nature of atmospheric particles, and the attendant large variation in particle density and shape. The equivalent particle diameter is measured in microns in the range $10^{-3}\mu$ to 50μ . Most methods of collection of atmospheric particulates are based on their aerodynamic or mass

¹Methods of Air Sampling and Analysis, Intersociety Committee, American Public Health Association, Washington, D.C. (1972)

properties. No change in the momentum of the particle should occur during sampling in order to get a representative sample of the aerosol. This type of sampling (isokinetic) is not required for smaller particles because they exhibit small inertial effects.

Little information is known about the chemical composition of the atmospheric aerosol. Sampled dustfall particulates usually contain high levels of iron and aluminum oxides, and calcium oxide. Suspended particles are generally considered to consist of organic compounds, metals and inorganic salts, such as sulfates, nitrates and chlorides. The organic portion of the collected particulates consists mostly of organic acids, alcohols, and other water soluble organic acids and neutral compounds.

The sampling techniques for atmospheric particulates are based on sedimentation, filtration, aerodynamic properties and precipitation. Sedimentation (the gravity settling of particulates) is relied on for the accumulation of the larger particulates. A wide range of filtration methods is available for many sizes of particulates. Aerodynamic sampling devices separate particles by utilizing their size and density differences. Examples of this technique are the cascade and Lundgren

impactors. Precipitation of particles from the atmosphere is accomplished by either thermal or electrical methods.

The collection efficiency of devices used to remove particulates from the atmosphere may be determined by their efficiency of removal of the total weight of particles or the particle count. Devices that exhibit high efficiency for removal of the total mass of particles may not efficiently remove the total number of particles.

The particulate sample may not accurately reflect the composition of the atmospheric aerosol from which it was sampled because the chemical and physical properties of the particulates can be altered after collection by condensation, adsorption, chemical reactions, and agglomeration. In the collection of sulfates by high volume samplers, the relative humidity has a significant effect on the determination of the total amount of sulfate in the sample. Also, some organic particles may be lost because they are volatile. New compounds may be formed from reactions in the collected material during sampling and storage. The size distribution of the particulates can also be changed by fragmentation and agglomeration during the sampling process.

3.1.1 Filtration

As a method for the collection of suspended particulates from the atmosphere, filtration permits great flexibility in selection of the sampling rate, sampling duration, and filter media. Filtration is a complex process based on such phenomena as inertial impaction, interception, diffusion, electrostatic attraction, gravimetric and adhesion forces, and reentrainment. The collection efficiency of specific sized particles by a medium is a function of particle size, porosity of medium, face velocity, aerosol composition, load, pH of medium, humidity and temperature. The total mass and particle count efficiencies of a filter system must be determined experimentally on the aerosol of interest.

The filter medium for atmospheric particulate sampling is selected on the basis of its collection efficiency, its inertness for reaction catalysis and the analytical techniques to be performed. The two major types of filters employed for atmospheric sampling are fiber and membrane filters. The fiber filters are prepared from either glass or cellulose. Glass fiber filters are non-hygroscopic and are generally used for gravimetric analysis. Because glass fiber filters are not contaminated with trace elements, they are usually selected when wet

chemical analyses are to be performed on the sample. The filter is almost 100% efficient for collecting the total mass of suspended particulates at high velocity sampling rates. Because of these characteristics, the glass filter has been selected as the principle filter medium in the high volume sampler used by the Environmental Protection Agency to evaluate ambient air concentrations of suspended solids and many inorganic salts and metals. But, because glass fibers will absorb sulfur dioxide and promote its catalysis to sulfate, the Environmental Protection Agency is evaluating other media for sulfate aerosol determination. Although the cellulose fiber filter has been shown to be inert in the conversion of sulfur dioxide to sulfates, it has not replaced the glass fiber filter because of its hygroscopic nature and the presence of chemical contaminants. The collection efficiency of the cellulose filter dramatically changes with variations of humidity. A wide range of collection efficiencies for cellulose fiber filters has been observed, depending on the manufacturer and the filter type. Cellulose filters are not usually selected for the filter medium if wet chemical analysis of particulates is to be performed because the binders usually interfere with the analysis. The use of either filter allows sam-

pling at high volume flow rates with low pressure drops because of their loose structure. These filters are usually employed when collection of a large mass of the aerosol is desired.

Membrane filters are thin (150 μ) with controlled pore size and usually consist of a dry, stabilized cellulose-ester-gel membrane. A wide range of sizes of membrane filters with various pore diameters is available commercially. Membrane filters are not affected by water, saturated aliphatics, and aromatics, but they are dissolved by methanol, esters and ketones. The ash content of the filter is barely traceable. The filter is thermally stable within the temperature range of the atmosphere. Membrane filters have been designed for aerosol assay at flows of 0.05 to 5.0 liters $\text{cm}^{-2} \text{min}^{-1}$ at 1 cm of mercury differential pressure. Because the particulates are trapped on the surface of the filter rather than in the interstices, the method is amenable to x-ray diffraction or fluorescence, and light microscopy techniques. This kind of filter has demonstrated nearly 100% mass removals for particulates from 0.05 to 10 μ , and has been used for collection of fine particulates from auto reactions and chemical particles in the atmosphere. Because of its relatively high resistance to flow, the filter is selected primarily for low volume flow sampling techniques.

The high volume air sampler method is the most widely used and accepted method for suspended particulate measurement in the United States. The high volume sampler uses fiber filters and can sample an aerosol at the rate of $2.12 \text{ liters min}^{-1}$, which permits the sampling of a large volume of aerosol over a relatively short period of time. An illustration of the high volume sampler is shown in Figure 3-1. This sampler has many advantages, being relatively inexpensive, rugged, and durable. With a glass fiber filter it is well suited for high volume flow sampling of particles in the 0.1 to 10μ range. The sampling method is capable of measuring wide ranges of particle concentration. The instrument has demonstrated a high level of precision, but it has been difficult to determine its accuracy. A study was performed by EPA in Texas in which twelve high volume samplers that simultaneously collected a synthetic atmosphere exhibited an average error of 3.6%. Current research is being performed to determine whether the anisokinetic sampling by the high volume sampler introduces a significant error in accuracy to the procedure. The high volume sampler has been the predominant method for collecting atmospheric particulates for chemical analysis. However, this method is limited for size analysis, because it does not provide for fractionization of composition sampled

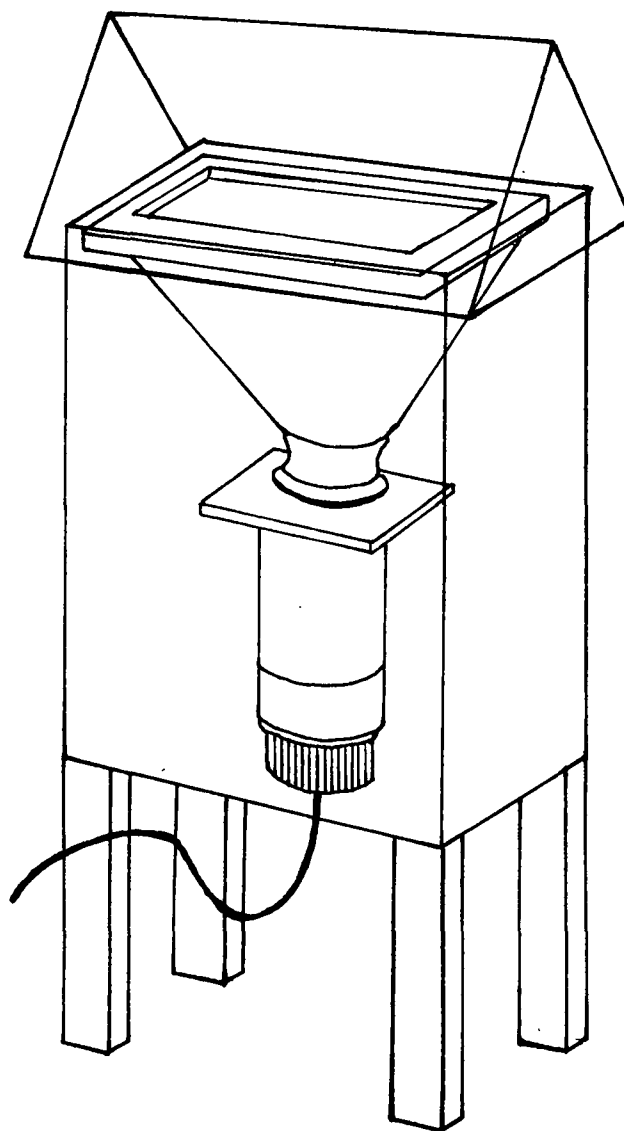


Figure 3-1: High Volume Air Sampler With Shelter

into an individual size and shape, or for the analysis of specific fractions of particles collected.

A source of error in measuring the concentration of the total suspended particulates by the high volume sample is caused by the method of estimation of sample volume flow rate. The sample volume flow rate is estimated by arithmetically averaging the initial and final flow rates for the sample period. Another source of error is the influence of humidity on the sample weight. At a relative humidity of 55%, a 40% increase in the sample weight has been observed compared to samples collected from dry air. Also, above 70% relative humidity, particulates in the range of 0.01 μ to 1 μ serve as nuclei for vapor condensation. Other sources of error in high volume sampling can be created by the volatilization of organic aerosols collected on the filter and the change in the collection efficiency of the filter with variations of volume flow rate.

Particles collected on fiber filters from the high volume sampler are usually analyzed by atomic absorption spectrophotometry and wet chemical methods which generally require many hours of sample preparation. Cellulose fiber filters and membrane filter samples have been analyzed by x-ray diffraction and fluorescence.

Bonner, et al², who analyzed various cellulose fiber filters from high volume samplers, estimated that the accuracy of this sampling and analysis system is $\pm 10\%$ and that its reproducibility is better than 5%. The x-ray fluorescence technique offers the advantage of being nondestructive, permitting further chemical analysis by x-ray diffraction or microchemical methods. Dittrich and Cothorn³ analyzed a twenty-four hour sample, collected on a glass fiber filter with a high volume sampler for Ti, Fe, Cu, Zn, Pd, Cd and Sn using x-ray fluorescence with a sensitivity limit of 0.1 microgram/ m^3 of air. Mitsugi et al⁴ analyzed glass fiber filters from low volume samplers which were operated for a month at a sample volume rate of 20 ℓ /min. The filter was molded into briquets and assayed by x-ray fluorescence for the elements of V, Mn, Ni, Cu, As, Br, Mo, Cd, Cr, Sn, Sb and Pb. The detection limits observed were 0.002 $\mu g/m^3$ for Mn, Ni, Cu, and As; 0.004 $\mu g/m^3$ for Br, Mo, Cd and Sn; 0.006 $\mu g/m^3$ for V and Pb; 0.009 $\mu g/m^3$ for Cr; and 0.013 $\mu g/m^3$ for Sb. The polynuclear aromatic

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2. Bonner, N. A., F. Bayun, and D. C. Camp. California University, Livermore, California. Lawrence Radiation Laboratory Report, Atomic Energy Commission Contract # W-7405-ENG-48.
 3. Dittrich and Colthorn. JAPCA 21: 716-719 (1971).
 4. Mitsugi, Hidikatsu, Yoshihiro Nakagawa, and Nobuhiro Takata. Institute Hyogo Project Report 5: 1-6 (1973).

hydrocarbons collected on glass fiber filters from high volume samplers can be analyzed by benzene extraction with subsequent spectrophotometric or GC analysis, or by cyclohexane extraction followed with separation by column chromatography and analysis by spectrophotometric or microanalytic methods.

A new generation of samplers is currently being developed by the Environmental Protection Agency. A prototype dichotomous sampler has been constructed⁵ which divides the aerosol sample into two fractions at a separation point of 2 μ diameter. The particles are evenly distributed over the two membrane collecting filters. The filters are in cassettes in order to permit rapid analysis by x-ray fluorescence. Thirty-six samples can be analyzed by a small computer-controlled unit for nineteen elements without requiring the operator's attention, thus producing great savings in operations cost for sample analysis.⁵ Because sulfur dioxide absorbed on small particles causes the same physiological response as the particulate sulfates, the analysis

5. Dzubay, T. C. and R. K. Stevens. Second Joint Conference of Sensing of Environmental Pollutants, Washington, D. C. December 10-12, 1973.

for elemental sulfur by x-ray fluorescence of the small particulates gives an excellent indication of the health hazard of the sulfur compounds in the aerosol.

Other methods for analysis of atmospheric particulates collected on filters are neutron activation analysis and photon activation analysis. Neutron activation analysis for trace elements of Cl, V, Mn, Cu, and Br has demonstrated reproducibilities of greater than 10%.⁶ The rare earth elements in atmospheric particulates collected by a high volume sampler on Whatman 41 cellulose filters were analyzed by Potts, et al.⁷ using neutron activation analysis. The elements La, Ce, Nd and Sm were found in the ppm range, whereas Eu, Gd, Tb, Yb and Lu were found in the sub ppm range for three American midwestern cities. Photon activation analysis has been used to measure as many as nineteen elements in a particulate sample.⁸ Elements that are readily analyzed by photon activation analysis to the submicrogram range include Pb, Ni, As, Zn, Sb, Br and I. The sensitivity of this method is

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6. Heindryckx, R. and R. Dams. "Evaluation of Three Procedures for Neutron Activation Analysis of Elements in the Atmospheric Aerosol using Short-Lived Isotopes." Radiochem., Radioanaly. Lett., 16:209-225, (1974).
 7. Potts, Mark J., Charles W. Lec, and James R. Gadiux. "Rare Earth Element." Env. Science Tech. 8:585-7 (1974).
 8. Zoller, W.H. "Photon Activation Analysis." Second Joint Sensing Environmental Pollution, Washington, D.C., December 10-12, 1973.

intermediate between neutron activation and atomic absorption. The destructive ring-oven technique can also analyze atmospheric particles collected on Whatman 41 filter paper. Substances that are measured by this method are Fe, Ni, Cu, and SO_4 ⁹, Cd¹⁰ and Pb¹¹.

Another system employing filtration to collect atmospheric particulates is the paper tape sampler. The tape sampler collects particles on a narrow strip of paper and evaluates the darkness or density of the soiled area by its reflectance or transmittance. This method has been specified for continuous monitoring of fine particulates by the American Society of Testing and Materials¹². The paper tape can be impregnated with various chemicals in order to determine the concentration of specific gases in the air. Natusch, et al.¹³ developed a method for collecting hydrogen sulfide on paper tapes impregnated with silver nitrate and determined the optical density of the metal

9. West, Philip W. and Sham L. Sachdev. "Air Pollution Studies - The Ring Oven Technique." Journal of Chemical Education, 46:96-98, (1969).
10. Dharmarajan, V. and P. W. West. "Microdetermination of Cadmium Airborne Particulates by Means of the Ring-Oven Technique." Anal. Chim Acta 57:469-72 (1971).
11. Jungreis, E. and P.W. West. "Microdetermination of Lead by the Ring-Oven Technique Applicable to Air Pollution Studies." Israel J. of Chem. 7:413-16 (1969).
12. Amer. Soc. of Testing and Materials. 1971 Annual Book of ASTM Standards, Part 23, ASTM, Phil., PA, (1971).
13. Natusch, D.F., J.R. Sewell & R.L. Tanner. "Determination of Hydrogen Sulfide in Air - Assessment of Impregnated Paper Tape Methods." Anal. Chem. 46:410-15 (1974).

sulfide formed. The sensitivity of this analytical method is in the parts per billion range. MacLeod and Lee¹⁴ determined by anodic stripping voltametry the trace elements of Cd, Pb, and Cu collected by paper tape samplers. The analytical sensitivities of anodic stripping voltametry were sufficient to characterize diurnal variation of the metals in samples collected in Chicago and Washington. The tape sampling method is not suited for air evaluation based on volumetric or mass measurement, but is used to determine the relative level of contaminant concentration.

3.1.2 Inertial Separation

Atmospheric particles can be collected according to size by utilizing their aerodynamic properties. Instruments using this principle are usually called inertial impactors or fractionators because they divide the aerosol into fractions. The removal of particles is accomplished when a stream of particle-laden air approaches a flat, solid surface in its path. As the plate is approached, the air velocity changes markedly, allowing the air to flow around the plate. The particle, however, because of its own inertia, will follow a separate path and will

14. MacLeod, Kathryn E. and R. E. Lee, Jr. "Selected Trace Metal Determination of Spot Tape Samples by Anodic Stripping Voltammetry." Anal. Chem. 45:2380 (1973).

usually impact on the surface of the plate. The collection efficiency will vary with the air stream velocity and the orientation of the plate.

Inertial impactors are classified as wet impingers or dry impactors. Wet impingers collect particles in water or some other liquid, but have such a low flow rate ($2.81 \text{ liters min}^{-1}$) that they do not usually collect sufficiently large particulate samples for trace substance analysis. Dry impactors are usually in the "cascade" configuration, which provides aerodynamic size separation of particles by decreasing the size of the impaction jets at successive stages (Figure 3-2). As the jet size is increased, successively smaller mass particles obtain sufficient velocity to impact on collection surfaces.

Common types of wet impingers used to collect gas samples are the midget and the Greenburg-Smith impinger. Wet impingers are efficient in the collection of particles greater than approximately 0.001μ diameter. The sampling volume rate for Smith-Greenburg impingers is approximately $0.028\text{m}^3/\text{min.}$, while the midget impinger is around 2.81 liters/min. Samples collected in impingers are generally analyzed by colorimetry, or any other microchemical method, or by light or atomic absorption.

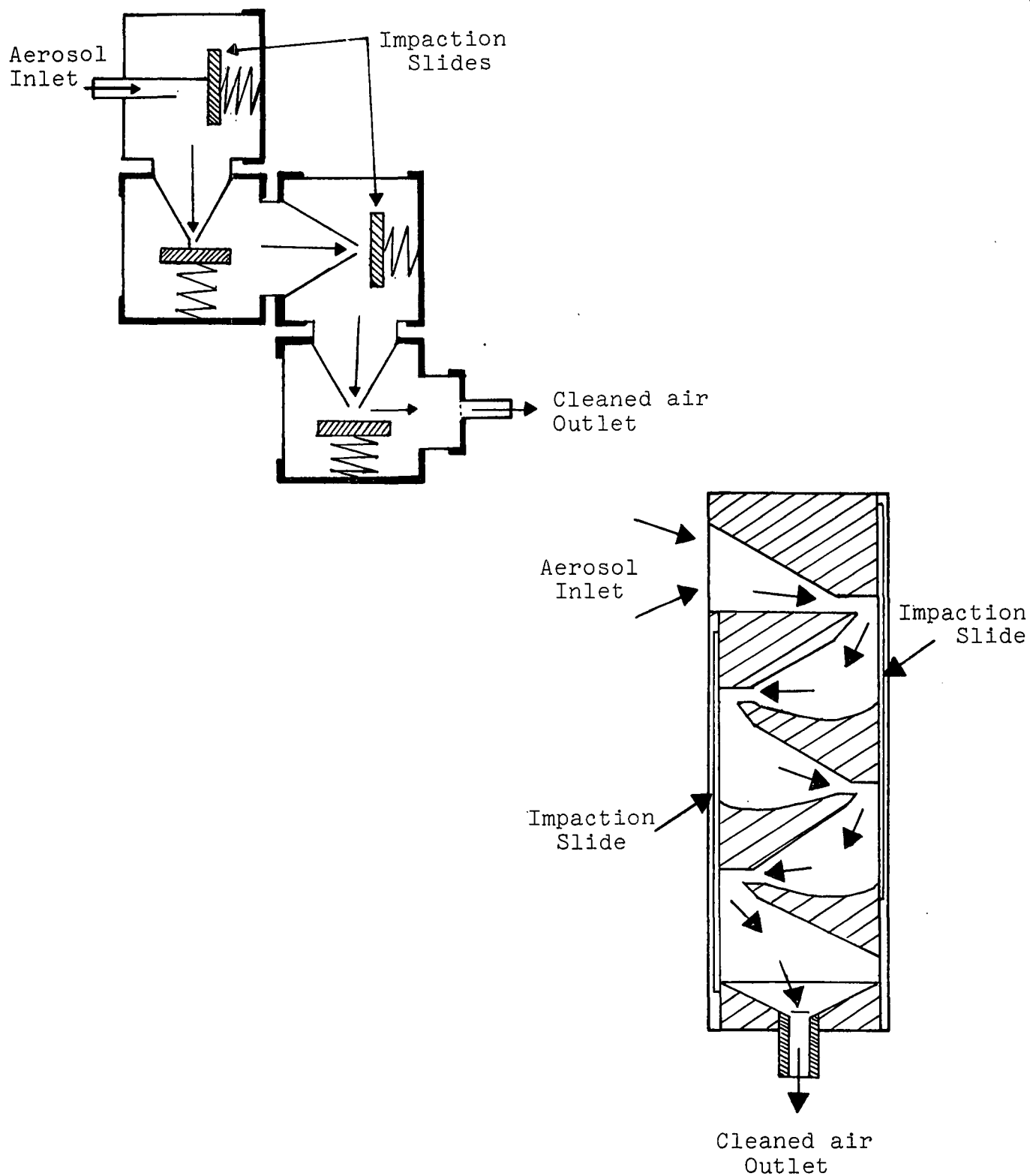


Figure 3-2. Common Cascade Impactor Designs

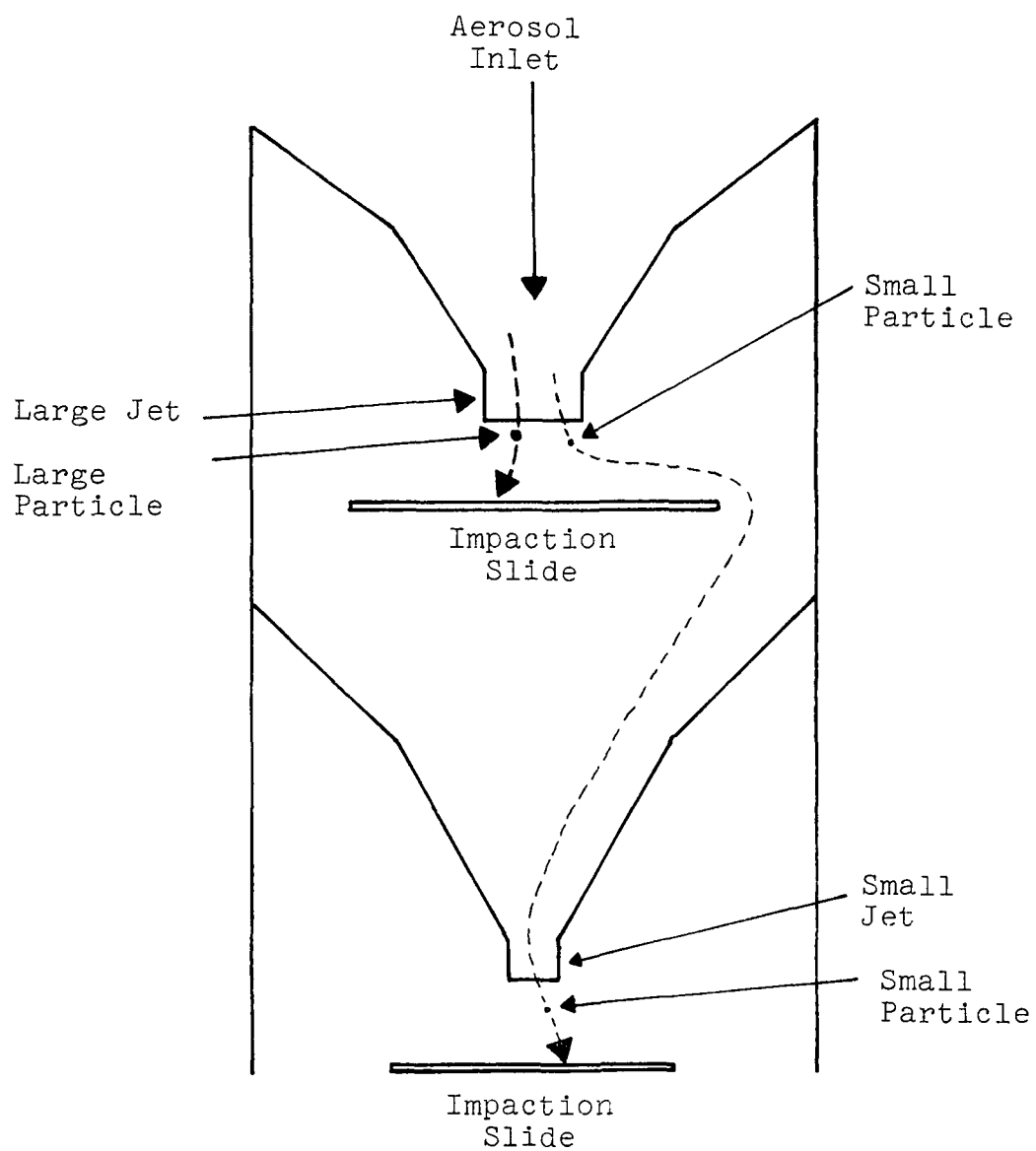


Figure 3-2 (cont'd): Common Cascade Impactor Designs

A. S. Landry¹⁵ has developed a method for the determination of atmospheric Pd and Cd, using wet impingers as collection devices. Some other trace substances that have been collected for analysis by wet impingement are As¹⁶, Be¹⁷ and Asbestos¹⁸.

Two dry inertial separators currently used for air sampling are the Andersen Sampler (Figure 3-3) and the Lundgren Impactor (Figure 3-4). The Andersen Sampler uses multijet impaction stages with progressively smaller jet openings. Particles are collected on plates on each stage, and size distribution is usually determined by gravimetric analysis of each plate. The collection surface for the Andersen Sampler may be glass or cellulose fiber filters, membrane filters of mylar, glass or teflon. The Andersen Sampler can normally operate from 28.32 liter min⁻¹ metric units to greater than 0.85 m³

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15. Landry, A.S. "The Simultaneous Determination of Lead and Zinc in Atmospheric Samples". J. Ind. Hyg. Toxic., 29:163-74 (1949).
 16. Katz, Morris. Measurement of Air Pollutants, Guide to the Selection of Methods, World Health Organization, Geneva (1969).
 17. Black, M. and R. E. Sievers. "Environmental Analysis Problems Created by Unexpected Volatile Beryllium Compounds in Various Samples." Analytical Chemistry 45:1773-1775 (1973).
 18. Homes, S. "The Measurement of Asbestos Dust." Staub Reinhaltung Luft, 33:64-66 (1973)

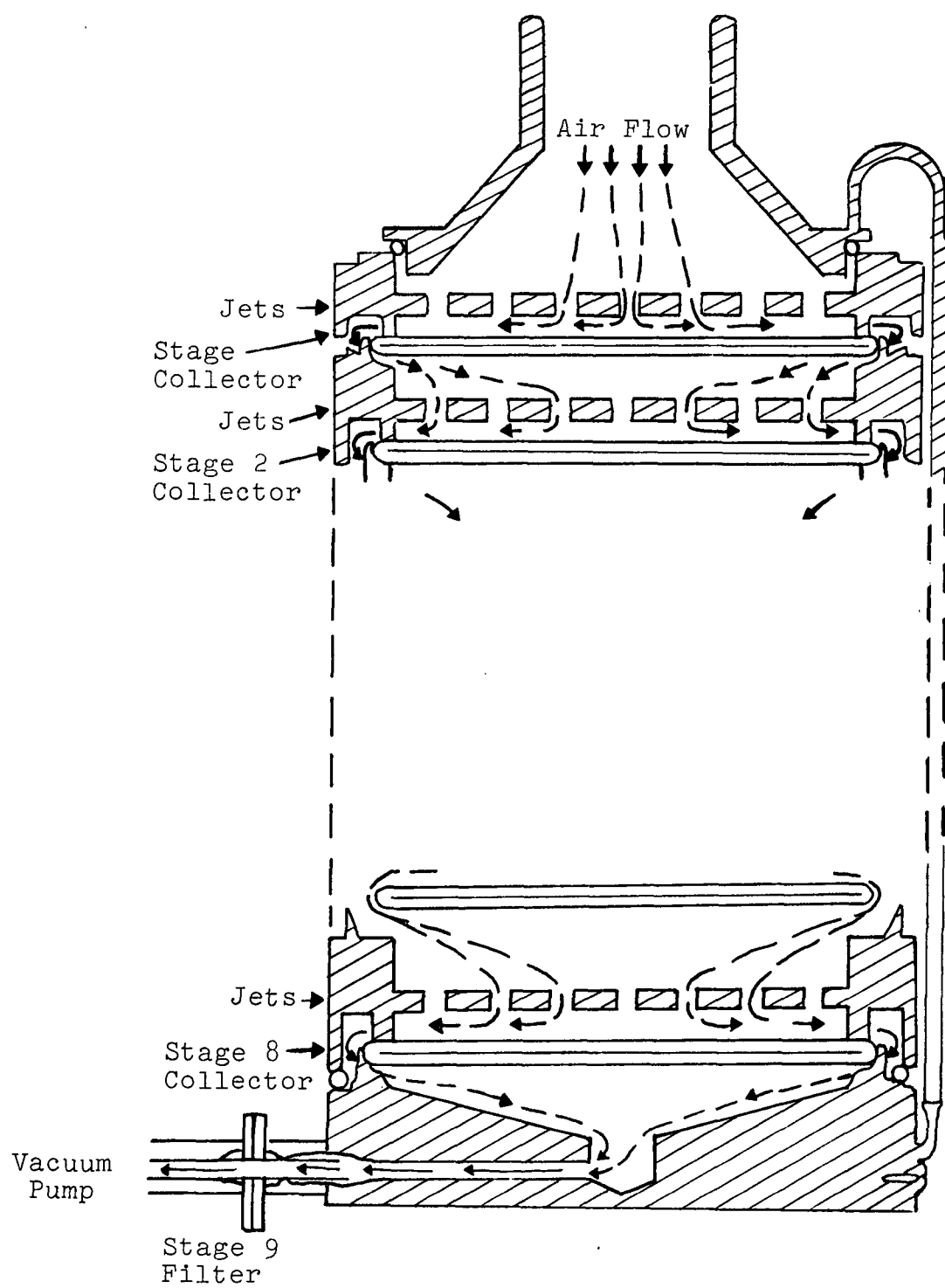


Figure 3-3: Andersen Air Sampler

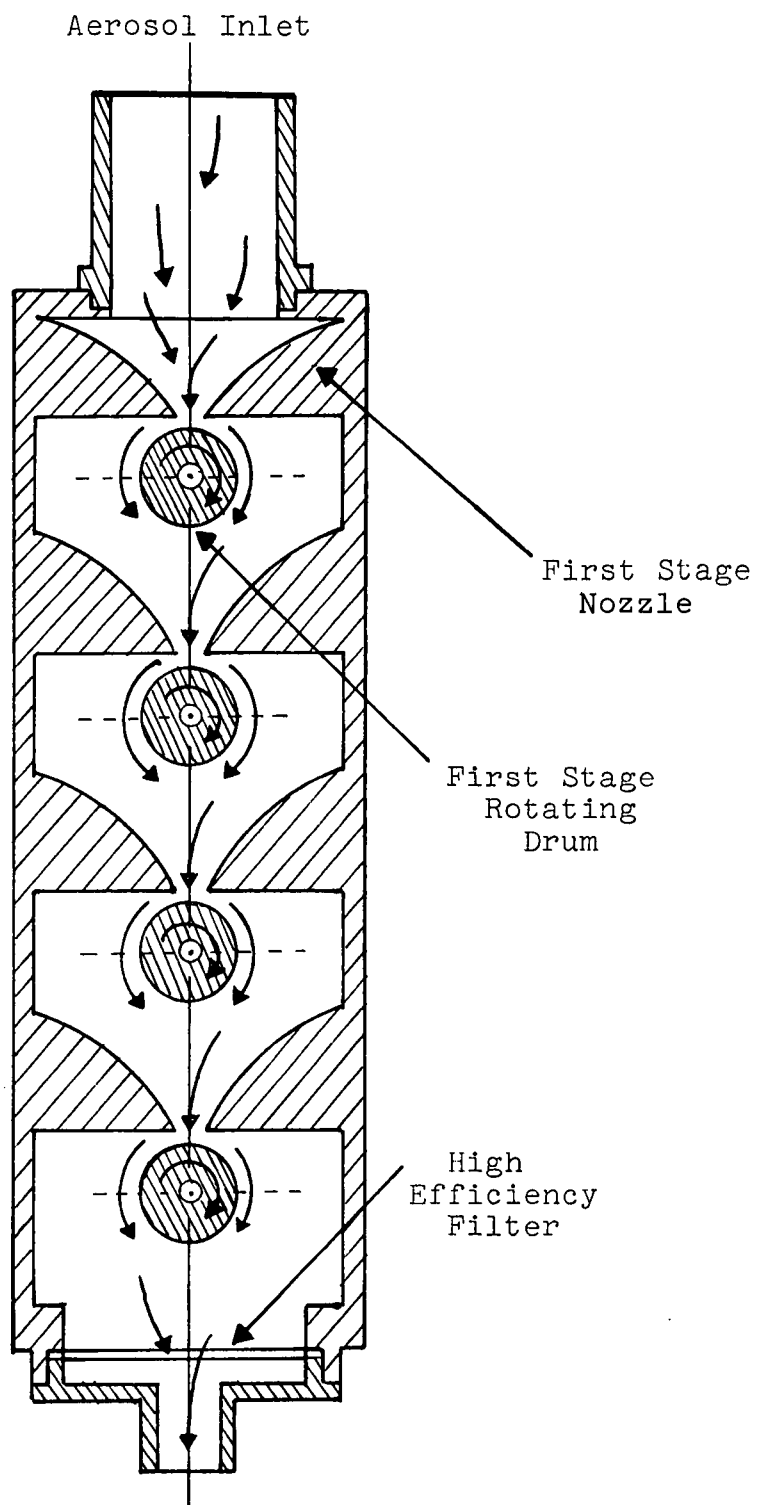


Figure 3-4: Lundgren Impactor

min⁻¹ metric units and collect particles in the size range of 0.45 to 11 μ with 95% collection efficiency. It has been widely used for studies of atmospheric size distribution and for size distribution of specific aerosols such as sulfates, lead compounds, and nitrates. A network of ten Andersen Samplers, modified to operate at 0.14 m³ min⁻¹ metric units has been used in the National Air Sampling Network (NASN).

Recently, a modified four-stage Andersen Sampler has been developed which is a high volume sampling head. The sampling head can sample at a rate of 0.57 m³ min⁻¹ metric units and fits directly onto the top of the standard high volume sample unit. It sizes particles from 1.1 μ to 7 μ and above, and if a standard high volume sampler filter is used as a back-up, particles in the range 0.1 to 1.1 μ are also collected. It was designed to be readily adaptable to routine air sampling, and field tests by EPA showed very satisfactory performance. This instrument makes it possible to sample total suspended particulates and particle size distribution at the same time. EPA is using this sizing head in connection with its current NASN operations.

Fugas, et al.¹⁹ performed simultaneous tests on a modified Andersen Sampler and a high volume sampler. The investigators found that significant amounts of particulates were lost by adhesion to the walls of the Andersen Sampler. They observed losses of suspended particles of (35%), Fe (15%), and Mg (16%). Only Pb particles were not affected by wall losses. Roesler, et al.²⁰ noted that significant conversion of SO₂ to sulfates occurred when the ambient aerosol was collected by a six-stage Andersen impactor, as compared with a high volume sampler. In six four-hour samples, the average sulfate concentration was two to three times as large as the average concentration from the high volume sampler. Because of these problems with artifact formation and wall losses, modified Andersen samplers should not be used for measuring the total suspended solids, Fe, Mg, or sulfate concentrations of an atmospheric aerosol. The same methods of analyzing particulate samples collected by the Andersen Samplers can be performed as

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19. Fugas, Mirka, J. Hrsak, and Dragica, Steiner-Slereb. "Wall Losses With the Modified Andersen Cascade Impactor." Inst. Natl. Rechn. Chim Appl. Dixieme Colloq. Atmos. Pollues., Proc., Paris, France, May 3-5, 1972.
 20. Roesler, H., J.R. Stevenson and J.S. Nader. "Size Distribution of Sulfate Aerosols in the Ambient Air". JAPCA, 15:576-79 (1965).

described in Section 3.1.1. Dams, et al.²¹ evaluated the material used on the collection surfaces of the Anderson Samplers. They found mylar and teflon to be unsatisfactory, but concluded that polyethylene is an excellent collection surface for neutron activation analysis.

The Lundgren Impactor uses rotating drums with mylar filters or foil as collection surfaces. This impactor increases the total collection surface area and permits evaluation of changes in size distribution with time. The impactor can operate from 0.014 to 0.14 m³ min⁻¹ with a normal sampling period of 24 hours. The impactor will sample particles from 0.3 to 10μ. The Lundgren Impactor has never been widely used in routine sampling, although its performance appears to be very comparable to the modified Andersen Sampler for aerosol particle size fractionation without the problems of sulfate artifact formation and large losses due to collection of particles on the walls. Blander, et al.²² used a Lundgren

21. Dams, R., K. A. Rahn, and J. W. Winchester. "Evaluation of Filter Materials and Impaction Surfaces for Nondestructive Neutron Analysis of Aerosols". Environ. Sci. Tech. 6:441-448 (1972).

22. Blander, M., P.T. Cunningham, et al. "Chemistry of Airborne Particulates." Chemical Eng. Div. Phys. Inorganic Chem. Semiannual Rept. Jan.-June, 1973, Argonne Nat'l. Laboratory, Argonne, IL (1973).

sampler to fractionate urban aerosol particles in order to analyze for Pb, Br, Fe, and Zn. From x-ray fluorescence and ion-microprobe mass analysis, the concentrations of Pb and Br were observed to decrease with increasing particle size, while Fe and Zn concentrations showed the opposite trend. In general, all analytical techniques used in the analysis of filters can be used to assay the particle distribution collected by the Lundgren sampler.

3.1.3 Electrostatic Precipitation

Electrostatic precipitators remove particles from the air or from a gas stream by imparting an electrical charge to the particles, causing them to move and adhere to a grounded or oppositely charged collection surface. This basic principle of operation is common to the different designs available. A high electrical potential difference is applied between the discharge and collecting electrodes.

A corona of charged gas ions is produced around the discharging electrode. As particles flow around the discharging electrode, they are charged by colliding with the ions. The charged particles are then attracted to the oppositely charged electrode, where they are neutralized and collected. The collecting electrode is

usually a tube or set of parallel plates surrounding the discharge electrode, which is commonly a platinum wire.

Conventional electrostatic precipitation is generally selected when a relatively large sample collection is desired for all suspended particulates particle sizes. The electrostatic precipitator offers two advantages over filtration: the sampling efficiency is not influenced by sampling rate, and the collected sample is in a readily recoverable form. The sampling rate for the electrostatic precipitator ranges from 7 to 85 liter min⁻¹, with efficiencies near 100% for particulates ranging from 0.01 μ to 10 μ . Frazer²³ evaluated the collection efficiency of an electrostatic precipitator for the collection of various particle sizes using electron microscope screens. The minimum collection efficiency was found for particles with a 2 μ diameter, with increased efficiencies exhibited for larger and smaller particulates.

The precipitation force with an electrostatic precipitator is gentle, and sample alteration through shattering of larger particulates is usually avoided.²⁴

23. Frazer, D.A. "The Collection of Submicron Particles by Electrostatic Precipitation." Am. Ind. Hyg. Quart. 17:75-79 (1956)

24. Williams, F.W. & E. Umstead. "Determination of Trace Contaminants in Air by Concentrating on Porous Polymer Beads." Anal. Chem. 40:2232-4 (1968).

The collected samples can be analyzed for metals and inorganics by the non-destructive analytical methods described in the section on Filtration (Section 3.1.1). The microchemical and other destructive methods for analyzing organics can be performed on the samples directly, or after other non-destructive analytical methods.

As particle samplers, electrostatic precipitators are extremely precise, but are only accurate in collecting particles with specific size, shape and electrical properties. Because of their high purchase and operating cost and their unreliability under field conditions, they are not often used for routine air sampling.

3.1.4 Thermal Precipitation

Thermal precipitation is based on the principle that particles under the influence of a temperature gradient will move towards a region of lower temperature. The basic components of a thermal precipitation collector are a hot wire suspended near a glass microscope slide. A steep thermal gradient is created between the heated wire and the unheated collecting surface, causing the particles to be deposited on the glass slide.

Very low flow rates (10 or $20 \text{ cm}^3 \text{ min}^{-1}$) are provided by a vacuum pump or by water displacement. Sampling

is virtually 100% efficient for the particle size range of 0.01 to 10 μ . Collection forces are gentle, so disaggregation or shattering of particles do not occur. High cost and very low flow rate make these instruments impractical for routine ambient measurements. Currently, their primary application is in the efficient collection of particles for microscopic analysis.

3.2 Gas Sampling

The primary methods employed for concentrating trace gases and vapors from the atmosphere are absorption, adsorption, and condensation. In absorption, gases or vapors are trapped by diffusing them into a liquid or reacting them with a chemical absorbing agent into a solution. Absorption is generally used for sampling gases from aerosols that can be prefiltered without causing interferences.

Adsorption occurs when a gas is trapped by a solid which holds the desired molecule with weak chemical bonds or by electrostatic forces.

Condensation is a desirable method for collecting non-reactive, insoluble gases and vapors, and non-polar hydrocarbons. A major problem with condensation is that a large amount of water vapor is generally collected along with the trace gases. Because the gases

or vapors are collected at low temperatures, reactions between the collected substances are inhibited. The preservation of the chemical composition of the sample by condensation (cryogenic trapping) is an important advantage over the other methods for collecting gases and vapors previously described.

3.2.1 Absorption

The absorbers conventionally used for air sampling are those in which the gas samples bubble through a fixed quantity of liquid. The most desirable type of collection system is chemisorption, in which gas or vapor reacts with an absorbent in solution to form a non-volatile product. In the chemisorption system, insoluble products may be formed from the reaction of the collected gas or vapor with the collecting reagent. In order to insure that significant losses of the product do not occur, special precautions must be taken.

Physical absorption, the other type of collection system, is accomplished by absorbing the gas in a liquid to form a solution with appreciable vapor pressure. Since physical absorption is reversible, the vapor pressure of the solution limits the amount of gas that can be absorbed by the liquid. Therefore, strong chemical

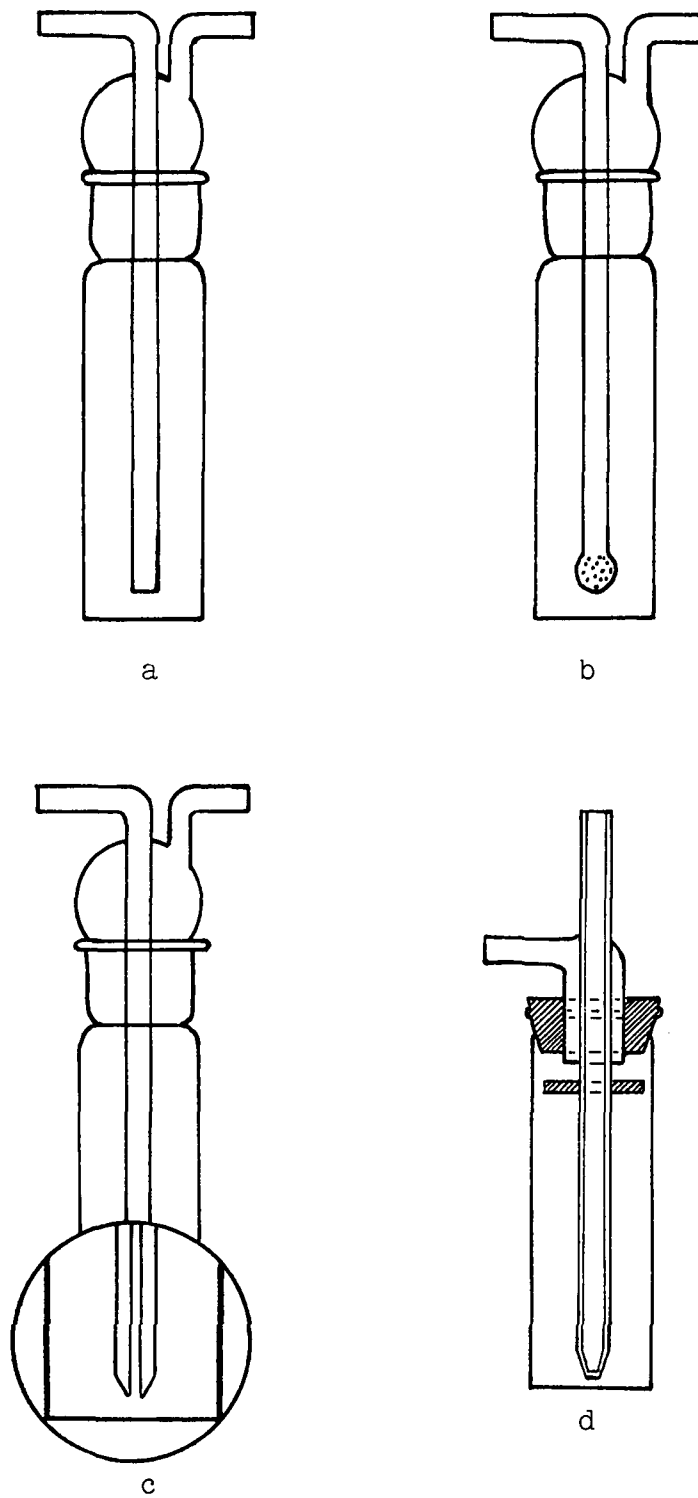
reactions are usually required in order to attain high collection efficiencies. Low collection efficiencies are usually obtained with physical absorption unless the gas or vapor to be sampled has a high solubility in the liquid and the ratio of gas to liquid volume is small. The ratio of the volumes is minimized by decreasing the bubble size and increasing the volume of collecting solution. In physical absorption systems, the formation of binary or tertiary mixtures in the absorbent solution can severely limit the system's collection efficiency. These mixtures can result in lowering of the boiling point of the collected gas, resulting in its loss by evaporation. An attempt to lower the vapor pressure of the collected gas or vapor by refrigeration may cause loss of collection efficiency by increasing the viscosity of the liquid.

Absorbers frequently employed for air sampling are:

(a) simple impingers; (b) fritted glass absorbers; (c) spiral absorbers; and (d) packed towers. Examples of these various types are illustrated in Figure 3-5.

No device will exhibit the same efficiency for all gases under all conditions. Characteristic ranges of use for some of the absorbers are displayed in Table 3-1. The following are a few questions to consider in the selection of an absorption device for gas sampling systems:

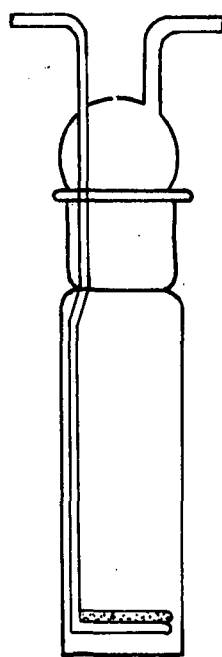
Figure 3-5: Absorption Devices



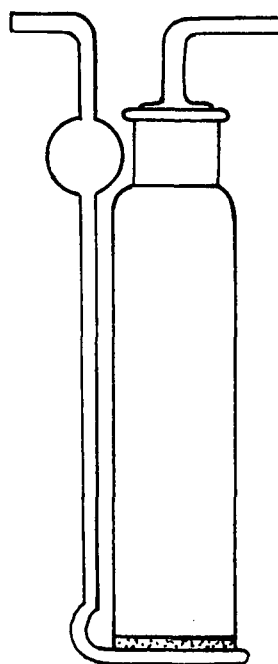
Simple Bubble Absorbers

American Public Health Association. Methods of Air Sampling and Analysis, Washington, D.C. (1972)

Figure 3-5 (cont'd): Absorption Devices

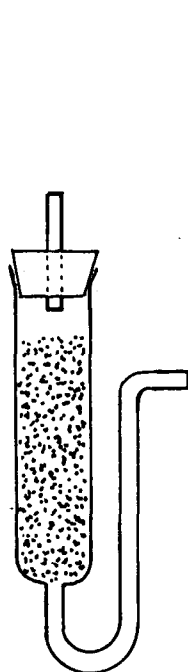


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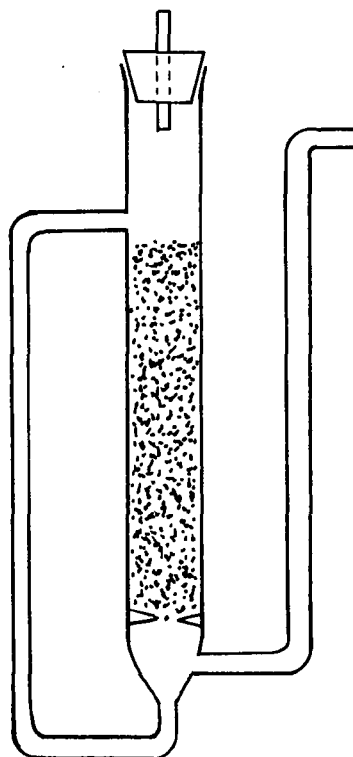


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Bubbler Absorbers with Diffusers



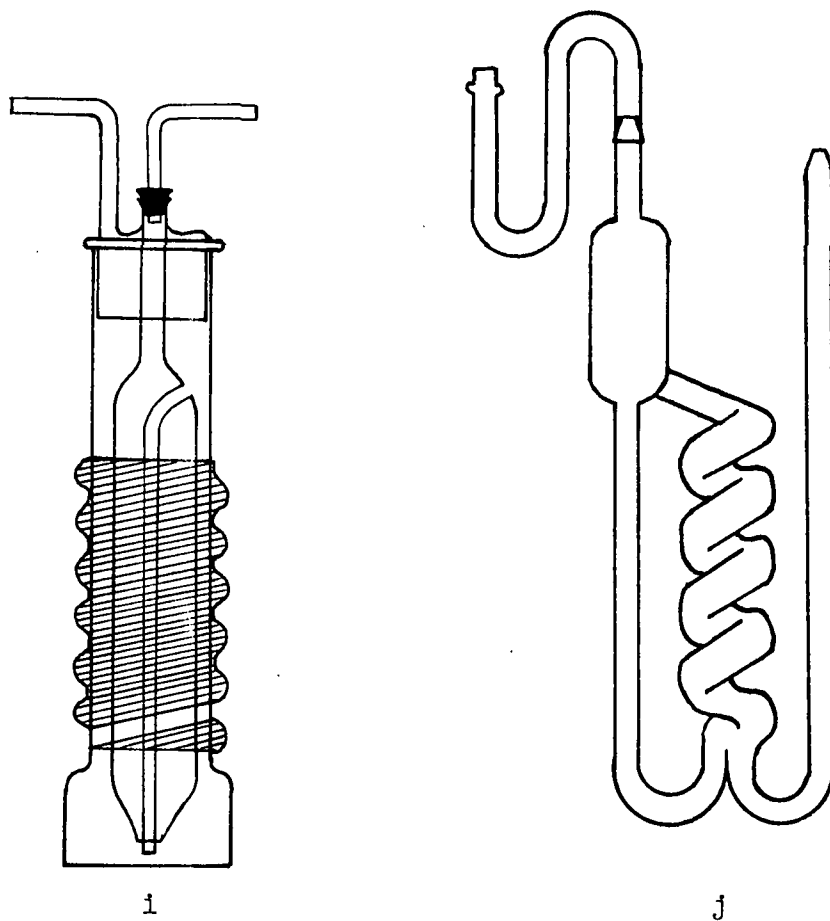
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-Bead-Packed Tower Absorbers

Figure 3-5 (cont'd): Absorption Devices



-Spiral Type Absorbers

American Public Health Association. Methods of Air Sampling and Analysis, Washington, D.C. (1972)

TABLE 3-1

CHARACTERISTICS OF ABSORBERS -- APPROXIMATE RANGE OF USE

Type of Absorber	Absorbent Capacity, ml	Sample Rate, ml per min.	Remarks
Simple Bubbler (Figure 3-5 a-d)	5 to 100	5 to 3,000	Simple, non-plugging, short gas-liquid contact.
Bubbler With Diffuser (Figure 3-5 e & f)	1 to 100	500 to 100,000	Easy to use, good gas liquid contact, subject to plugging.
Spiral (Figure 3-5 i & j)	10 to 100	40 to 500	Effective only at low flow rates.
Bead-packed Tower (Figure 3-5 g & h)	5 to 50	500 to 2,000	Efficient only at low flow rates, Resistance variable.

- 1) At the desired sampling flow rate, will a decrease in bubble size significantly increase the collection efficiency of the gas or vapor?
- 2) What amount of absorbing species is required in order to collect the expected concentration of gas or vapor?
- 3) Will sufficient contact time of the gas with the liquid exist to insure efficient collection if the selected absorber is used?

The particulates in the atmospheric aerosol may interfere with the chemical analysis that follows absorption sampling, and may cause clogging of the collection and meter devices. If prefiltration is installed for the collection of trace contaminants, any particles collected on the filter should be analyzed in order to insure quantitative analysis of sampled gas or vapor. The filter should also be non-reactive and non-absorbing to the sampled gas or vapor.

Many dusts are highly absorbent, and often carry large amounts of gaseous components in trace amounts on particles. For the analysis of lead in the air, for example, absorption without prefiltration for alkyl lead can cause gross overestimations of lead concentration because of the con-

tribution of lead from inorganic particulates. The use of a pretreatment -- such as dehydrating agents to remove moisture, soda lime for carbon dioxide, and liquid scrubbing containing oxidizing agents -- can result in large systematic errors in analysis. The use of silica based or anhydrous calcium sulfate granules for removal of water or interfering substances may partially or completely absorb the gas or vapor to be sampled as well.

Absorption is principally used for sampling inorganic atmospheric gases that are soluble in an aqueous solution. Because most absorption processes are accomplished by chemisorption, and are analyzed by colorimetric and spectrophotometric techniques, the reagent is usually selected on the basis of its efficiency in collecting the substances of interest and its ability to limit the collection of substances that would cause interferences or masking during analysis. In most cases, the absorbent reagent will trap a group of elements or compounds. The substance of interest will be selected by using its chemical or physical properties in the subsequent analytical technique. An aqueous potassium iodide solution will accumulate halogens such as Br_2 and Cl_2 from the sampled air. The specific element of analysis is then selected by precipitation during sample preparation. Table 3-2 displays some of the

specific reagents for chemisorption of a few inorganic gases.

The major difficulties in using absorption for the analysis of trace gases are the limited-volume sampling rate, the susceptibility of the solutions to freezing and evaporation, and the requirements for large amounts of sample preparation for most analytic techniques. Because the flow rates for most absorbers are in the range of a few liters per minute, high gas collection efficiencies are required in order to obtain reasonable sampling times.

3.2.2 Adsorption

Adsorption is largely a surface phenomenon. The amount of absorbate collected in the process is dependent on the total surface of adsorbent and the mass of the adsorbate. Other factors that control efficiencies of adsorption are: (1) nature of adsorbate and adsorbent; (2) geometric state of the adsorbent; (3) temperature; (4) velocity of the air stream; and (5) concentration of gas of interest and other gas in the stream.

The adsorptive capacity of most columns is from 15 to 30 percent by weight. An adsorptive bed operates at high efficiency until just before the capacity of the bed is

reached. The adsorptive capacity of an adsorbent closely parallels the critical temperature of the gas: gases with critical temperatures below -50°C and boiling points below -150°C are almost never adsorbed at normal temperature and pressure. Compounds and gases with critical temperatures between zero and 150°C are moderately adsorbable, and adsorption is not quantitative at normal pressure and temperature. The compounds and gases of low boiling point can be adsorbed only if the adsorbent is refrigerated. Heavy organic vapors (vapors with boiling points greater than 0°C) are easily adsorbed at normal temperature and pressure by activated charcoal. Most heavy vapors demonstrate increased adsorptivity with decreasing temperature.

Most common adsorbents are roughly granular in form, and are supported in columns through which air is passed. Those commonly used for air sampling are activated charcoal, silica gel, activated alumina and other active earths. The electrically polar adsorbents, such as silicous oxides, metallic oxides, and active earth compounds, attract polar gases. Lipophilic polymeric resins may be used to concentrate many organic vapors.

Since the polarity of the adsorbed compounds or elements determines the strength of their binding on alumina or silica gels, components with a higher polarity will displace those with a lesser polarity. If the adsorption

of benzene on silica gel is attempted in the presence of phenol and acetic acid in the sampled air, the adsorbent will act according to the principles of gas chromatography. The benzene previously adsorbed on adsorptive beds will be replaced by the phenol and acetic acid until the collection process is completed. Under high humidity conditions the polar adsorbent may be deactivated by saturation with water vapor. Activated charcoal is usually selected for the collection of non-polar gases and vapors from the atmosphere since it exhibits this effect to only a limited degree. On the other hand, recent studies utilizing gas chromatographic techniques have shown that precautions must be taken in the application of activated charcoal because charcoal can act as a catalyst or oxidizing agent to some gases and vapors adsorbed.

Methods employed for the desorption of the gases from adsorptive beds are heating, while driving hot air or super-heated steam through the bed, or eluting the adsorbates with a liquid solvent. Compounds desorbed from beds by steam or hot air can be condensed and collected in oil and aqueous fractions. The application of steam sometimes presents difficulties for the desorption of organic compounds because it can cause the compounds to hydrolyze, resulting in a quantitative change in the yield of the

accumulated substance. The elution of an adsorptive bed by increasing vacuum may be used for fractionization.

The tenacity of the forces which provide for quantitative extraction of the components from the air may cause great difficulty when desorption of the collected compounds is attempted. Mercury metal or volatile organic derivatives are not desorbed quantitatively either by heating or by elution with liquid reagents from silica gel. Activated charcoal, when used for lead components, demonstrates the same properties. Some non polar aromatic hydrocarbons are irreversibly adsorbed in activated charcoal.

Organic molecules from the air can be readily adsorbed on lipophilic solid supports either at ambient temperature or with cooling (see Condensation - 3.2.3). The adsorption is due to the attraction between the lipophilic adsorbent and the lipophilic adsorbate. The adsorbing resin is usually packed into a tube through which an air sample is drawn. This acts as a chromatography column with air as the eluting mobile phase. As with all such systems, an adsorbed compound will have a retention volume (of air introduced into the column) above which it begins to elute from the column. The size of the air sample which can be used to accumulate a given substance is determined by this volume, which depends upon the adsorbent, the column

temperature and the air flow rate.

The adsorbents which have been used to collect organic compounds from the air include many of the standard gas chromatography supports. In addition to these, both activated carbon and silica gel have been employed. These supports vary widely in their ability to adsorb hydrocarbons, and they are often chosen with characteristics which match the GC column in which the analysis takes place. Heat desorption into the GC or a trap (usually packed with another adsorbent and often cooled) is used almost universally, although some investigators have used solvent extractions to remove the concentrated organics from the adsorbing column. The desorption step will often limit the types of molecules which can be analyzed.

A major advantage of the lipophilic adsorbents is that they do not retain water vapor from the air and they are not affected by it. Silica gel has been used for analyses of benzene and toluene, but the air must be dried by molecular sieves before reaching the silica gel since moisture deactivates the silica. Desorption of polar organics from silica can be very difficult, making it a more limited accumulating material than many of the other adsorbents which are now in use.

Activated carbon has been used for collection of air

pollutants. Grob and Grob²⁴ in Zurich, Switzerland have made use of small traps containing about 25 milligrams of cigarette filter charcoal to collect pollutants from the Zurich atmosphere. The organic compounds were subsequently desorbed with carbon disulfide, a solvent selected for its intermediate polarity and for its low response in a flame ionization detector. Analysis of these extracts was performed with very elegant high resolution capillary column gas chromatography in combination with mass spectrometry. The compounds identified included a variety of normal alkanes, a considerable number of alkyl benzenes, and some alkyl substituted polycyclic aromatic hydrocarbons. All of these compounds could be attributed to pollution of the Zurich atmosphere by automobile exhaust.

The same authors also studied the collection efficiency of their system by using two carbon filters in series. The collection efficiency for compounds less volatile than dodecane was approximately 90%. However, for more volatile compounds the collection efficiency was 50% to 30%.

The Porapak series is another group of lipophilic adsorbents which have been used for the accumulation of

24. Grob, K. and G. Grob. "Gas-Liquid Chromatographic-Mass Spectrometric Investigation of C₆-C₂₀ Organic Compounds in an Urban Atmosphere". J. Chromatography, Vol. 62:1-13 (1971).

organic compounds from both air and water. Work by Williams and Umstead²⁵ has demonstrated the applicability of Porapak Q and S to the accumulation of small halogenated hydrocarbons from the atmosphere. The Porapak beads were 80-100 mesh packed in 6 foot by 1/4 inch stainless steel columns. Accumulation of various freons and chlorinated ethanes and ethylenes at a level of about one part per million was demonstrated for calibration samples. Precisions of about 10% and recoveries in excess of 95% were obtained with these techniques.

Another polymeric lipophilic adsorbent used in the analysis of air is Chromosorb 102. This is a high surface area, styrene-divinyl benzene copolymer which is very similar to the Porapak series. Air flow rates of approximately 4 liters per minute over collection periods of several minutes have given an analytical sensitivity of approximately 10^{-10} grams per liter of air. Satisfactory blank analyses with these materials were also carried out, indicating no contaminants in the resin.

Another lipophilic adsorbent of potential use is closely related to gas chromatographic column packings; it consists

25. Williams, F.W. and E. Umstead. "Determination of Trace Contaminants in Air by Concentrating on Porous Polymer Beads". Anal. Chem. 40:2232-4 (1968).

of support-bonded silicones such as stearic acid bonded to a silica by chemical ester bonds. W. A. Aue and Teli²⁶ have shown that these materials can accumulate organic compounds from polluted air with considerable effectiveness. In operation, the solid adsorbent is packed in a tube, air is pumped through it, the support is extracted with pentane, and the pentane extract is evaporated and analyzed by gas chromatography. The air samples analyzed included auto exhaust and chlorinated hydrocarbons, but these were laboratory studies and were not based on real environmental problems. The authors discuss two limitations of these materials. One is the occasional occurrence of artifacts, and the second is the restriction of these materials to collection of the less volatile components of the atmosphere.

J. P. Mieure and M. W. Dietrich²⁷ of the Monsanto Chemical Company in St. Louis used lipophilic adsorbants for the analysis of trace organic compounds in both air and water. They recommend the use of porous polymer bead columns of 4 to 6 inches in length, for which flow rates

26. W.A. Aue and P.M. Teli. "Sampling of Air Pollutants with Support-Bonded Chromatographic Phases." J. Chromatography , 62:15-27 (1971).

27. J.P. Mieure and W.B. Dietrich. "Determination of Trace Organics in Air and Water". J. Chrom. Sci., II:559-69 (1973).

of 1/2 to 2 liters per minute can be achieved. These short porous polymer bead columns were subsequently analyzed by insertion into either the injection port of a gas chromatograph or into the chromatographic oven directly preceeding the analytical column. These authors have used three lipophilic adsorbents:

- (1) Chromosorb 101, useful for acidic and neutral components;
- (2) Chromosorb 105, useful for low boiling components; and
- (3) Tenax GC, useful for basic, neutral and high boiling components.

They recommend the use of these three columns in parallel when attempting the analysis of an unknown air sample.

Zlatkis²⁸ has also found Tenax columns to be excellent adsorbers of volatile organic compounds from air.

The use of solid supports for the adsorption of organic molecules is even more advantageous for air analysis than it is in the analysis of aqueous samples. For quantitative results, the air volume which is pumped through the resin must be accurately measured. The sample, once collected,

28. Zlatkis, A., Lichtenstein, H.A. and A. Tishbee. Chromatographia, 6:67 (1973).

can then be easily stored in the collection column until analysis can be carried out. This form of storage is much more convenient than the use of large bags for grab samples.

More tests are needed to determine the stability of organic compounds from the air when they are adsorbed onto resins for long periods of time. It has been found, however, that very little sample loss occurs in sealed resin-filled tubes when they are stored at low temperature.

The resins used for adsorption must be carefully cleaned before they are used, but after collection there is very little sample manipulation needed in order to analyze the sample, thus minimizing the danger of contamination. Some automatic sampling and analysis devices based upon the use of a solid adsorbent have already been designed. For the analysis of most lipophilic organic molecules from air, the use of solid adsorbents appears to be preferable to cryogenic trapping, the other alternative.

A current application of adsorption for air sampling is the impregnation of filter materials of paper tape and other samplers with adsorbing reagents. Atmospheric hydrogen sulfide has been collected with a filter containing silver nitrate. Other compounds collected by this method are boron hydrides, Cl_2 , chromic acid, HF , and NH_3 . The sampling of mercury vapor by gold amalgamation has been

developed by Bullock and Kalb²⁹ for sampling source emissions containing high concentrations of sulfur dioxide. The adsorption of mercury onto silver wool has been evaluated for application for measuring ambient air concentrations of mercury vapor.³⁰ Scaringelli, et al.³¹ have advanced a method for determination of total mercury by adsorption on activated charcoal with analysis by ultra-violet spectrophotometry.

3.2.3 Condensation

Cryogenic trapping is probably the most popular method for accumulating volatile compounds from an aerosol. The basic principle involved is the lowering of the temperature far enough so that the desired accumulant is condensed into a trap. Liquid air, liquid nitrogen and dry-ice in acetone are usually employed for cooling the trap.

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29. Bullock, C. and G. W. Kalb. The Determination of Mercury in Stack Gases of High SO₂ Content by the Gold Amalgamation Technique. Trádet, Inc., Environmental Protection Agency, EPA #R2-73-153.
30. Long, S.J., et al. "Atomic Absorption Determination of Elemental Mercury Collected from Ambient Air on Silver Wool". 165th National Chemical Society Meeting, Dallas, April 8-13, 1973.
31. Scaringelli, F.P., et al. "Determination of Total Mercury in Air by Charcoal Adsorption and Ultraviolet Spectrophotometry." 165th Nat'l. Amer. Chem. Soc. Meeting, Dallas, April 8-13, 1973.

The choice of cooling temperature depends upon the volatility of the desired compound as well as the volatilities of the major components of a given air sample.

A major difficulty with cryogenic trapping is the large amount of water in most atmospheric samples (1 l of air at 25°C and 50% relative humidity contains 11 mg. of water vapor). Accumulated organic trace compounds are thus swamped by the volume of condensed water in the sample and usually must be extracted from it with an organic solvent. When liquid N₂ or liquid air are used, CO₂, O₂, and sometimes N₂ condenses in the trap. This problem can be overcome by lowering the pressure across the trap or by allowing these gases to escape as the sample slowly warms. Water vapor and CO₂ can also be adsorbed before trapping with molecular sieves and ascarite, respectively.

A second problem with cryogenic trapping involves the formation of a micro-fog which passes through the trap in the air stream and causes loss of a portion of the sample. The use of packing materials in a trap or filters before the outlet reduces this problem, but Kaiser³² has found that a 4 mm column of molecular sieves cooled with liquid N₂ at

32. Kaiser, R.E. Anal. Chem., 45:965 (1973).

a flow rate of 2ℓ/hr. has a constant loss of about 10 ppb of hydrocarbons. Kaiser has developed a temperature gradient trap which cools the sample gradually, which he believes has eliminated this problem for packed traps.

The types of traps used for the collection of organic matter vary widely. The simplest are either thin metal tubes, usually coiled, which run through a Dewar containing the liquid N₂ or stoppered tubes with air inlet and outlet tubes. Often two or more of these tubes are used in series to assure complete trapping of the volatile components. Much of the current work involves the use of adsorbents in the trap to further hinder the loss of organic volatiles. Methane has been accumulated on carbon molecular sieves at liquid nitrogen temperature³³ while it passed through an unpacked tube at the same temperature.³⁴ A number of different GC phases have been used in these traps, including Carbowax, silica gel, molecular sieves, Porapaks and Chromosorbs.

Once the volatile constituents of the atmosphere have been trapped, they must be made suitable for further

33. Kaiser, R.E. Anal. Chem. 45:965 (1973).

34. Cooper, J.C., Birdseye, H.E. and R.J. Donnelly. Environ. Sci. & Tech., 8:671 (1974).

analysis. For organic molecules this almost invariably means the use of a GC or GC/MS. The usual preparative procedure is to extract the organic fraction from the water that has been trapped with it, using an organic solvent such as ether, chloroform or benzene. This solvent can then be reduced in volume or used as is for injection into a GC. This procedure can lead to the loss of water-soluble organics such as oxygenated derivatives. If only the volatile components are of interest, they may be distilled from the water and heavier organics into another cold-trap or directly into a GC. Most of the packed columns are designed for simple heat desorption of the organic compounds directly into a GC column or occasionally into another cold trap. The development of the flame-ionization detector (FID) has greatly facilitated this type of analysis because of its relative insensitivity to water vapor, simplifying the separation needed prior to analysis.

Two major difficulties with cryogenic trapping are the clogging of lines with ice during sample collection and its inconvenience as a field method for sampling. In most studies, a bag sample is taken and the cryogenic trapping is done in the laboratory. The use of low temperatures has a great advantage, however, because it decreases the possibility of reactions when the trapped pollutants become

more concentrated. This usually means that samples can be stored at low temperatures for periods of a few weeks before an analysis is performed. For samples of high volatility (i.e., methane), some form of cryogenic trapping is absolutely necessary for accumulation.

GLOSSARY OF TERMS

Aerosol: Finely divided solid or liquid particles suspended in a gas. The term refers to a colloidal system, with both a dispersed medium (particles) and a dispersion medium (a gas). Includes particles whose sizes range from 50μ to 0.01μ or less. ($1\mu = 10^{-6}$ M.)

Atmospheric Aerosol: An aerosol whose dispersion medium is air.

Particulate Size: For air pollution, the radius or diameter of a spherical particle having the same fall velocity with a density of 1 gm. cm^{-3} . The diameter is expressed in microns (μ). This definition is necessary because of the large variation of the density and shape of atmospheric particles.

Total Suspended Particles: The term is used to describe the actual measurement of suspended particles. It is expressed as mass/volume of air sampled, usually in ug/m^3 .

TABLE 3-2

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

The table has been prepared from a review of current literature concerning sampling of inorganic substances from air. Because much of the current literature collected did not contain information about the specific methods of accumulation of the substances, they were not included in the table. Some of the original research for development of sampling and analytical techniques was reviewed in order to obtain an evaluation of the collection efficiency of specific devices for the substances of interest.

Because surveys of the literature concerning the analysis of mercury, ozone, nitrogen oxides and sulfur oxides in air currently exist, the methods for concentrating these substances have not been included in the table.

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
AsH ₃ (Arsine)	Fritted tube absorber	Silver diethyl dithio carbamate (0.5%)	0.5 l/min			Spectrophotometry	Saltzman (1961)
Boron Hydrides (pentaborane, decaborane)	Continuous or field model analyzer	TTC* reagent on paper or tape	1 l/min	4 min sampling time	0.1 ppm	Reflectance photometry	Kuhns, Forsyth, and Masi (1956)
Boron Hydrides	Vigreux bubbler	TTC in alkaline solution	0.5 l/min	1 hr sampling time	0.2 ppm	Spectrophotometry	Hill et al. (1960)
Br ₂	Midget Impinger	KI reagent	0.5 l/min			Spectrophotometry	Saltzman (1961)
COCl ₂ (Phosgene)	Adsorption	15% Kel-F10 on Chromosorb T or MS 5A					Basu, King and Lynn (1972)
Cl ₂	Impinger	NaOH		T = 20-40°C	1 µg	Colorimetry-0-Tolidene	Katz (1969)
Cl ₂	Midget Impinger	KI reagent	0.5 l/min			Spectrophotometry	Saltzman (1961)
Cl ₂	Absorber	Methyl orange and H ₂ SO ₄			.1 ppm		Katz (1969)

*TTC = triphenyltetrazolium chloride

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Cl ₂	Adsorption	15% Kel-F10 on Chromosorb T, Silica Gel, or MS 5A					Basu, King and Lynn (1972)
Cl ₂	Adsorption	Modified Elinson Apparatus-K boride-, Na ₂ CO ₃ - soaked silica gel with fluore-sceine					Elinson et al.(1973)
Cl ₂	Filtration	Alkaline filter paper				Alizarin complexion	Ito (1972)
ClO ₂	Midget Impinger	KI reagent	0.5 l/min			Spectro-photometry	Saltzman (1961)
CrO ₃ (Chromic Acid)	Filtration	Filter paper impregnated with 5-di-phenylcarbazide reagent	600 ml hand pump		99% at .1mg/m ³	Colorimetry	Silverman and Ege (1947)
Fluorides	Impinger	NaOH and H ₂ SO ₄			0.6 µg/m ³	Alizarin lake	Katz (1969)

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Fluorides	Impinger	Titanochromotropate			.2 ppm in solution	Colorimetry	Katz (1969)
Fluorides	Fritted glass scrubber	H ₂ O or dilute alkali	8 l/min		100% 1 ppb	Spectrophotometry-Lanthanide-Alizarin complexan reaction	West, Lyles and Miller (1970)
Fluorides	Bubble or impinger followed by ion exchange				100% 0-10 ppb	Titration	Nielsen and Dangerfield (1955)
HCN	Midget Impinger	5% NaOH	0.5 l/min			Colorimetry	Saltzman (1961)
HCN	Fritted tube	5% NaOH	0.1 l/min			Colorimetry	Saltzman (1961)
HCN	H1 Vol Sampler	Glass fiber filter impregnated with nickel ammoniate	6 l/min	10 min sampling	100% 0.5 ug	Colorimetry	Tanaka et al. (1973)
HCl	Midget Impinger	0.01 N NaOH				Titrimetry	Saltzman (1961)

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
HCl and Cl ⁻	Midget Impinger	Distilled water	2m ³		0.5 ppm	Spectrophotometry; Nephelometry; Silver Nitrate titration	Katz (1969)
HF	Filtration	Alkaline filter paper				Alizarin completion	Ito (1972)
HF	Silver tubes Filled with silver beads	NaCO ₃ dried on beads at 200°C	66 l/min		95% 2-500 µg/m ³		Buck and Stratmann (1965)
HF	Glass impinger	0.1 M NaOH	1 l/min			Colorimetry	Saltzman (1961)
H ₂ S	Adsorption	Modified Elinson Apparatus- Zn acetate-soaked marble powder with BaCl ₂					Elinson et al. (1973)
H ₂ S	Absorber	Alkaline Cadmium hydroxide solution					American Chemical Society (1973)

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
H ₂ SO ₄ (Sulfuric Acid)	Filtration	Mineral Wool			80-100%		Billings, Kurker and Silverman (1958)
H ₂ SO ₄	Absorber	50% ethanol	30 l/min		95.5%		Fudura et al. (1973)
NH ₃	Impinger	Dilute H ₂ SO ₄			1 µg	Nesslerization to form color	Katz (1969)
NH ₃	Impinger	H ₂ SO ₄			4-10 ppb	Indophenol analysis, automatic	Torii (1973)
NH ₃	Midget Impinger	Boric Acid	1 l/min		0.5-0.9 ppm	Indophenol Analysis	Muramatsu et al. (1973)
NH ₃	Filtration	Glass fiber filter impregnated with sodium nitroprussiate	1 l/min			Technicon auto-analyzer Indophenol analysis	Chuone et al. (1974)
NH ₃	Filtration	Glass fiber filter soaked in 20% H ₂ SO ₄ and dried	10 l/min			NH ₄ salts analyzed by pyridine pyrazolone	Kadowaki et al. (1973)

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator Type	Specific Accumulator Formulation	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
OsO_4	Filtration	Low-resistance Whatman filter paper	75 l/hr	2 hr sampling		Colorimetry	McLaughlin et al. (1946)
PH_3 (Phosphine)	Adsorption	1% Mercuric chloride in 0.1% cresol red in ethanol			0.06 μg		Muthu and Majumder (1973)
Pb and Zn (simultaneously)	Greenburg-Smith Impinger	5% HNO_3	1 cfm	35 min		Polarography	Landry (1947)
Pb and Zn	Electrostatic precipitator		3 cfm	12 min		Polarography	Landry (1947)
SiCl_4	Adsorption	Kel-F10 on Chromosorb T					Basu, King and Lynn (1972)

ACCUMULATION OF INORGANIC SUBSTANCES FROM AIR
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TABLE 3-3

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR BY COMPOUND

This table lists those compounds which have been analyzed in air samples along with the accumulation method which was used.

The accumulator is the system used for accumulation (i.e. midjet impinger) or, if a chemical is given, it refers to an absorbent which was used in a column (i.e. Texax).

The desorption or extraction medium generally refers to the method of taking materials from an absorbent resin for analysis. Where collection was by absorption, however, this column contains the name of the absorbing liquid.

All other information is given if it was included in the original article.

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
C ₁ - C ₄	Carbon molecular sieves-10cm	Temperature gradient <200°C	12l/min 4l	-20° to liquid N ₂ temperature gradient	94% at .01 ppb	GC-FID, Carbon molecular sieves -20° -200°C	Kaiser (1973)
C ₁ - C ₁₂	Porapak Q & S		2l/hr	-100°C		GC-FID 10% DC 200 on Supelcort Q -60 - 150°C	Kaiser (1970)
C ₂ - C ₁₂	Alumina	<400°C temp. gradient	1.5l/hr	-20°C		GC-FID DC-200 on Supelcort Q. -60° - 150°C	Kaiser (1970)
C ₂ - C ₆	Molecular sieves Chromosorb P deactivated with di n-butyl phthalate	" 120°C	.5l/min (5 min)	-80°C through drying agents MgClO ₄ or KCO ₃	-100%	GC-FID didecyl phthalate on Chromosorb P 90°C or tri-m-tolyl phosphate on Chromosorb W 93° or 73°C	Williams (1965)
C ₂ - C ₄	Cryogenic trap	heat with hot water	1l	liq. N ₂ 77°K	89-100%	GC-FID hexadecane on firebrick 40°C	Feldstein, Balestrieri (1965)
C ₂ - C ₈	Cryogenic trap Glass-wool filter	distillation	1l/min 200l	liq. O ₂ (CO ₂ removed with ascarite)		Mass spectra	Shepherd, et al. (1951)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
C ₂ - C ₅	Carbowax	room tempera- ture	77cc	liq. N ₂ 77°K		GC-FID dibutyl maleate 0°C	Kopczynski, et. al. (1972)
C ₂ - C ₅	Acid silica gel #58	room tempera- ture	90cc/min 72cc	liq. N ₂		GC-FID acid silica gel 30°C	Lonneman, et. al. (1974)
C ₂ - C ₁₀	10% Carbowax- 1540 on gas Chromosorb Z	room tempera- ture	100cc	liq. N ₂	1-3000ppb	GC-FID 15% dibutyl maleate or acid Chromo- sorb G 25°C	Altshuller, et. al. (1971)
C ₂ - C ₅	Silica gel #15	heat	≤100L	21° - 24°C		GC-FID	Bellar, Sigsby (unpublished)
C ₂ - C ₅	12X molecular sieves	heat		"		"	"
C ₂ - ?	Cryogenic trap			liq. N ₂ 77°K ≤24"Hg pressure	95%	total carbon analyzer	Cooper, Birdseye, Donnelly (1974)
C ₃ - C ₅	Layer trap OV-17 on Chromosorb G silica gel, & molecular sieves 5X & 13X	heat	≤400L	21° - 24°C		GC-FID	Bellar, Sigsby (unpublished)
C ₃ - C ₅	Porapak Q	heat	≤50L	"	95%	"	"

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
C ₃ - C ₅	Chromosorb 103		≤10%	22°C	95%	GC-FID	Bellar, Sigsby (unpublished)
C ₃ - C ₅	Silica gel #58		≤25%	21° - 24°C		GC-FID	"
C ₄ - C ₇ C ₄ - C ₈	10% Carbowax 1540 on Gas Chrom Z	room tempera- ture	90cc 100cc/min	liq. N ₂		GC-FID dibutyl maleate acid silica gel 30°C	Kopczynski, et al. (1972) Lonneman, et al. (1974)
C ₄ - C ₁₅	Tenax GC		50-200cc/min		>90%	GC-FID etched Ni with Emulphor 30° - 170°C	Bertsch, Chang, Zlatkis (1974)
C ₄ - C ₁₅	Dexsil 300 on Chromosorb W	heat		-140°C to liq. N ₂ temp. gradient		GC-FID DC 200 on Kieselguhr -80° - 150°C	Kaiser (1973)
C ₄ - C ₅	10% Carbowax 1540 on fire- brick	hot water heating	40cc/min (4 min)	liq. N ₂	.1ppb	GC-FID bs-2(methoxy ethyl) adi- pate 37°C	Bellar, Brown, Sigsby Jr. (1963)
C ₅ - C ₆	Cryogenic trap	hot water heating	1%	liq. N ₂	99%	GC-FID silicon on firebrick 40°C	Feldstein, Balestrieri (1965)
C ₁ - C ₁₀	Cryogenic trap	room tempera- ture	500cc	liq. air		GC- polyethylene glycol or DC 550 sili- cone	Hughes, Hurn (1960)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
C ₆ - C ₂₀	Cigarette-filter charcoal	extraction with CS ₂ 75°C	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
C ₇ - C ₁₄	Support-bonded silicones (C ₁₈ H ₃₇ SiO _{3/2}) on Chromosorb A	extraction with pentane	18l/min		Quantitative	GC-FID OV-101 on Chromosorb W R.T. - 130°C	Aue, Teli (1971)
C ₈ - C ₁₈	Graphitized carbon black	400°C	.5l/min 200l			GC-FID OV-101 Capillary columns	Raymond, Guiochon (1974)
C ₉ - C ₁₈ alkanes	Tenax GC			25° - 60°C		GC-FID 5% Dexsil 300 on Chromosorb W	Mieure, Dietrich (1973)
C ₁ - C ₃	10% sucrose acetate on Gas Chrom Z	100°C		-55°C		GC-FID Porapak Q 160°C	Bellar, Sigsby, Jr. (1907)
<u>Alkanes and Alkenes</u>							
Isobutane	Carbowax 1540	heat-hot water	30-300cc/min	Cincinnati Air Liquid N ₂	6.0 ppb	GC-FID	Bellar, Brown Sigsby (1963)
n-Butane					24.4		
Butene-1					<1.5		
trans-Butene-2					1.0		
cis-Butene-2					1.1		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Butadiene-1,3 Isobutylene Isopentane n-Pentane 3-Methyl- butene-1 Petene-1 2-Methyl- butane-2 cis-Pentene-2	Carbowax 1540	heat-hot water	30-300cc/min 60-300cc/min	Cincinnati Air Liquid N ₂	1.0ppb <1.5 15.5 7.6 2.6 1.6 3.8 1.7	GC-FID	Bellar, Brown, Sigsby (1963)
Methane Ethane Ethylene Acetylene	Chromosorb 103	heat	<5%	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
Propane n-Butane n-Pentane n-Hexane			20cc/min	head-space	retention volume (l) <10 <20 50 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Methane Ethane Acetylene	Porapak	heat	<5l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
Propane n-Butane n-Pentane n-Hexane	Porapak Q	heat	20cc/min	head-space	retention volume (l) <50 <100 <250 >500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
n-Dodecane n-Tridecane n-Hexadecane n-Pentadecane n-Nonane n-Decane Limonene n-Pentane 2,3 Dimethyl butane n-Hexane Methyl Cyclo- pentane n-Octane 2-Methyl Octane n-Undecane	Tenax GC	heat	50-200cc/min	atmosphere in Houston	1.3-15 ppb 1.6-4.4 0.0-5.7	GC-FIC capillary columns	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
n-Hexane	Tenax GC	heat	20cc/min	head-space	retention volume (l) >500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
n-Hexadecane	Cigarette Filter Charcoal	extraction c CS ₂ 72°	2.5 l/min 25m ³			GC-MS	Grob & Grob (1971)
n-Heptadecane							
n-Octadecane							
n-Nonadecane							
n-Eicosane							
Limonene							
Isododecane							
Isotridecane							
n-Dodecane							
2-Methyl- hexane							
n-Heptane							
Isooctane							
n-Octane							
Isononane							
n-Nonane							
Isodecane							
n-Decane							
Isoundecane							
n-Undecane							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
n-Tridecane Isotetradecane n-Tetradecane n-Pentadecane	Cigarette Filter Carbon	extraction c CS ₂ 72°	2.5 l /min 25m ³			GC-MS	Grob & Grob (1971)
n-Pentadecane n-Hexadecane n-Heptadecane n-Dodecane Isotridecane Isotetradecane n-Decane Isoundecane Decahydronaphthalene Isododecane 1-octene n-Octane 1-nonene Isonane n-Nonane Isodecane	Graphitized Carbon Black	400°C	.5 l /min 200 l			GC-FID OV-101 capillary column	Raymond, Guiochon (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Ethylene	Silica Gel	100°C	550cc/min	0°C	>90% (0.2ppm)		Stitt, Tomimatsu (1953)
Ethylene			2500cc/min	-78°C	100%		Stitt, Tomimatsu (1953)
Acetylene			200cc/min	-78°C	10ppb	color reaction	Hughes, Gordon (1959)
Ethane	12x Molecular Sieves	heat	<20l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
Ethane	Layer Trap (7% OV-17 on chromosorb G + silica gel + 13x & 5A molecular sieves)	heat	<25l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
Ethylene			≤50l				
Acetylene	Scrubber Hg(ClO ₄) ₂ on diatomaceous earth			Room temperature; must be dry		GC-FID	Mitsuo, Aoyama, Yamaki (1974)
<u>Halogenated</u>							
Hexachloro- butadiene	Chromosorb 101	hexane	3l/min	ambient temperature up to 90°C	88-100%	GC-FID OV-17 150°C	Mann, et al. (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Methylene chloride Chloroform	Chromosorb 103	heat	20cc/min	head-space	retention volume (l) 500 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
CCl ₄ C ₂ Cl ₃ H	Chromosorb P deactivated with di-n-butyl phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100%	GC- didecyl phthalate on Chromosorb P 90°C	Williams (1965)
Methylene- chloride Chloroform	Porapak Q	heat	20cc/min	head-space	retention volume (l) 500 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
1,1 Dichloro- ethylene Cl ₂ FCCF ₂ Cl CHCl ₃ CH ₃ CCl ₃ Chloro- ethylene	Porapak Q	on-column adsorption heat	100cc-500cc	30°-50°C		GC- coulometric detector	Williams & Umstead (1968)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
CCl ₂ F ₂ CCl ₃ F CH ₂ Cl ₂ CH ₂ =CCl ₂ Cl ₂ FCCClF ₂	Porapak S	on-column adsorption heat	100cc-500cc	30°-50°C		GC- coulometric detector	Williams & Umstead (1968)
BrF ₂ CCBrF ₂ CHCl ₃ CH ₃ CCl ₃ CHCl=CCl ₂ CCl ₂ =CCl ₂	Porapak S	on column concentration	100cc	30°-50°C		GC- coulometric detector	Williams & Umstead (1968)
CHCl ₃ Tetrachloro- ethylene	Tenax GC	heat	50-200cc/min	atmosphere in Houston	retention volume (l) 500	GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
Methylene- chloride Chloroform	Tenax GC	heat	20cc/min	head-space	500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
CH ₂ Cl ₂ CHCl ₃ Cl ₃ COH Cl ₂ COHCl Cl ₂ CCCl ₂	Tenax GC	heat	20cc/min	head-space	3-8 ppb 7-12 8-16 9-40 3-6	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
General esters & ethers	Activated carbon	heat	13cfm			IR GC-capillary columns	Turk, D'Angio (1962)
Trichloro- ethylene		decane	500cc/min 60l		95-100%	GC- SE-30 on Chromosorb 110°C	Herbolsheimer, Funk, Drasche (1972)
C ₂ Cl ₄	Cigarette filter charcoal	CS ₂	2.5l/min			GC-MS	Grob & Grob (1971)
Tetrachloro- ethylene		extraction \bar{c} CS ₂ 72°C	2.5l/min 25m3			GC-MS	Grob & Grob (1971)
Trichloroethane C ₂ Cl ₄ C ₂ H ₂ Cl ₄ C ₂ Cl ₃ H	Silica Gel	cumene, pyridine or Et ₂ O		dry ice	90%	Colorimetry	Ogata, et al. (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Dichloro- methane	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
CCl ₄ Cl ₃ C ₂ H	Cryogenic Trap Glass Wool Filter	distillation	1l/min 200l	liquid O ₂ CO ₂ removed with ascarite		Mass spectra	Shepherd, et al. (1951)
Perfluoroiso- butylene Hexafluoro- propene	Midget impinger	MeOH	<1ft ³ /hr		95%	Colorimetry	Marcali, Linch (1966)
CH ₃ Br	Impinger	5% KOH in EtOH			79%	Titration	Blinn and Gunther (1949)
<u>Acids</u>							
N-butyric acid Isovaleric acid	Filter paper with 10% NaOH	concentrated HCl/hexane	2-6l/min		<99%	GC TMCBA on Chromosorb W 139°C	Okita, et al. (1973)
<u>Alcohols and Thiols</u>							
Methanethiol 2-propanethiol	Chromosorb P deactivated with dibutyl- phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	-100%	GC- didecylphal- ate on Chromosorb P 90°C	Williams (1965)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
EtOH	Chromosorb P deactivated with di-n-butyl phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100%	GC- didacyl phthalate on Chromosorb P 90°C	Williams (1965)
Propanol	Porapak Q					GC-Porapak Q 160°C	Bellar, Sigsby (1970)
Methanol	Polyethylene Glycol 400			24°C		GC- Celite 545 + Apiezon L	Novak, Vašák, Janák (1965)
Methanol Ethanol 2-Propanol	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Allyl Alcohol 2 Methyl Propane-2-ol 2-Methyl Propanol	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Propylene Glycol Triethylene Glycol	Folin aeration tubes	water	300l 20-30l/min		70-80% 95-97%	Colorimetry	Wise, Puck, Stral (1967)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Esters</u>							
EtOAc	Chromosorb P deactivated with di-n-butyl phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100%	GC- didecyl phthalate on Chromosorb P 90°C	Williams (1965).
Methyl Acetate	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Ethyl acrylate	Midget impinger	KMnO ₄ /NaOH	300cc/min			Colorimetry	Gisclard, Robinson, Kuczo, Jr. (1958)
Methyl methacrylate							
Maleic annhydride	Chromosorb 104	(not removed)	.25l/min		80	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				50		
	Oxypropio- nitrile/Poracil C 80/100				90		
	Porapak Q				90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Maleic anhydride	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al. (1975)
	Carbowax 400/ Poracil C 100/120				95		
	Chromosorb 101				95		
	Tenex GC				80		
	Activated carbons				0-90		
β-Propio- lactone	Chromosorb 104	(not removed)	.25l/min		98	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				50		
	Oxypropio- nitrile/Poracil C 80/100				98		
	Porapak Q				90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
β-Propio- lactone	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al. (1975)
	Carbowax 400/ Poracil C 100/120				90		
	Chromosorb 101				95		
	Tenax GC				95		
	Activated carbons				90-95		
<u>Ketones and Aldehydes</u>							
Acetone	Chromosorb P deactivated with di-n-butyl phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100	GC- didecyl phthalate on Chromosorb P 90°C	Williams (1965)
Acrolein							
Acetone	Porapak Q					GC-Porapak Q 160°C	Bellar, Sigsby (1970)
Butanone							
Cyclohexanone	Silicone E301 on Celite 545	120°C	100cc/min ≤230cc	.5mm Hg pressure drop		GC-FID Silicone E301 on celite	Cropper, Kaminsky (1963)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference	
Ostenal	Cigarette Filter Charcoal	extraction c CS ₂ 72°	2.5l/min 25ml ³	24°C		GC-MS	Grob & Grob (1971)	
Acetone	Polyethylene Glycol 400					GC- Celite 545 + Apiezon L	Novak, Vařák, Janák (1965)	
Acetaldehyde	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min				GC-FID Porapak Q	Bellar, Sigsby (1970)
Acrolein								
Propanal								
Acetone								
2-Methylpro- panal								
Butanal								
Methylethyl- ketone								
Methyl Formate								
Ethyl Formate								
Methyl Acrolein								
Vinyl Methyl Ketone								
2,3-Butane- dione								

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Cyclobutanone Crotonaldehyde 2,2-Dimethyl Butanal	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Methyl Ethyl Ketone	Chromosorb 104 20% Tricresyl phosphate on Chrom W(HP) 100/120 25% Didecyl phthalate on Chrom P 100/120 Oxyproprio- nitrile/Poracil C 80/100 Porapak Q 20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh Carbowax 400/ Poracil C 100/120	(not removed)	.25%/min		98% 20 50 98 90 90 90	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Methyl ethyl ketone	Chromosorb 101 Tenax GC Activated carbons	(not removed)	.25l/min		95% 95 90-95	GC-FID	Pellizzari, et al. (1975)
<u>Ethers and Oxides</u>							
Ether Propyl ether	Chromosorb P deactivated with di-n-butyl phthalate	120°C	5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100	GC- didecyl phthalate on Chromosorb P 90°C	Williams (1965)
bis(chloro- methyl) ether	Porapak Q	180°C	15l 1.5l/min		100	Mass spectra	Collier (1972)
bis-Chloro- meth	Porapak Q	180° under vacuum	15l at 1.5l/min		Quantitative to 20l	Mass. spec.	Collier (1972)
Ethylene Oxide	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
2,3-Butylene Oxide							
Isobutylene Oxide							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,2-Butylene Oxide	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Tetrahydro- furan							
1,2 Dichloro- ethyl ethyl ether	Chromosorb 104 20% Tricresyl phosphate on Chrom W(HP) 100/120 25% Didecyl phthalate on Chrom P 100/120 Oxyproprio- nitrile/Poracil C 80/100 Porapak Q 20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh Carbowax 400/ Poracil C 100/120 Chromosorb 101 Tenax GC Activated carbons	(not removed)	.25l/min		98% 20 50 98 90 90 90 95 95 90-95	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Styrene epoxide	Chromosorb 104	(not removed)	.25%/min		90%	GC-FID	Pellizzari, et al. (1975)
	20% Tricesyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				80		
	Oxypropio- nitrile/Poracil C 80/100				96		
	Porapak Q				95		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				90		
	Chromosorb 101				95		
	Tenax GC				90		
	Activated carbons				30-95		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Butadiene diepoxide	Chromosorb 104	(not removed)	.25L/min		90%	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				80		
	Oxypropio- nitrile/Poracil C 80/100				96		
	Porapak Q				95		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				90		
	Chromosorb 101				95		
	Tenax GC				90		
	Activated carbons				30-95		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Propylene oxide	Chromosorb 104	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				80		
	Oxypropio- nitrile/Poracil C 80/100				96		
	Porapak Q				95		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				90		
	Chromosorb 101				95		
	Tenax GC				90		
	Activated carbons				30-95		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Bis-(2-chloro-ethyl) ether	Chromosorb 104	(not removed)	.25l/min		80%	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120	20					
	25% Didecyl phthalate on Chrom P 100/120	50					
	Oxypropio- nitrile/Poracil C 80/100	90					
	Porapak Q	90					
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	90					
	Carbowax 400/ Poracil C 100/120	95					
	Chromosorb 101	95					
	Tenax GC	80					
Activated carbons	0-90						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Bis-(chloro- methyl) ether	Chromosorb 104	(not removed)	.254/min		80%	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				50		
	Oxypropio- nitrile/Poracil 80/100				90		
	Porapak Q				90		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				95		
	Chromosorb 101				95		
	Tenax GC				80		
	Activated carbons				0-90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Other Aliphatics</u>							
Methyl nitrate Ethyl nitrate Butyronitrate Methyl sulfide Methyl disulfide	Chromosorb P deactivated with di-n- butyl phthalate	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100%	GC- didecyl phthalate on Chromosorb P 90°C	Williams (1965)
Trimethyl phosphate	Graphitized carbon black	heat	<5 l	25°	100	GC	Raymond, Guiochon (1975)
Acetonitrile Methyl nitrate Acrylonitrile Nitromethane Propionitrile	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Acrylonitrile	Midget Impinger	KMnO ₄ /NaOH	300cc/min			Colorimetry	Gisclard, Robinson, Kuczo, Jr. (1958)
Cyclohexyl- mine		.01N HCl	1.5l/min		100	Colorimetry	Watrous, Schultz (1950)
Nitroglycerin Ethylene glycol dinitrate	Midget Impingers with Fritted Glass	95% EtOH	1l/min		~100		Einert, et al. (1963)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Dichloro- nitro-ethane	Impinger	H ₂ SO ₄			95-98%		Jones, Riddick (1952)
2-nitropro- pane	Fritted glass bubbler	conc. H ₂ SO ₄ silica gel 2-propanol H ₂ O	.5l/min		100 99 75 50-100	Colorimetry	Viles (1960)
Methyl amine	Filter paper with 9% oxalic acid	concentrated HCl/hexane	2-6l/min		<99	GC TMCBA on Chromosorb W 139°C	Okita, et al. (1973)
Ethyl methane sulfonate	Chromosorb 104	(not removed)	.25l/min		98%	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				50		
	Oxyproprio- nitrile/Poracil C 80/100				98		
	Porapak Q				90		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Ethyl methane sulfonate	Carbowax 400/ Poracil C 100/120	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al. (1975)
	Chromosorb 101				95		
	Tenax GC				95		
	Activated carbons				90-95		
N-Nitroso- diethylamine	Chromosorb 104	(not removed)	.25l/min			GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120						
	25% Didecyl phthalate on Chrom P 100/120						
	Oxyproprio- nitrile/Poracil C 80/100						
	Porapak Q 20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
N-Nitroso- diethylamine	Carbowax 400/ Poracil C 100/120 Chromosorb 101 Tenax GC Activated carbons N-Nitroso- diethylamine	(not removed)	.25L/min			GC-FID	Pellizzari, et al. (1975)
Nitromethane	Chromosorb 104 20% Tricresyl phosphate on Chrom W(HP) 100/120 25% Didecyl phthalate on Chrom P 100/120 Oxyproprio- nitrile/Poracil C 80/100 Porapak Q 20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	(not removed)	.25L/min		98% 20 50 98 90 90	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Nitromethane	Carbowax 400/ Poracil C 100/120	(not removed)	.25%/min		90%	GC-FID	Pellizzari, et al. (1975)
	Chromosorb 101				95		
	Tenax GC				95		
	Activated carbons				90-95		
Sulfolane	Chromosorb 104	(not removed)	.25%/min		90	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phthalate on Chrom P 100/120				80		
	Oxypropio- nitrile/Poracil C 80/100				96		
	Porapak Q				95		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Sulfolane	Chromosorb 101	(not removed)	.25L/min		95%	GC-FID	Pellizzari, et al. (1975)
	Tenax GC				90		
	Activated carbons				30-95		
1,3-Propane- sultone	Chromosorb 104	(not removed)	.25L/min		80	GC-FID	Pellizzari, et al. (1975)
	20% Tricresyl phosphate on Chrom W(HP) 100/120				20		
	25% Didecyl phythalate on Chrom P 100/120				50		
	Oxyproprio- nitrile/Poracil C 80/100				90		
	Porapak Q				90		
	20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh				90		
	Carbowax 400/ Poracil C 100/120				95		
	Chromosorb 101				95		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,3-Propane- sultone	Tenax GC Activated carbons	(not removed)	.25l/min		80% 0-90%	GC-FID	Pellizzari, et al. (1975)
<u>General Aromatics</u>							
C ₇ - C ₁₀	10% Carbowax 1540 on Gas Chrom Z	room temperature	100cc	liq. N ₂	1-50ppb	GC-FID bis(M-phenoxy -phenoxy) benzene & apiezon 70°C	Altshuller, et al. (1971)
C ₇ - C ₉	Carbowax	room temperature	77cc			GC-FID 10% poly- ethylene glycol on Gas Chrom Z	Kopczynski, et al. (1972)
C ₈ - C ₁₁	Tenax GC	heat		25° - 60°C		GC-FID Dexsil 300 on Chromo- sorb W	Mieure, Dietrich (1973)
C ₆ - C ₂₀	Cigarette filter charcoal	CS ₂	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
C ₈ - C ₁₈	Graphitized carbon black	400°C	.5l/min 200l			GC-FID OV-101 capillary columns	Raymond, Guiochon (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
C ₆ - C ₁₀	Silica Gel 58 Silica Gel 15	250°C heat	≤50 ≤500	21°-24°C 21°-24°C		GC-FID GC-FID	Bellar, Sigsby (unpublished)
	Glass beads	120°	72cc	liq. N ₂	2-130ppb	GC-FID bis(m-phenoxy -phenoxy) benzene with apiezon 73°C	Lonneman, Bellar, Altshuller (1968) Lonneman, et al. (1974)
<u>Unsubstituted Aromatics</u>					retention volume (l)		
Benzene	Chromosorb 103	heat	20cc/min	head-space	500	GC-FID	Bellar, Lichtenberg (1974)
Naphthalene					500	C ₁₀₁	
Benzene	Porapak Q	heat	20cc/min	head-space	500	GC-FID	Bellar, Lichtenberg (1974)
Naphthalene					500	C ₁₀₁	
Benzene	Glass Beads	heat	72cc	liquid N ₂	15ppb	GC	Lonneman, Bellar, Altshuller (1968)
Naphthalene	Tenax GC	heat	50-200cc/min	25°-60°C	>90%	GC-FID Dexsil 300 on Chromo- sorb W	Mieure, Dietrich (1973)
	Tenax GC	heat	50-200cc/min	atmosphere in Houston		GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
Benzene	Tenax GC	heat	50-200cc/min	atmosphere in Houston	1.3-15ppb	GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Benzene	Tenax GC	heat	20cc/min	head-space	retention volume (l) >500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
Benzene	Silicone elastomer E-301 con Celite 545 or polythyene glycol 400			24°		GC-FID 75% Celite 545 25% Apiezon L	Novak, Vařák, Janák (1965)
Indene							
Napthalene							
Benzene	Silicone E301 on Celite 545	120°C	<29cc	.5mm Hg pressure drop		GC-FID Silicone E301 on celite and others	Cropper, Kaminsky (1963)
Pyridine			<110cc				
Benzene	Cigarette filter charcoal	CS ₂	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
Benzofuran							
Naphthalene							
Diphenyl							
Fluorene							
Benzothia- zole		extraction Ā CS ₂ 72°					
Biphenyl							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Acenaphthene Dibenzofuran Fluorene Benzofuran Benzene Napthalene	Cigarette filter charcoal	extraction \bar{c} CS ₂ 72°	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
Acenaphthene Fluorene Napthalene Diphenyl Indane Indene	Graphitized Carbon Black	400°C	.5l/min 200l			GC-FLD OV-101 capillary column	Raymond, Guiochon (1974)
Benzene	Silica Gel 58 Silica Gel 15	250°C heat	<50 ≤500	21°-24°C 21°-24°C		GC-FID GC-FID	Bellar, Sigsby (unpublished)
	Acid Treated Silica Gel		60cc/min		20ppm	formaldehyde stain	Hubbard, Silverman (1950)
	Silica Gel	isooctane	1.2-1.5l/min			UV	Maffett, Doherty, Compton (1962)
	Silica Gel	isopropanol	1-3l/min	dried with 5A molecular sieves	80%	GC-FID polypropylene glycol on diatomaceous earth	Whitman, Johnston (1964)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Benzene	Silica Gel	cyclohexane/ heptane $C_2H_2Cl_2/C_2F_2Cl_4$	1-3l/min	dry air sat. air	80-100% 30%	UV	Elkins, Pagnotto, Compton (1962)
Benzene Furan	1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)
Benzene	Cryogenic Trap Glass Wool Filter	distillation	1l/min	liq. O_2		Mass spectra	Shepherd, et al. (1951)
	Midget Impinger	isooctane	200cc/min	cool air before sampling	90-100%	UV	Andrews, Peterson (1947)
	Layer Trap (7% OV-17 on chromosorb G + silica gel + 13x & 5A molecular sieves)	heat	<500l	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
	Glass Beads	120°	72cc	liq. N_2	.2-130ppb	GC-FID bis(m-phenoxy -phenoxy) benzene with apiezon 73°C	Lonneman, Bellar, Altshuller (1968)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Substituted Nonpolar</u>							
Toluene Xylene	10% Carbowax 1540 on gas Chrom Z	room temperature	100cc	liq. N ₂	1-50ppb	GC-FID bis(M-phenoxy -phenoxy) benzene & apiezon 70°C	Altshuller, et al. (1971)
Toluene Xylenes Styrene	Carbowax	room temperature	77cc			GC-FID 10% poly- ethylene glycol on Gas Chrom Z	Kopczynski, et al. (1972)
Hexachloro- benzene	Chromosorb 101	hexane	3l/min		88-100%	GC-ECD OV-17 150°C	Mann, et al. (1974)
Toluene Chlorobenzene o-Dichloroben- zene 1,2,4-Tri- chlorobenzene	Chromosorb 103	heat	20cc/min	head-space	retention volume (l) 500 500 500 500	GC-FID C ₁₀₁	Bellar Lichtenberg (1974)
Toluene	Porapak Q	heat	20cc/min	head-space	retention volume (l) 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Toluene	Glass Beads	heat	72cc	liquid N ₂	37 ppb	GC	Lonneman, Bellar, Altshuller (1968)
Ethyl Benzene					6		
p-Xylene					6		
m-Xylene					16		
o-Xylene					8		
Isopropyl Benzene					3		
n-Propyl Benzene					2		
3,4 diethyl Toluene					8		
Mesitylene					2		
t-Butyl Benzene					-		
sec-Butyl Benzene					-		
n-Butyl Benzene					-		
Toluene			50-200cc/min		>90%	GC-FID Etched Ni with Emul- phor 30°-170°C	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Xylenes	Tenax GC	heat	50-200cc/min	atmosphere in Houston	>90	GC-FIC Etched Ni with Emul- phor 30°-170°C	Bertsch, Chang, Zlatkis (1974)
1,2,4-Tri- methyl- benzene			50-200cc/min			GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
o-Diethyl- benzene							
Methyl Propyl- benzene							
Dichloro- benzene							
Methylindane							
Methylnaph- thalene							
Styrene							
1,3,5-Tri- methyl- benzene							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Isobutyl Benzene	Tenax GC	heat	50-200cc/min	atmosphere in Houston		GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
o-Ethyl toluene							
Dimethyl- ethyl- benzene							
Toluene					0.3-9.7		
Ethyl Benzene					3.1-4.5		
p-Xylene					2.1-3.4		
m-Xylene					5.9-7.8		
o-Xylene					3.0-4.8		
m-ethyl toluene					1.5-4.0		
			20cc/min	head-space		GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
Toluene					retention volume (1) 500		
Chlorobenzene					500		
o-Dichloro- benzene					500		
1,2,4-Tri- chlorobenzene					500		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Acetophenone	Support-bonded silicones	extraction with pentane	42/min	filtered to 5 μ		GC-FID OV-101 on Chromosorb W R.T. - 130°C	Aue, Tel1 (1971)
Toluene	<u>Silicones</u>			24°		GC-FID 75% Celite 545 25% Apiezon L	Novak, Vasák, Janák (1965)
Xylenes	Silicone elastomer E-301 on Celite 545 or polyethylene glycol 400						
Toluene	Silicone E301 on Celite 545	120°C	<29cc <77cc <110cc <210cc	.5mm Hg pressure drop		GC-FID Silicone E301 on celite and others	Cropper, Kaminsky (1963)
Xylenes							
Benzyl chloride			100cc/min <500cc			GC-FID Silicone E301 on Celite	
<u>Aromatics</u>	<u>Cigarette- filter</u>						
C ₆ - C ₂₀	<u>Charcoal</u>	CS ₂	2.52/min 25m ³			GC-MS	Grob & Grob (1971)
Toluene							
Methylindan							
2-Methyl- naphthalene		extraction c CS ₂ 72°	2.52/min 25m ³				

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1-Methyl- naphthalene	<u>Cigarette filter Charcoal</u>	extraction \bar{c} CS_2 72°	2.5L/min 25m ³			GC-MS	Grob & Grob (1971)
2,6-Dimethyl- naphthalene							
1,6-Dimethyl- naphthalene							
1,8-Dimethyl- naphthalene							
Methyldi- phenyl							
m-Ethyl- toluene							
1,3,5-Tri- methylbenzene							
o-Ethyl- toluene							
1,2,4-Tri- methylbenzene							
p-cymene							
n-Butylben- zene							
Methylpropyl- benzene							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
sec-Butylbenzene	Cigarette Filter Carbon	extraction \bar{c} CS_2 72°	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
1,2,3-Tri- methyl- benzene							
Ethyl-di- methyl- benzene							
Toluene							
Ethylbenzene							
Xylene							
m-xylene							
o-xylene							
n-Propyl- benzene							
Ethyl-toluene							
Dichloro- benzene							
1,2,3,5- Tetramethyl- benzene							
Methylindane							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,2,3,4- Tetramethyl- benzene	<u>Graphitized</u> <u>Carbon Black</u>	400°C	.5l/min 200l			GC-FID OV-101 capillary column	Raymond, Guiochon (1974)
1,2,4,5- Tetramethyl- benzene							
1,4-Dimethyl- naphthalene							
2,3-Dimethyl- naphthalene							
Hexamethyl- benzene							
Diphenyl- methane							
Pentamethyl- benzene							
2-methylnaph- thalene							
1-methylnaph- thalene							
n-Tetra- decane 2-ethyl- naphthalene							
1-Ethyl-naph- thalene							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,6-Dimethyl- naphthalene	<u>Graphitized Carbon Black</u>	400°C	.5l/min			GC-FID OV-101 capillary columns	Raymond, Guiochon (1974)
sec-Butyl- benzene							
1,2,3-Tri- methylbenzene							
P-cymene							
Diethylben- zene							
n-Butylben- zene							
Ethyl-di- methyl- benzene							
1,2,4,5- Tetramethyl- benzene							
Toluene							
Ethyl- benzene							
m-xylene							
Styrene							
o-Xylene							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
n-Propyl- benzene p-ethyl toluene 1,3,5-Tri- methylben- zene p-xylene m-ethyl toluene	Graphitized Carbon Black	400 C	.5L/min 200L			GC-FID OV-101 capillary columns	Raymond, Guiochon (1974)
PCB (Aroclors 1242 or 1248) PCB's	Polyurethane Foam	pet. ether	.4-.8m ³ /min		>90% .39ng/m ³	GC-ECD OV-17/OF-1	Bidleman, Olney (1974a)
			12-34 hr	5-20°C	96-99%		Bidleman, Olney (1974b)
Toluene Styrene Ethyl benzene Propyl ben- zene Isopropyl benzene	Silica Gel 58 Silica Gel 15	250°C heat	<50 ≤500	21°-24°C 21°-24°C		GC-FID GC-FID	Bellar, Sigsby (unpublished)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Toluene	Acid treated Silica Gel		60cc/min		20ppm	formaldehyde stain	Hubbard, Silverman (1950)
Toluene	Silica Gel	isooctane	1.2-1.5l/min	dried with 5A molecular sieves	80%	GC-FID polypropyl- ene glycol on diatoma- ceous earth	Whitman, Johnston (1964)
Xylenes							
Toluene	Cryogenic Trap Glass Wool Filter	distillation	1l/min	liq. O ₂ CO ₂ removed with ascarite		Mass spectra	Shepherd, et al. (1951)
Xylene							
Methyl vinyl- pyridine	Impingers Midget Impinger	KMnO ₄ /NaOH	300cc/min			Colorimetry	Gisclard, Robinson Kuczo, Jr. (1958)
PBC's	n-hexane			cooled prior to collection			Okuno, Tsuji (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Toluene Styrene Ethyl benzene Propyl benzene Isopropyl benzene	Layer Trap Layer Trap (7% OV-17 on chromosorb G + silica gel + 13x & 5A molecular sieves)	heat	≤500l	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
Toluene Xylenes <u>Substituted Polar</u>	Glass beads	120°	72cc	liq. N ₂	2-130ppb	GC-FID bis(m-phen- oxy-phenoxy Benzene with apiezon 73°C	Lonneman, Bellar, Altshuller (1968) Lonneman, et al. (1974)
Phenols	Chromosorb 103	heat	20cc/min	head-space	retention volume (l) 500	GC-FID C ₁₀₁	Bellar Lichtenberg (1974)
Phenols	Porapak Q				500		
Acetophenone Tolualdehyde Methyl acetophenone Benzal- dehyde	Tenax GC	heat	50-200cc/min		>90%	GC-FID etched Ni with Emul- phor	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
p-Tolual- dehyde	Tenax GC	heat	50-200cc/min	atmosphere in Houston		GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
m-Tolual- dehyde							
Acetophenone							
o-Tolual- dehyde							
Phenols	Tenax GC			25°-60°C		GC-FID Dexsil 300 on Chromo- sorb W	Mieure, Dietrich (1973)
p-cresol						GC-FID SiliconeE301 on celite	Cropper, Kaminsky (1963)
Benzaldehyde	Silicone E301 on Celite 545	120 C	100cc/min 230cc	.5mm Hg pressure drop			
Benzoyl chloride							
Aniline							
Nitrobenzene							
Benzaldehyde	Cigarette- Filter Charcoal	CS ₂	2.5l/min			GC-MS	Grob & Grob (1971)
Acetophenone							
Benzaldehyde		extraction c CS ₂ 72°	2.5l/min 25m ³				
4-Methylbenz- aldehyde							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Acetophenone	Cigarette Filter Carbon	extraction \bar{c} CS ₂ 72°	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
2-Methyl- benzalde- hyde							
Dimethyl- benzalde- hyde							
Dimethyl- phthalate	Graphitized Carbon Black	heat	1-2l	20°	87-104%	GC	Raymond, Guiochon (1975)
Phenol	Acid Treated Silica Gel		60cc/min		20ppm	Formaldehyde stain	Hubbard, Silverman (1950)
Xylidine	Midget Impinger	isooctane	200cc/min	cool air before sampling	90-100%	UV	Andrews, Peterson (1974)
2-amino pyridine	Sintered glass	H ₂ O	1.5l/min		100%	Colorimetry	Watrous, Schulz (1950).
p-chloro nitroben- zene	Bead-filled tube	fuming nitric acid					

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Aniline	Chromosorb 104	(not removed)	.25L/min		80%	GC-FID	Pellizzari, et al.
Bis-(2-chloroethyl ether							
N-Nitroso-diethylamine							
Bis-(chloromethyl) ether							
Maleic anhydride							
1,3-Propane sultone	20% Tricresyl phosphate on Chrom W(HP) 100/120				20%		
Aniline							
Bis-(2-chloroethyl) ether							
N-Nitroso-diethylamine							
Bis-(chloromethyl) ether							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Maleic anhydride 1,3-Propane- sulfone	20% Tricresyl phosphate on Chrom W(HP) 100/120	(not removed)	.25l/min		20%	GC-FID	Pellizzari, et al.
Aniline Bis-(2- chloroethyl) ether N-Nitroso- diethylamine Bis-(chloro- methyl) ether Maleic anhydride 1,3-Propane sulfone	25% Didecyl phthalate on Chrom P 100/120	(not removed)	.25l/min		50	GC-FID	Pellizzari, et al.
Aniline Bis-(2- chloro- ethyl) ether	Oxypropio- nitrile/Poracil C 80/100				90		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
N-Nitroso- diethylamine	Oxypropio- nitrile/Poracil C 80/100 Porapak Q	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al.
Bis-(chloro- methyl) ether							
Maleic anhydride							
1,3-Propane sulfone							
Aniline					90%		
Bis-(2- chloro- ethyl)ether							
N-Nitroso- diethylamine							
Bis-(chloro- methyl) ether							
Maleic anhydride							
1,3-Propane sulfone							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Aniline Bis-(2-chloro-ethyl) ether N-Nitroso-diethylamine Bis-(chloromethyl) ether Maleic anhydride 1,3-Propane sulfone	20% Carbowax 600 on Chromosorb W(HP) 100/200 mesh	(not removed)	.25L/min		90%	GC-FID	Pellizzari, et al.
Aniline Bis-(2-chloro-ethyl)ether N-Nitroso-diethylamine	Carbowax 400/ Poracil C 100/120				95%		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Bis-(chloro methyl) ether	Carbowax 400/ Poracil C 100/120	(not removed)	.254/min		95%	GC-FID	Pellizzari, et al.
Maleic anhydride							
1,3-Propane sulfone							
Aniline	Chromosorb 101				95		
Bis-(2- chloro- ethyl)ether							
N-Nitroso- diethylamine							
Bis-(chloro methyl) ether							
Maleic anhydride							
1,3-Propane sulfone							
Aniline	Tenax GC				80		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Bis-(2-chloro-ethyl) ether	Tenax GC	(not removed)	.25l/min		80%	GC-FID	Pellizzari, et al.
N-Nitroso-diethylamine							
Bis-(chloro methyl) ether							
Maleic anhydride							
1,3-Propane sultone							
Aniline	Activated carbons				0-90%		
Bis-(2-chloro-ethyl) ether							
N-Nitroso-diethylamine							
Bis-(chloro methyl) ether							

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Maleic anhydride	Activated carbons	(not removed)	.25l/min		0-90%	GC-FID	Pellizzari, et al.
1,3-Propane sulfone							
<u>Pesticides</u>							
Lindane	<u>Silicones</u>		18l/min			GC OV-101 on Chromosorb W R.T.-130°C	Aue, Telf (1971)
Heptachlor	Support-bond- ed silicones						
Aldrin							
Dieldrin							
Heptachlor expoxide							
p,p'-DDT	<u>Polyurethane</u>	pet. ether	.4-.8m ³ /min		.009-.009ng/m ³	GC-ECD	Bidleman, Olney (1974a)
o,p'-DDT	<u>Foam</u>				.008-.037ng/m ³	OV-17/QF-1	
Chlordane			.5m ³ /min		.005-0.25ng/m ³		
Lindane	Impingers	ethylene glycol	1.5m ³ /hr	cooled prior to collection	poor 100%	Titration	Herzel, Lahmann (1973)
	Polyethylene- Coated Silica						
Aldrin							
Dieldrin							
DDT							

TABLE 3-4

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR BY ACCUMULATOR

This table lists those compounds which have been analyzed in air samples along with the accumulation method which was used.

The accumulator is the system used for accumulation (i.e. midget impinger) or, if a chemical is given, it refers to an adsorbent which was used in a column (i.e. Texax).

The desorption or extraction medium generally refers to the method of taking materials from an adsorbent resin for analysis. Where collection was by adsorption, however, this column contains the name of the adsorbing liquid.

All other information is given if it was included in the original article.

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Carbowax</u>	<u>Aliphatics</u>						
Carbowax	C ₂ - C ₅	room temperature	77cc	liq. N ₂ 77°K		GC-FID dibutyl maleate 0°C	Kopczynski, et al. (1972)
10% Carbowax 1540 on Gas Chrom Z	C ₂ - C ₁₀	room temperature	100cc	liq. N ₂	1-3000 ppb	GC-FID .15% dibutyl maleate or acid Chromo- sorb G 25°C	Altshuller, et al. (1971)
10% Carbowax 1540 on Gas Chrom Z	C ₄ - C ₈	room temperature	90cc 100cc/min	liq. N ₂		GC-FID dibutyl maleate acid silica gel 30°C	Kopczynski, et al. (1972) Lonneman, et al. (1974)
10% Carbowax 1540 on fire- brick	C ₄ - C ₅	hot water heating	40cc/min (4 min.)	liq. N ₂	.1	GC-FID bs-2(methoxy- ethyl) adi- pate 37°C	Bellar, Brown, Sigsby, Jr. (1963)
	<u>Aromatics</u>						
10% Carbowax 1540 on Gas Chrom Z	C ₇ - C ₁₀ Toluenes Xylenes Alkyl benzene	room temperature	100cc	liq. N ₂	1-50	GC-FID bis(m-phenoxy -phenoxy) benzene & apiezon 70°C	Altshuller, et al. (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Carbowax</u> <u>1540</u>	Isobutane	heat-hot water	30-300cc/min 60-300cc/min	Atmosphere in Cincinnati Liquid N ₂	6.0 ppb	GC-FID	Bellar, Brown, Sigsby (1963)
	n-Butane				24.4		
	Butene-1				< 1.5		
	trans-Butene-2				1.0		
	cis-Butene-2				1.1		
	Butadiene-1,3				1.0		
	Isobutylene				< 1.5		
	Isopentane				15.5		
	n-Pentane				7.6		
	3-Methyl- butene-1				2.6		
	Pentene-1				1.6		
	2-Methyl butene-2				3.8		
	cis-Pentene-2				1.7		
Carbowax	C ₇ - C ₉	room temperature	77cc			GC-FID 10% poly- ethylene glycol on Gas Chrom Z	Kopczynski, et al. (1972)
	Toluene						
	Xylenes						
	Alkyl benzenes						
	Styrene						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	Ethyl methane sulfonate	(not removed)	.252/min		90%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone				90%		
	Styrene epoxide						
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
20% Carbowax 600 on Chromo- sorb W(HP) 100/120 mesh	Aniline Bis-(2-chloro- ethyl) ether N-Nitroso- diethylamine Bis-(chloro- methyl) ether Maleic anhydride 1,3-Propane- sultone	(not removed)	.254/min		90%	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Carbowax 400/ Poracil C 100/120	Aniline Bis-(2-chloro- ethyl) ether N-Nitroso- diethylamine Bis-(chloro- methyl) ether Maleic anhydride 1,3-Propane- sultone	(not removed)	.25L/min		95%	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Carbowax 400/ Poracil C 100/120	Ethyl methane sulfonate	(not removed)	.25L/min		90%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone				90%		
	Styrene epoxide						
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 101	Ethyl methane sulfonate β-Propiolactone N-Nitroso- diethylamine 1,2 Dichloro- ethyl ethyl ether Nitromethane Methyl ethyl ketone Styrene epoxide N-Nitroso- diethylamine Butadiene diepoxide Glycidaldehyde Sulfolane Propylene oxide	(not removed)	.25l/min		<div> <div>95%</div> <div>95%</div> </div>	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 101	Aniline Bis-(2-chloro-ethyl) ether N-Nitroso-diethylamine Bis-(chloro-methyl) ether Maleic anhydride 1,3-Propane-sultone	(not removed)	.25l/min		95%	GC-FID	Pellizzari, et al. (1975)
Chromosorb 101	Acidic and neutral compounds	heat	.5-2l/min	ambient temperature up to 90°C		GC-FID	Mieure, Dietrich (1973)
	Hexachloro-butadiene Hexachloro-benzene	hexane	3l/min		88-100%	GC-ECD OV-17 150°C	Mann, et al. (1974)
Chromosorb 102	Organics (bp>60°C)	120°C	4l/min 10l	2 psi pressure drop	>95% .1 ppb	GC-FID Carbowax 20N 60-180°C	Dravnieks, et al. (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 103	Low-boiling compounds	heat	.5-2l/min	ambient temperature up to 90°C		GC-FID	Mieure, Dietrich (1973)
	Methane	heat	<5l	22°C		GC-FID	Bellar, Sigsby (Unpublished)
	Ethane	heat	<5l	22°C	95%	GC-FID A	Bellar, Sigsby (Unpublished)
	Ethylene	heat	<5l	22°C		GC-FID	Bellar, Sigsby (Unpublished)
	Acetylene						
	Propane	heat	20cc/min	head-space	retention volume (l) <10	GC-FID	Bellar, Lichtenberg (1974)
	n-Butane				<20	C ₁₀₁	
	n-Pentane				50		
	n-Hexane				500		
	Benzene				500		
	Toluene				500		
	Methylene chloride				500		
	Chloroform				500		
	Phenols				500		
	Napththylene				500		
	Chlorobenzene				500		
	o-Dichloro- benzene				500		
	1,2,4-Trichloro- benzene				500		
	C ₃ - C ₅ aliphatics		≤10l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 104	Ethyl methane sulfonate	(not removed)	.25L/min		98%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone				90%		
	Styrene epoxide						
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb 104	Aniline Bis-(2-chloro- ethyl) ether N-Nitroso- diethylamine Bis-(chloro- methyl) ether Maleic anhydride 1,3-Propane- sultone	(not removed)	.25l/min		80%	GC-FID	Pellizzari, et al. (1975)
Chromosorb P deactivated with di- n-butyl phthalate	Methanethiol 2-propanethiol C ₂ - C ₆ aliphatics	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	-100 -100	GC- didecyl- phthalate on Chromosorb P 90°C GC-FID didecyl- phthalate on Chromosorb P 90°C or tri-m-tolyl phosphate on Chromosorb W 93° or 73°C	Williams (1965) Williams (1965)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Description or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Chromosorb P deactivated with di- n-butyl phthalate	EtOH Acetone EtOAc Ether C ₁ -C ₄ aldehydes Propyl ether Acrolein Methyl nitrate Ethyl nitrate Butyronitrate CCl ₄ C ₂ Cl ₃ H Methyl sulfide Methyl disulfide	120°C	.5l/min	-80°C dried with MgClO ₄ or KCO ₃	~100%	GC- didecyl- phthalate on Chromosorb P 90°C	Williams (1965)
<u>Dexsil</u> Dexsil 300 on Chromosorb W	C ₄ - C ₁₅ aliphatics	heat		-140°C to liq. N ₂ temp. gradient		GC-FID DC 200 on Kieselguhr -80°-150°C	Kaiser (1973)
<u>Porapak</u> Porapak Q & S	C ₁ - C ₁₂ aliphatics		2l/hr.	-100°C		GC-FID 10% DC 200 on Supelcort Q -60°-150°C	Kaiser (1970)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Porapak Q	Methane Ethane	heat	<5l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
	Acetylene	heat	<5l	22°C		GC-FID	Bellar, Sigsby (Unpublished)
	C ₃ - C ₅ aliphatics	heat	≤50l	22°C	95	GC-FID	Bellar, Sigsby (Unpublished)
<u>Porapaks</u>							
Porapak Q	bis(chloro- methyl) ether	180°C	15l 1.5l/min		100	Mass spectra	Collier (1972)
	Acetone Propanol Butanone					GC-Porapak Q 160°C	Bellar, Sigsby (1970)
	C ₁ - C ₃ chlorinated & fluorinated alkanes and alkenes		1.7l		Quantitative	GC-ECD Porapak 100°-166°C	Williams, Umstead (1968)
	Propane n-Butane n-Pentane n-Hexane Benzene Toluene	heat	20cc/min	head-space	retention volume (l) <50 <100 <250 >500 500 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulant	Accumulator	Description or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Porapak Q	Methylene- chloride	heat	20cc/min	head-space	retention volume (%) 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
	Chloroform				500		
	Phenols				500		
	Naphthalene				500		
	1,1 Dichloro- ethylene	on-column adsorption heat	100-500cc	30°-50°C		GC- coulometric detector	Williams, Umstead (1968)
	Cl ₂ FCCF ₂ Cl						
	CHCl ₃						
	CH ₃ CCl ₃						
	Chloro- ethylene						
	bis-Chloro- methyl ether	180° under vacuum	15% at 1.5% / min		Quantitative to 20%	Mass Spec.	Collier (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Porapak Q	Ethyl methane sulfonate	(not removed)	.25L/min		90%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone				95%		
	Styrene epoxide						
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Porapak Q	Aniline Bis-(2-chloro-ethyl) ether N-Nitroso-diethylamine Bis-(chloro-methyl) ether Maleic anhydride 1,3-Propane-sultone	(not removed)	.25l/min		90%	GC-FID	Pellizzari, et al. (1975)
Porapak S	CCl ₂ F ₂ CCl ₃ F CH ₂ Cl ₂ CH ₂ =CCl ₂ Cl ₂ FCCClF ₂ BrF ₂ CCBrF ₂ CHCl ₃ CH ₃ CCl ₃ CHCl=CCl ₂ CCl ₂ =CCl ₂	on-column adsorption heat	100cc-500cc 100cc	30°-50°C		GC-coulometric detector	Williams, Umstead (1968)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Glass Beads	Benzene	heat	72cc	liquid N ₂	15 ppb	GC	Lonneman, Bellar, Altshuller (1968))
	Toluene				37		
	Ethyl Benzene				6		
	p-Xylene				6		
	m-Xylene				16		
	o-Xylene				8		
	Isopropyl Benzene				3		
	n-Propyl Benzene				2		
	3,4 Ethyl Toluene				8		
	Mesitylene				2		
	t-Butyl Benzene				-		
	sec-Butyl Benzene				-		
	n-Butyl Benzene				-		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	Volatile organics	300°C	.5-2l/min			GC-FID etched Ni with Emul- phor 30°-170°C	Zlatkis, Lichtenstein, Tishbee (1973)
	Basic, neutral and high- boiling com- pounds	heat	.5-2l/min	Ambient temperature up to 90°C		GC-FID	Mieure, Dietrich (1973)
	C ₄ - C ₁₅ aliphatics		50-200cc/min		>90%	GC-FID etched Ni with Emul- phor 30°-170°C	Bertsch, Chang, Zlatkis (1974)
	C ₉ - C ₁₈ alkanes			25° - 60°C		GC-FID 5% Dexsil 300 on Chromosorb W	Mieure, Dietrich (1973)
	<u>Aromatics</u>						
	C ₈ - C ₁₁ Alkyl benzenes Naphthalene Toluene Naphthalene Xylenes Alkyl benzenes	heat	50-200cc/min	25° - 60°C	>90%	GC-FID Dexsil 300 on Chromo- sorb W GC-FID Etched Ni with Emul- phor 30°-170°C	Mieure, Dietrich (1973) Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	<u>Oxygenated Compounds</u> Acetophenone Tolualdehyde Methyl acetophenone 1,2,4-Trimethyl- benzene o-Diethylben- zene n-Dodecane Methyl Propyl- benzene Dichlorobenzene n-Tridecane Benzaldehyde Methylindane p-Tolualdehyde m-Tolualdehyde Acetophenone o-Tolualdehyde Naphthalene Methylnaph- thalene n-Hexadecane n-Pentadecane	heat	50-200cc/min	Atmosphere in Houston	>90%	GC-FID etched Ni with Emul- phor GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	Ethyl methane sulfonate	(not removed)	.25l/min		95%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso-diethylamine						
	1,2 Dichloro-ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone				90%		
	Styrene epoxide						
	N-Nitroso-diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	Aniline	(not removed)	.25l/min		80%	GC-FID	Pellizzari, et al. (1975)
	Bis-(2-chloro-ethyl) ether						
	N-Nitroso-diethylamine						
	Bis-(chloro-methyl) ether						
	Maleic anhydride						
	1,3-Propane-sultone						
	Styrene	heat	50-200cc/min	atmosphere in Houston		GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)
	1,3,5-Trimethylbenzene						
	Isobutyl Benzene						
	1-Methyl, 2-Ethyl Benzene						
	Dimethylethylbenzene						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Tenax GC</u>	Benzene n-Nonane Toluene n-Decane Ethyl Benzene p-Xylene m-Xylene o-Xylene m-Methylethyl benzene Limonene n-Pentane 2,3 Dimethyl butane n-Hexane Methyl Cyclo- pentane n-Octane 2-Methyl octane CHCl ₃ Tetrachloro- ethylene n-Undecane	heat	50-200cc/min	atmosphere in Houston	1.3-15 ppb 1.6-4.4 0.3-9.7 1.0-2.7 3.1-4.5 2.1-3.4 5.9-7.8 3.0-4.8 1.5-4.0 0.0-5.7	GC-FID capillary columns	Bertsch, Chang, Zlatkis (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Tenax GC	n-Hexane Benzene Toluene Methylene- chloride Chloroform Chlorobenzene o-Dichloro- benzene 1,2,4-Tri- chlorobenzene	heat	20cc/min	head-space	retention volume (l) >500 500 500 500 500 500 500 500	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
	CH ₂ Cl ₂ CHCl ₃ Cl ₃ CCH ₃ Cl ₂ CCHCl Cl ₂ CCCl ₂	heat	20cc/min	head-space	3-8 ppb 7-12 8-16 9-40 3-6	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974b)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Tenax GC</u>	Phenols P-cresols Alcohols Ketones Phthalate esters			25° - 60°C		GC-FID Dexsil 300 on Chromosorb W	Mieure, Dietrich (1973)
<u>Silicones</u>							
Support-bonded silicones (C ₁₈ H ₃₇ SiO _{3/2}) on Chromo- sorb A	C ₇ - C ₁₄ aliphatics	extraction with pentane	18l/min		Quantitative	GC-FID OV-101 on Chromosorb W R.T. - 130°C	Aue, Teli (1971)
Support-bonded silicones	Acetophenone	extraction with pentane	4l/min	filtered to 5μ		GC-FID OV-101 on Chromosorb W R.T. - 130°C	Aue, Teli (1971)
	Lindane Heptachlor Aldrin Dieldrin Heptachlor epoxide		18l/min			GC- OV-101 on Chromosorb W R.T. - 130°C	Aue, Teli (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Silicones</u>							
Silicone elastomer E-301 on Celite 545 or polyethylene glycol 400	C ₆ - C ₁₀ Benzene Toluene Xylenes Alkyl benzenes Indene Naphthalene			24°C		GC-FID 75% Celite 545 25% Apiezon L	Novak, Vašák, Janák (1965)
Silicone E301 on Celite 545	Benzene Toluene Pyridine Xylenes	120°C	≤29cc ≤77cc ≤110cc ≤210cc	.5mm Hg pressure drop		GC-FID Silicone E301 on celite and others	Cropper, Kaminsky (1963)
	Cyclohexanone Benzaldehyde Benzoyl chloride	120°C	100cc/min ≤230cc	.5mm Hg pressure drop		GC-FID Silicone E301 on celite	Cropper, Kaminsky (1963)
	C ₄ - C ₆ alcohols	120°C	100cc/min	.5mm Hg pressure drop		GC- Silicone E301 on celite	Cropper, Kaminsky (1963)
	Aniline Nitrobenzene	120°C	100cc/min	.5mm Hg pressure drop		Silicone E301 on celite	Cropper, Kaminsky (1963)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Silicones</u>							
Silicone E301 on Celite 545	Benzyl chloride Benzotri- chloride Benzal chloride	120°C	100cc/min ≤500cc	.5mm Hg pressure drop		GC-FID Silicone E301 on Celite	Cropper, Kaminsky (1963)
SE-52 on Chromosorb W or Charcoal	C ₁ - C ₂ chlorinated alkanes and alkenes	heat heat	50cc/min 10¢/min	dry ice	60-100% 80-100	GC-ECD SE-52 on Chromosorb W	Murray, Riley (1973)
<u>Activated Carbon</u>							
	Alkanes and alkenes	heat desorp- tion into liquid N ₂ trap	13cfm			IR and GC- capillary columns	Turk, D'Angio (1962)
	General aromatics						
	General esters & ethers	heat	13cfm			IR GC-capillary columns	Turk, D'Angio (1962)
	Trichloro- ethylene	decane	500cc/min 60¢		95-100	GC- SE-30 on Chromosorb 110°C	Herbolsheimer, Funk, Drasche (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Activated carbons	Ethyl methane sulfonate β-Propiolactone N-Nitroso- diethylamine 1,2 Dichloro- ethyl ethyl ether Nitromethane Methyl ethyl ketone Styrene epoxide N-Nitroso- diethylamine Butadiene diepoxide Glycidaldehyde Sulfolane Propylene oxide	(not removed)	.25L/min		<div> <div>90-95%</div> <div>30-95%</div> </div>	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Activated carbons	Aniline	(not removed)	.25l/min		<div> <div></div> 0-90% </div>	GC-FID	Pellizzari, et al. (1975)
	Bis-(2-chloro-ethyl) ether						
	N-Nitroso-diethylamine						
	Bis-(chloro-methyl) ether						
	Maleic anhydride						
	1,3-Propane-sultone						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Cigarette- filter</u> <u>Charcoal</u>	C ₆ - C ₂₀ aliphatics	extraction with CS ₂ 75°C	2.5l/min 25m ³			GC-MS	Grob & Grob (1971)
	<u>Aromatics</u> C ₆ - C ₂₀ Toluene Benzene Benzofuran Methylindan Naphthalene Alkyl benzenes Diphenyl Fluorene Alkyl naphthalenes	CS ₂	2.5l/min. 25m ³			GC-MS	Grob & Grob (1971)
	<u>Others</u> Benzaldehydes Acetophenone C ₁₀ alcohols C ₂ Cl ₄	CS ₂	2.5l/min			GC-MS	Grob & Grob (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Cigarette Filter Charcoal	2-Methylnaph- thalene n-Hexadecane 1-Methylnaph- thalene Benzothiazole n-Heptadecane Diphenyl 2,6-Dimethyl- naphthalene 1,6-Dimethyl- naphthalene 1,8-Dimethyl- naphthalene n-Octadecane Methyldiphenyl Acenaphthene n-Nonadecane Dibenzofuran Fluorene n-Eicosane Benzaldehyde Benzofuran	extraction \bar{c} CS_2 72°	2.5l/min 25 m ³			GC-MS	Grob & Grob (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Cigarette Filter Charcoal	1-Ethyl-3- methylbenzene Limonene 1,3,5-Tri- methylbenzene Isododecane 1-Ethyl-2- methylbenzene 1,2,4-Tri- methylbenzene Octenal 1-Isopropyl-4- methylbenzene n-Butylbenzene Propylmethyl- benzene sec-Butylben- zene 1,2,3-Tri- methylbenzene Isotridecane n-Dodecane Ethyl dimethyl- benzene	extraction C CS ₂ 72°	2.5 l/min 25 m ³			GC-MS	Grob & Grob (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Cigarette- Filter Char- coal</u>	2-Methylhexane n-Heptane Isooctane Benzene n-Octane Isononane n-Nonane Toluene Isodecane Tetrachloro- ethylene n-Decane Ethylbenzene 1,4-Dimethyl- benzene Isoundecane 1,3-Dimethyl- benzene 1,2-Dimethyl- benzene n-Propylbenzene n-Undecane 1-Ethyl-4-methyl- benzene	extraction \bar{c} CS_2 72°C	2.5 l/min 25 m ³			GC-MS	Grob & Grob (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Cigarette Filter Carbon	Dichlorobenzene 1,2,3,5-Tetra- methylbenzene n-Tridecane Methylindane 1,2,3,4-Tetra- methylbenzene Isotetradecane 1,2,4,5-Tetra- methyl benzene 4-Methylbenzal- dehyde 3-Methylbenzal- dehyde n-Tetradecane Acetophenone 2-Methylbenzal- dehyde Naphthalene n-Pentadecane Dimethylbenzal- dehyde	extraction C CS ₂ 72°	2.5l/min 25 m ³			GC-MS	Grob & Grob (1971)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Graphitized Carbon Black</u>	1-4-Dimethyl- naphthalene	400°C	.5l/min 200 l			GC-FID OV-101 capillary column	Raymond, Guiochon (1974)
	2-3-Dimethyl- naphthalene						
	Hexamethyl- benzene						
	Acenaphthene						
	n-Pentadecane						
	Diphenylmethane						
	n-Hexadecane						
	Fluorene						
	n-Heptadecane						
	n-Octadecane						
<u>Graphitized Carbon Black</u>	Dimethyl- phthalate	heat	1-2 l	20°	87-104%	GC	Raymond, Guiochon (1975)
	Trimethyl phosphate		<5 l	25°	100%		
	Naphthalene	400°C	.5 l/min 200 l			GC-FID OV-101 capillary columns	
	n-Dodecane						
	Isotridecane						
	Tridecene						
	Pentamethyl- benzene						
	2-Methylnaph- thalene						
	1-methylnaph- thalene						
	Isotetradecane						
	Diphenyl						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Graphitized Carbon Black	n-Tetradecane 2-ethylnaphthalene 1-Ethylnaphthalene 1,6-dimethylnaphthalene sec-Butylbenzene n-Decane 1,2,3-Tri-methylbenzene p-cymene Indane Isoundecane Indene Diethylbenzene n-Butylbenzene Ethyl dimethylbenzene Decahydronaphthalene n-Undecane 1,2,4,5-Tetramethylbenzene Isododecane	400°C	.52/min 200 μ			GC-FID OV-101 capillary columns	Raymond, Guiochon (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Graphitized Carbon Black	C ₈ -C ₁₈ aliphatics <u>Aromatics</u> C ₈ -C ₁₈ Toluene Alkyl benzenes Styrene 1-octene n-octane Ethylbenzene 1-nonene m-xylene Isonane o-Xylene n-Nonane Isodecane n-Propyl- benzene p-ethyl toluene 1,3,5-Tri- methylbenzene p-xylene m-ethyl toluene	400°C	.5l/min 200l			GC-FID OV-101 capillary columns	Raymond, Guiochon (1974)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Polyurethane Foam</u>	PCB (Aroclors 1242 or 1248)	pet. ether	.4-.8 m ³ /min		>90% .3-9ng/m ³	GC-ECD OV-17/QF-1	Bidleman, Olney (1974a)
	p,p'-DDT o,p'-DDT Chlordane PCB's		.5 m ³ /min 12-34 hr	5-20°C	.009-.090ng/m ³ .008-.037ng/m ³ .005-0.25ng/m ³ 96-99%		Bidleman, Olney (1974b)
<u>Alumina</u>	C ₂ -C ₁₂ aliphatics	≤400°C temp. gradient	1.5l/hr	-20°C		GC-FID DC-200 on Supelcort Q. -60°-150°C	Kaiser (1970)
<u>Silica Gel</u>	Propane	heat	20cc/min	head-space	retention volume (l) >50	GC-FID C ₁₀₁	Bellar, Lichtenberg (1974)
	n-Butane n-Pentane n-Hexane Benzene Toluene				>500 500 500 500 500		

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Silica Gel</u>	<u>Aliphatics</u>						
Silica Gel	Ethylene	100°C	550cc/min	0°C	>90% (.02ppm)		Stitt, Tomimatsu (1953)
	Ethylene	100°C	2500cc/min	-78°C	100%		Stitt, Tomimatsu (1953)
	Acetylene		200cc/min	-78°C	10ppb	color reaction	Hughes, Gordon (1959)
Silica Gel 58	Acetylene	250°C	≤254	22°C		GC-FID	Bellar, Sigsby (Unpublished)
Acid Silica Gel 58	C ₂ - C ₅	room temp.	90cc/min 72cc	liq. N ₂		GC-FID acid silica gel 30°C	Lonneman, et al. (1974)
Silica Gel 15	C ₂ - C ₅	heat	≤1004	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
Silica Gel 58	C ₃ - C ₅		≤254	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
	<u>Aromatics</u>						
Silica Gel 58	C ₆ - C ₁₀	250°C	≤50	21°-24°C		GC-FID	Bellar, Sigsby
Silica Gel 15	Benzene	heat	≤500	21°-24°C		GC-FID	(Unpublished)
	Toluene						
	Styrene						
	Ethyl benzene						
	Propyl benzene						
	Isopropyl benzene						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Silica Gel</u>	<u>Aromatics</u>						
Acid Treated Silica Gel	C ₆ - C ₁₀ Benzene Toluene Phenol		60cc/min		20ppm	formaldehyde stain	Hubbard, Silverman (1950)
Silica Gel	Benzene Toluene	isooctane	1.2-1.5l/min			UV	Maffett, Doherty, Monkman (1956)
	Benzene Toluene Xylenes	isopropanol	1-3l/min	dried with 5A molecular sieves	80%	GC-FID polypropylene glycol on diatomaceous earth	Whitman, Johnston (1964)
	Benzene	cyclohexane/ heptane C ₂ H ₂ Cl ₂ /C ₂ F ₂ Cl ₄	1-3l/min	dry air sat. air	80-100 30	UV	Elkins, Pagnotto, Comproni (1962)
	<u>Halogenated Aliphatic</u>						
	Trichlorethane C ₂ Cl ₄ C ₂ H ₂ Cl ₄ C ₂ Cl ₃ H	cumene, pyri- dine or Et ₂ O		dry ice	90	Colorimetry	Ogata, et al. (1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Molecular Sieves</u>	<u>Aliphatics</u>						
Carbon Molecular Sieves - 10cm	C ₁ - C ₄	Temperature gradient <200°C	12l/min 4l	-20° to liquid N ₂ temperature gradient	94% at .01 ppb	GC-FID Carbon molecular sieves -20°-200°C	Kaiser (1973)
12x Molecular Sieves	Ethane	heat	<20l	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
13x Molecular Sieves	Ethylene		100l/min (10 min)		2ppb	GC-FID Porapak Q 70°C	Harbourn, McCambley, Trollope (1973)
Molecular Sieves	C ₂ - C ₆	≤400°C temperature gradient				GC-FID DC-200 on Supelcort Q -60°-150°C	Kaiser (1970)
12x Molecular Sieves	C ₂ - C ₅	heat		21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
<u>Polyethylene Glycol</u>							
Polyethylene Glycol 400	Methanol Acetone			24°C		GC- Celite 545 + Apiezon L	Novak, Vašák, Janák (1965)
Polyethylene Glycol 400 on Stainless Steel Nets	Insecticides	benzene				GC	Beyermann, Eckrich (1974 & 1973)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
25% Didecyl phthalate on. Chrom P 100/120	Ethyl methane sulfonate	(not removed)	.25l/min		50%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone						
	Styrene epoxide				80%		
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
25% Didecyl phthalate on Chrom P 100/120	Aniline	(not removed)	.25L/min		50%	GC-FID	Pellizzari, et al. (1975)
	Bis-(2-chloro-ethyl) ether						
	N-Nitroso-diethylamine						
	Bis-(chloro-methyl) ether						
	Maleic anhydride						
Oxypropio- nitrile/Poracil C 80/100	1,3-Propane-sultone	(not removed)	.25L/min		98%	GC-FID	
	Ethyl methane sulfonate						
	β-Propiolactone						
	N-Nitroso-diethylamine						
	1,2 Dichloro-ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
Oxypropioni- trile/Poracil C 80/100	Styrene epoxide	(not removed)	.25L/min		96%	GC-FID	Pellizzari, et al. (1975)
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide				90%		
	Aniline						
	Bis-(2-chloro- ethyl) ether						
	N-Nitroso- diethylamine						
	Bis-(chloro- methyl) ether						
	Maleic anhydride						
	1,3-Propane- sultone						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
20% Tricresyl phosphate on Chrom W(HP) 100/120	Ethyl methane sulfonate	(not removed)	.25l/min		20%	GC-FID	Pellizzari, et al. (1975)
	β-Propiolactone						
	N-Nitroso- diethylamine						
	1,2 Dichloro- ethyl ethyl ether						
	Nitromethane						
	Methyl ethyl ketone						
	Styrene epoxide				20%		
	N-Nitroso- diethylamine						
	Butadiene diepoxide						
	Glycidaldehyde						
	Sulfolane						
	Propylene oxide						

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
20% Tricresyl phosphate on Chrom W(HP) 100/120	Aniline Bis-(2-chloro- ethyl) ether N-Nitroso- diethylamine Bis-(chloro- methyl) ether Maleic anhydride 1,3-Propane- sultone	(not removed)	.25l/min		20%	GC-FID	Pellizzari, et al. (1975)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,2,3-tris (2-cyanoethoxy)- propane on Chromosorb W	Methanol Acetaldehyde Acrolein Propanal Acetone 2-Methylpro- panal Butanal Methylethyl- ketone Benzene Ethylene Oxide Methyl Formate Ethanol Propylene Oxide Acetonitrile Furan Dichloromethane Methyl Nitrate Ethyl Formate Acrylonitrile 2-Propanol Methyl Acetate	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
			Sample Volume				
1,2,3-tris (2-cyanoethoxy) propane on Chromosorb W	Nitromethane Allyl Alcohol 2,3-Butylene Oxide Methyl Acrolein Isobutylene Oxide Propionitrile 2-Methyl Furan 1,2-Butylene Oxide 2-Methyl Propane-2-ol Tetrahydro- furan Vinyl Methyl Ketone 2,3-Butanedione Cyclobutanone Crotonaldehyde 2,2-Dimethyl Butanal 2-Methyl Propanol	heat	50cc/min			GC-FID Porapak Q	Bellar, Sigsby (1970)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
1,2,3-tris (2 cyanoethoxy) Propane	General Aromatics C ₃ - C ₄ Alcohols, esters, alde- hydes, oxides, ketones	heat	50cc/min			GC-FID Porapak Q 160°C	Bellar, Sigsby (1970)
1,2,3-tris (2 cyanoethoxy) Propane on Chromosorb W			50cc/min			GC- Porapak Q 160°C	Bellar, Sigsby (1970)
<u>Cryogenic Trap</u>	<u>Aliphatics</u>						
Cryogenic Trap	C ₂ - C ₄	heat with hot water	1L	liq. N ₂ 77°K	89-100%	GC-FID hexadecane on firebrick 40°C	Feldstein, Balestrieri (1965)
Cryogenic Trap Glass-wool Filter	C ₂ - C ₈	distillation	1L/min 200L	liq. O ₂ (CO ₂ removed with ascarite)		Mass spectra	Shepherd, et al. (1951)
Cryogenic Trap	C ₂ - ?			liq. N ₂ 77°K ≤24" Hg pressure	95	total carbon analyzer	Cooper, Birdseye, Donnelly (1974)
	C ₅ - C ₆	hot water heating	1L	liq. N ₂	99	GC-FID silicon on firebrick 40°C	Feldstein, Balestrieri (1965)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Cryogenic Trap</u>	<u>Aliphatics</u>						
Cryogenic Trap	C ₆ - C ₁₀	room temp.	500cc	liq. air		GC- polyethylene glycol or DC 550 sili- cone	Hughes, Hurn (1960)
	<u>Others</u>						
Cryogenic Trap Glass Wool Filter	Benzene Toluene Xylene	distillation	1l/min	liq. O ₂ CO ₂ removed with ascarite		Mass spectra	Shepherd, et al. (1951)
Cryogenic Trap	C ₁ - C ₅ Alcohols Aldehydes Ketones Epoxides	room temp.	500cc	liquid air		GC-TC polyethylene glycol 400	Hughes, Hurn (1960)
Cryogenic Trap Glass Wool Filter	CCl ₄ Cl ₃ C ₂ H	distillation	1l/min 200l	liquid O ₂ CO ₂ removed with ascarite		Mass spectra	Shepherd, et al. (1951)
<u>Impingers</u>							
Midget Impinger	Benzene Xylidine	isooctane	200cc/min	cool air before sampling	90-100%	UV	Andrews, Peterson (1947)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Impingers</u>							
Midget Impinger	Ethyl acrylate Methyl methacrylate Acrylonitrile Methyl vinyl Pyridine	KMnO ₄ /NaOH	300cc/min			Colorimetry	Gisclard, Robinson, Kuczo, Jr. (1958)
	Cyclohexyl- amine	.01N HCl	1.5l/min		100%	Colorimetry	Watrous, Schultz (1950)
Midget Impingers with Fritted Glass	Nitroglycerin Ethylene glycol dinitrate	95% EtOH	1l/min		~100%		Einert, et al. (1963)
Midget Impinger	Perfluoroiso- butylene Hexafluoro- propene R-C=CF ₂	MeOH	<1 ft ³ /hr		95%	Colorimetry	Marcalli, Lynch (1966)
Impinger	Nitroparaffins Dichloro-nitro- ethane CH ₃ Br PBC's	H ₂ SO ₄ 5% KOH in EtOH n-hexane			95-98% 79%		Jones, Riddick (1952)
				cooled prior to collection		Titration	Blinn, Gunther (1949) Okuno, Tsuji (1972)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Impingers</u>							
Impingers Polyethylene- Coated Silica	Lindane Aldrin Dieldrin DDT	ethylene glycol	1.5 m ³ /hr		poor 100%		Herzel, Lahmann (1973)
<u>Layer Trap</u>	<u>Aliphatics</u>						
Layer Trap (7% OV-17 on chromosorb G + silica gel + 13x & 5A molecular sieves)	Ethane	heat	<25%	22°C	95%	GC-FID	Bellar, Sigsby (Unpublished)
	Ethylene	heat	≤50%				Bellar, Sigsby (Unpublished)
	C ₃ - C ₅	heat	≤400%	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)
	<u>Aromatics</u>						
	Benzene Toluene Styrene Ethyl benzene Propyl benzene Isopropyl benzene	heat	≤500%	21°-24°C		GC-FID	Bellar, Sigsby (Unpublished)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate / Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Scrubbers</u>							
Scrubber $\text{Hg}(\text{ClO}_4)_2$ or $\text{HgSO}_4\text{-H}_2\text{SO}_4$ on diato- maceous earth	paraffins			Room temperature must be dry		GC-FID	Mitsuo, Aoyama, Yamaki (1974)
Scrubber $\text{Hg}(\text{ClO}_4)_2$ on diatomaceous earth	acetylene						
Scrubber $\text{Hg}(\text{ClO}_4)_2$ or $\text{H}_2\text{SO}_4\text{-HgSO}_4$ on diatomaceous earth	general aromatics						
<u>Others</u>							
Sintered glass absorber	aromatic isocyanates	DMF/HCl	1 l/min		100%	Colorimetry	Meddle, Radford, Wood (1969)
Sintered glass	2-amino pyridine	H_2O	1.5 l/min		100%	Colorimetry	Watrous, Schulz (1950)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Others</u>							
Bubbler	aromatic isocyanates	.4NHCl/HOAC	2 ft ³ /hr		95%	Colorimetry	Marcali (1957)
Fritted glass bubbler	nitroalkanes 2-nitro- propane	conc. H ₂ SO ₄ silica gel 2-propanol H ₂ O	.5l/min		100 99 75 50-100 2 tubes	Colorimetry	Jones (1963)
Bubbler with glass beads	organic R-C=S com- pounds	EtOH with diethylamine	500cc/hr			Colorimetry	Viles (1940)
Glass beads	C ₆ - C ₁₀ benzene alkyl benzenes toluene xylenes	120°	72cc	liq. N ₂	2-130 ppb	GC-FID bis(m-phenoxy -phenoxy) benzene with apiezon 73°C	Lonneman, Bellar, Altshuller (1968) Lonneman, et al. (1974)
Bead-filled tube	p-chloro nitrobenzene	fuming nitric acid	1.5l/min		100%	Colorimetry	Watrous, Schulz (1950)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

Accumulator	Accumulant	Desorption or Extraction Medium	Sampling Rate Sample Volume	Collection Parameters	Recovery and Sensitivity	Associated Analytical Method	Reference
<u>Others</u>							
Folin aeration tubes	Propylene Glycol Triethylene Glycol	Water	300± 20-30±/min		70-80% 95-97 - 2 tubes	Colorimetry	Wise, Puck, Stral (1967)
Filter paper with 10% NaOH	N-butyric acid Isovaleric acid	concentrated HCl/hexane	2-6±/min		<99	GC TMCBA on Chromosorb W 139°C	Okita, et al. (1973)
Filter paper with 9% oxalic acid	methyl amine trimethyl amino						
10% sucrose acetate on Gas Chrom Z	C ₁ - C ₃ alcohols esters epoxides aldehydes	100°C		-55°C		GC-FID Porapak Q 160°C	Bellar, Sigsby (1970)

ACCUMULATION OF ORGANIC SUBSTANCES FROM AIR

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SECTION FOUR

ACCUMULATION FROM SOLIDS

4.0 Introduction

The accumulation of toxic substances from solids is a broad and ill-defined topic covering such diverse solids as soil, plant and animal tissue, sediment, and particulate matter in the air. In an effort to narrow the scope of study for this section, the solids chosen for detailed analysis are soils and plant tissue. These two areas were not chosen to imply that they are in any way more important than any other solid but, rather, they were chosen to be illustrative of all solids. The main accumulator systems applicable to solids are solvent extraction, head space analysis and biological accumulation. Biological accumulation is considered elsewhere (Section Five) in this report.

The method of toxic substance retention (ion exchange, incorporation, etc.), type of medium (organic vs. inorganic), and composition of the solid are all factors that must be considered in choosing an extractant for a particular solid. For instance, before extractions from animal tissues can be performed, it is necessary to

separate the lipids from the other tissue constituents. In contrast to this, many plant tissues are simply dry ashed before extraction.

4.1 Soils and Plants

The soil is a dynamic chemical-biological system composed of a complex matrix of inorganic matter that holds animal material, water and gases. The complexity of the soil medium can be ascertained by looking at the wide range of characteristics of its constituents. For instance, the inorganic matrix is composed of material ranging in size from meters to millimicrons, a difference of 9 orders of magnitude or more. Likewise the organic matter in the soil varies from large tree roots to viruses.

In reality the soil acts as an accumulator for many of the end products of today's society. In recent years the soil has served as a receptor for municipal wastewater, applied by spray irrigation and, when used in this manner, the soil filters out most of the water's impurities. The soil also accumulates many of the materials which fall on its surface, whether they are dusts from a smoke stack or atmospheric gases dissolved in the rain-water which falls on the earth's surface.

A clear understanding of the mechanisms by which these added materials are held in the soil is necessary to propose accumulation techniques. These retention mechanisms can be broken down into the following six areas:

- 1) Surface filtration
- 2) Ion exchange
- 3) Plant uptake
- 4) Chemical immobilization
- 5) Clay entrapment, and
- 6) Microorganism activities.

These six retention mechanisms will be examined individually in order to illustrate some of the problems which will be encountered in applying accumulator techniques to remove soil "pollutants".

Surface filtration occurs when a "pollutant" is too large to infiltrate through the soil pores. If the compound is organic in nature, the soil microorganisms will decompose it into smaller particles until it can be incorporated into the soil. If the "pollutant" is inorganic in nature, it will remain on the surface unaffected. Whether it is organic or inorganic in nature, it is susceptible to surface runoff forces as long as it remains on the soil surface.

Ion exchange is simply the reversible process by which cations and anions are exchanged between solid and liquid phases. The general mode of ion transfer in the soil is that of cation exchange, with the organic colloids and clay minerals being the primary exchange sites. Since the clay minerals and organic colloids are generally negatively charged, they attract positively charged ions, thus neutralizing the system. The numerous cations which soil colloids have adsorbed to their exchange sites are held in varying degrees of tenacity, depending upon their charges and their hydrated and unhydrated radii. As a rule ions with a valence of 2 or 3 are held more tightly than are monovalent cations. Also, the greater the degree to which the ion is hydrated, the less tightly it is held.

There are several pertinent points to consider when selecting a solvent to extract a pollutant from the soil. First, the cation exchange phenomenon is pH dependent, the lower the pH the more H^+ ions are on the exchange sites and the fewer sites that are available for exchange. Second, heavy metals of high valence with low hydration will be held more tightly on the exchange sites than will monovalent ions. Finally, in most soils, anion exchange is very limited, so that most added anions will not be held in the soil by the exchange mechanism.

Most plant nutrition occurs through the plant's extensive root system. For root uptake to occur, the nutrients must be in the ionic form. The ionization of nutrients is accomplished by the soil microorganisms, by chemical reactions caused by the root, or is spontaneous in nature. In many cases the root is "blind" to the ionic species which is being taken into the plant. In this manner the plant acts as an accumulator by absorbing the most readily accessible ions from the soil solution. Once inside the plant, the ions may be preferentially translocated from one site in the plant to another, depending on the plant's metabolism, state of development, and species.

Chemical immobilization can occur in the soil when two or more ions coprecipitate out of the soil solution. This is the process by which soil minerals are formed. When this occurs the precipitate will follow well defined solubility rules. For example, HgSe (tiemannite) is insoluble in water but soluble in aqua regia. A knowledge of mineral and toxic substance solubility is necessary to differentiate between an extraction solvent which will selectively accumulate ions which are relatively free to enter the soil solution from those which are bound up in soil minerals.

Clay entrapment can occur in instances when there are expanding clay minerals of the montmorillonite type and anions of the correct radii present. The entrapment occurs when the clay is dehydrated and the ions lose their hulls of oriented water molecules. Ions whose diameters allow them to fit well into the lattice "holes" are then entrapped in the clay micelle. Some anions which are affected by this mechanism are K^+ , NH_4^+ , Rb^+ , Cs^+ , Ba^{+2} , Sr^{+2} and Li^+ . Once these ions are taken into the clay micelle they are not easily released by most soil reactions.

Microorganisms affect the availability of ions in many ways. In general, microbial affects can be delineated into the following six general areas:

- 1) release of inorganic ions during the decomposition of organic materials
- 2) removal of inorganic ions from solution and the disappearance of the available form of the element to satisfy the nutrient demands of the microflora (immobilization)

- 3) utilization of inorganic ions as energy sources, resulting in an oxidation
- 4) reduction of an oxidized state of the element in the absence of adequate O_2 .
- 5) Indirect transformations resulting from the activities or the products of microorganisms (i.e., changes in pH or alterations in the partial pressure of O_2 by microbial respiration causing oxidation state changes)
- 6) change in the total quantity of an element in the soil by fixation, assimilation, or gas formation.

Due to the extreme heterogeneity of the soil matrix, it is essential that an adequate sampling technique be derived so that the actual extraction is representative of the soil chemical composition. Interpretation of the extraction technique should also consider the possibility that some elements which are immobile in soil solutions, and therefore will not enter into the environment, will become quite mobile in the extractant. An example of this is the breakdown of minerals and release of ions by acid extraction.

A compendium of the literature relating to the accumulation of toxic substances from soils and plant

tissues is presented in Tables 4-7 through 4-10. The material is broken down into four areas -- accumulation of both organic and inorganic material from both plant tissue and from soil.

4.2 Accumulation by Extraction

In general, the extraction of inorganic material from soils is accomplished by leaching the soil with the salt of an acid or a chelating compound. This technique replaces the available ions from the soil exchange sites. Frequently ammonium acetate, sodium chloride or EDTA are used for this purpose. If a total digestion of soil inorganic matter is required, a strong acid - either H_2SO_4 or HClO_4 - is used, and the soil is frequently refluxed. The effectiveness of the accumulation process generally depends on the soil pH, soil texture, amount of extractant applied, and the organic content of the soil.

Inorganic nutrients are generally extracted from plant tissue after the tissue has undergone some pre-treatment such as macerization or dry ashing. The most frequently used extractants are acetonitrile and strong acids. Likewise, the accumulation of organic matter

from plant tissue generally follows the pretreatment of the sample.

The choice of extractant for organic accumulations is much more varied due to the diverse nature of the organic constituents. In Tables 4-9 and 4-10 the organic material is differentiated into nine areas.

While these nine areas are specifically all subdivisions of pesticide analyses, in the more general sense they are representative of all organic matter in the soil. Most soil and plant analyses for organic matter have been performed to determine the fate and metabolic pathways of applied pesticides. Since the chemical nature of pesticides is closely related to many toxic substances of interest, the extraction of these toxic lipophilic substances from soil will parallel the pesticide extractions. This can be verified by comparison of pesticide and toxic substance extractions from both air and water. The following list shows the grouping and possible extractants for each area:

1. Organochlorine pesticides - hexane: acetone
2. Organophosphorus pesticides - acetone or acetone: isopropanol
3. Acidic pesticides - KCl or ethyl ether
4. Triazine pesticides - chloroform or acetonitrile

or methanol: H₂O

5. Carbamate pesticides - polar: nonpolar solvent mixtures
6. Uracil pesticides - NaOH
7. Anilide and aniline pesticides - benzene or acetone
8. Benzonitrile and amide pesticides - benzene: isopropanol
9. Dipyridinium pesticides - strong acids

The choice of extractant is dependent on the type of organic matter desired, the moisture content of the sample, the pH of the sample, and the equilibration time required for sampling.

4.3 Headspace Analysis

Headspace analysis of soil samples can be accomplished by heating samples to 70°C, thus causing the volatilization of the various hydrocarbon species which are present. The vapors are then swept into a Tenax trap by a stream of purified nitrogen, and the trap is subsequently analyzed by devolatilization into a gas chromatographic column, as described in the section on the accumulation of organic matter from water (Section 2.1).

Compounds with volatilities less than octadecane and pyrene can be quantitatively analyzed under these conditions. The advantages of this headspace analysis technique for solid samples, as compared with extraction analysis, are a minimization of contamination, an increase in analysis speed, and the potential for automation.

TABLE 4-1

COMMON AND CHEMICAL NAMES OF ORGANOCHLORINE PESTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
Aldrin	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4- <u>endo-exo</u> -5,8-dimethanonaphthalene
Aroclors	Polychlorinated biphenyls
α -, β -, δ -and γ -BHC	α -, β -, δ - and γ -isomers of 1,2,3,4,5,6-hexachlorocyclohexane
Chlordane	1,2,3,4,5,6,7,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methanoindane
p,p' -DDE	1,1-dichloro-2,2-bis-(p -chlorophenyl) ethene
p,p' -DDT	1,1,1-trichloro-2,2-bis-(o -chlorophenyl) ethane
p,p' -DDT	1,1,1-trichloro-2,2-bis-(p -chlorophenyl) ethane
Dieldrin	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4- <u>endo-exo</u> -5,8-dimethanonaphthalene
Endosulfan	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide
Endrin	1,2,3,4,10,10-hexachloro-6,7,-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4- <u>endo-endo</u> -5,8-dimethanonaphthalene
Heptachlor	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene
Heptachlor epoxide	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-2,3-epoxy-4,7-methanoindene
p,p' -methoxychlor	1,1,1-trichloro-2,2-bis-(p -methoxyphenyl) ethane
TDE (DDD)	1,1-dichloro-2,2-bis-(p -chlorophenyl) ethane
Toxaphene	Chlorinated camphene containing 67 to 69% chlorine

TABLE 4-2

COMMON AND CHEMICAL NAMES OF ORGANOPHOSPHORUS INSECTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
Disulfoton	0,0-diethyl S-[2-(ethylthio)ethyl] phosphorodithioate
Dursban	0,0-diethyl-0-3,5,6-trichloro-2-pyridyl phosphorothioate
Dyfonate	0-ethyl S-phenyl ethylphosphonodithioate
Hinosan	0-ethyl S,S-diphenyl phosphorodithioate
Kitazin P	0,0-diisopropyl S-benzyl phosphorothioate
Methidathion	0,0-dimethyl S-(N-formyl-N-methylcarbo- myl/methyl) phosphorodithioate
Mevinphos	Mixture of isomers of methyl-3-hydroxycro- tonate dimethylphosphate
Phorate	0,0-diethyl S-(ethylthiomethyl) phosphorodithioate
POA (phoratoxon)	Oxygen analog of phorate
PSO	Sulfoxide analog of phorate
PSO ₂	Sulfone analog of phorate
POASO	Oxygen analog of the sulfoxide of phorate
POASO ₂	Oxygen analog of the sulfone of phorate
Zinophos	0,0-diethyl 0-2-pyrazinyl phosphorothioate

TABLE 4-3

COMMON AND CHEMICAL NAMES OF CARBAMATE, SUBSTITUTED UREA,
URACIL, BENZONITRILE, ANILIDES, ANILINES AND AMIDE PESTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
<u>CARBAMATES</u>	
Aldicarb	2-methyl-2(methylthio)propionaldehyde O-(methylcarbamoyl)oxime
Carbofuran	2,3,-dihydro-2,2-dimethyl-7-benzofuranyl- methylcarbamate
Chlorpropham (CIPC)	Isopropyl N-(3-chlorophenyl) carbamate
Propham (IPC)	Isopropyl N-phenylcarbamate
Triallate	S-2,3,3-trichloroallyl N,N-diisopropyl- thiolcarbamate
<u>URACILS</u>	
Bromacil	5-bromo-3- <u>sec</u> -butyl-6-methyluracil
Terbacil	3- <u>tert</u> -butyl-5-chloro-6-methyluracil
<u>ANILIDES and ANILINES</u>	
Alachlor	2-chloro-2',6'-diethyl-N-(methoxymethyl) acetanilide
DCA	3,4-dichloroaniline
DCNA	2,6-dichloro-4-nitroaniline
Propanil	3,4-dichloropropionanilide
TCAB	3,3',4,4'-tetrachloroazobenzine
<u>BENZONITRILES and AMIDES</u>	
Dichlobenil	2,6-dichlorobenzonitrile
2,6-DCBA	2,6-dichlorobenzoic acid
KERB	N-(1,1-dimethylpropynyl)-3,5-dichloro- benzamide

TABLE 4-4

COMMON AND CHEMICAL NAMES OF TRIAZINE AND DIPYRIDINIUM PESTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
<u>TRIAZINES</u>	
Atrazine	2-chloro-4-ethylamino-6-isopropylamino- <u>s</u> -triazine
Hydroxyatrazine	2-hydroxy-4-ethylamino-6-isopropylamino- <u>s</u> -triazine
Prometryne	2,4-bis(isopropylamino)-6-methylmercapto- <u>s</u> -triazine
Propazine	2-chloro-4,6-bis(isopropylamino)- <u>s</u> -triazine
<u>DIPYRIDINIUM PESTICIDES</u>	
Paraquat	1,1'-dimethyl-4,4'-dipyridinium salt usually dichloride or dimethylsulfate

TABLE 4-5

COMMON AND CHEMICAL NAMES OF ACIDIC PESTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
2,4-D	2,4-dichlorophenoxyacetic acid
Picloram	4-amino-3,5,6-trichloropicolinic acid
2,4,5-T	2,4,5-trichlorophenoxyacetic acid

TABLE 4-6

COMMON AND CHEMICAL NAMES OF SOME MISCELLANEOUS PESTICIDES

<u>Common Name</u>	<u>Chemical Name</u>
Amitrole	3-amino-S-triazole
Oryzemat	3-allyloxy-1,2-benzisothiazole 1,1-dioxide
Trifluralin	a,a,a-trifluoro-2,6-dinitro-N-N-dipropyl- p-toluidine

TABLE 4-7

INORGANIC ACCUMULATION FROM SOIL

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Al	CaCl ₂		Time, pH	Available Al	.7-38.9		Hoyt & Nyborg (1971)
Al	NaCl		pH	Available Al	.7-700		Hoyt & Nyborg (1971)
Al	KCl		pH	Available Al	2.0-586		Hoyt & Nyborg (1971)
Al	NH ₄ OAc		pH	Available Al	28.8-1,122		Hoyt & Nyborg (1971)
Al	HCl		pH	Available Al	.45-11.9		Hoyt & Nyborg (1971)
Al	Dithionate-Citrate-Bicarbonate		pH, Fe, PO ₄	Total Al	0.5	92-100	McKeague et al. (1971); Arshad et al. (1972)
Al	Ammonium Oxalate		pH, Fe, PO ₄	Amorphous inorganic and organic Al	0.1	98-104	McKeague et al. (1971); Arshad et al. (1972)
Al	Na ₄ P ₂ O ₇		pH, Fe, PO ₄	Organic complexed Al	0.6	92-100	McKeague et al. (1971); Arshad et al. (1972)
Al	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5-29,700		Massey & Barnhisel (1972)
Al	H ₂ O				0.1-0.6		Bradford et al. (1971)
Al	NH ₄ F		pH		105.0-4500		Tandon (1970)
As	HCl	Head space analysis			3.3-15.3	93-106	Melton et al. (1973)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Ba	H ₂ O				1.0-2.4		Bradford et al.(1971)
B	H ₂ O				2.0		Bradford et al.(1971)
B	hot H ₂ O	Special apparatus	Time dependent		0.7-4.1		John (1973)
Ca	H ₂ O				1.0-930		Bradford et al.(1971)
Ca	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Cd	HCl			Extractable only. A reference to which the amount of ions absorbed by plant is related			Lagerwerff (1971)
Co	H ₂ O			Water soluble only	.01-.14		Bradford et al.(1971)
Co	DPTA*-Sodium Acetate-CaCl ₂	Shake	pH, O/R	Labile pool nutrients	0.5		Lopez & Graham (1970)
*DPTA = Diethylene triamine pentaacetic acid							

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Co	NaOAc	Shake	pH	Available nutrients	0.2		Lopez & Graham (1972)
Cr	H ₂ O			Water soluble only	0.01-.017		Bradford et al. (1971)
Cu	H ₂ O			Water soluble only	0.01-.20		Bradford et al. (1971)
Cu	DPTA			Available Cu	0.2-3.2		Follett & Lindsay (1971)
Cu	DPTA-Sodium Acetate-CaCl ₂		pH, O/R	Labile pool nutrients	0.5		Lopez & Graham (1970)
Cu	NaOAc		pH	Available nutrients	0.1		Lopez & Graham (1972)
Cu	HCl		time, pH, organic matter phosphorus	Parent material, exchangeable Cu and Zn	1.6		Macias (1973)
Cu	Ammonium Acetate		time, pH, organic matter phosphorus	Parent material, exchangeable Cu and Zn	0.5		Macias (1973)
Cu	Ammonium oxalate		time, pH, organic matter phosphorus	Parent material, exchangeable Cu and Zn	2.6		Macias (1973)
Cu	Citric acid		time	Parent material, exchangeable Cu and Zn	0.7		Macias (1973)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Cu	EDTA*			Available nutrients	0.2-9.7		Haq and Miller (1972)
Cu	DTPA			Available nutrients	0.2-9.7		Haq and Miller (1972)
Cu	H ₂ O			Does not reflect uptake by plant			Roth et al. (1971)
Cu	KCl			Does not reflect uptake by plant			Roth et al. (1971)
Cu	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Fe	Dithionite-citrate-bicarbonate			Total Fe			McKeague et al. (1971)
Fe	Ammonium oxalate			Amorphous, inorganic, and organic Fe			McKeague et al. (1971)
Fe	Na ₄ P ₂ O ₇			Organic complexed Fe			McKeague et al. (1971)
Fe	H ₂ O			Water soluble only	0.01-.8		Bradford et al. (1971)
Fe	DPTA			Available Fe	1.7-168.3		Follett & Lindsay (1971)
Fe	CaCl ₂	Shake	pH	Nutrients available for plant growth	0.1		Lopez & Graham (1970)
*EDTA = ethylenediamine tetraacetic acid							

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Fe	DPTA-Sodium Acetate-CaCl ₂	Shake	pH, O/R	Labile pool nutrients	0.5		Lopez & Graham (1970)
Fe	NaCl, Sodium citrate, Citric acid, Sodium dithionite			Removes free Fe and MnO ₂ from clay			Anderson & Jeanne (1970)
Fe	HCl		Organic matter	Available Fe. Varying importance of total nutrient level and pH			Sorenson et al. (1971)
Fe	NaOAc		pH	Available nutrients	1.7		Lopez & Graham (1972)
Fe	EDHA-NaNO ₃	Chelate	Time, organic matter	Labile Fe	1.0		Johnson & Young (1973)
Fe	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Hg	H ₂ O			Water soluble only	.0002-.019		Bradford et al. (1971)
Hg	BaCl ₂		pH, Cl ⁻	Hg (II)	0.7		Hahne & Kroontje (1973)
Hg	HNO ₃ -H ₂ SO ₄ -K ₂ S ₂ O ₈			Total Hg	3.2-168	97-102.5	Melton et al. (1971)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
K	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
K	H ₂ O			Water soluble only	0.7-128		Bradford et al. (1971)
Li	H ₂ O			Water soluble only	0.03-1.08		Bradford et al. (1971)
Mg	H ₂ O			Water soluble only	0.4-400		Bradford et al. (1971)
Mg	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Mn	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Mn	H ₂ O		pH		0.2		Gupta (1972)
Mn	NaOAc		pH	Available nutrients	0.2		Lopez & Graham (1972)
Mn	CaCl ₂ solution	Shake	pH	Nutrients available for plant growth	0.1		Lopez & Graham (1970)
Mn	DPTA-Sodium Acetate-CaCl ₂	Shake	pH, O/R	Labile pool nutrients	0.5		Lopez & Graham (1970)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Mn	NaCl, Sodium citrate, Citric acid, Sodium dithionite			Removes free iron and MnO ₂ from clay			Anderson & Jenne (1970)
Mn	HCl		Organic matter	Available Mn. Varying importance of total nutrient level, pH			Sorenson et al. (1971)
Mn	KCl		pH	Exchangeable pH dep.	2.6-99.4		Hoyt & Nyborg (1971)
Mn	NH ₄ OAc			Exchangeable	4.6-111.9		Hoyt & Nyborg (1971)
Mn	Hydroquinone-NH ₄ OAc			Reducible	2.7-266.9		Hoyt & Nyborg (1971)
Mn	CaCl ₂			Soluble	3.0-68.5		Hoyt & Nyborg (1971)
Mn	HOAc			Soluble-plant available Mn	5.1-78.4		Hoyt & Nyborg (1971)
Mn	H ₃ PO ₄			Soluble	12.9-112.8		Hoyt & Nyborg (1971)
Mn	HCl			Soluble	0.2-31.7		Hoyt & Nyborg (1971)
Mn	DPTA			Available Mn	3.9-111.6		Follett & Lindsay (1971)
Mn	EDTA			Available nutrients	4.4-40.0		Haq and Miller (1972)
Mn	DPTA			Available nutrients	1.6-40.0		Haq and Miller (1972)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Mn	H ₂ O			Water soluble only	0.01-.95		Bradford et al.(1971)
Mo	H ₂ O			Water soluble only	0.01-22.0		Bradford et al. (1971)
Mo	H ₂ O: Dowex (1-X4) resin: NaCl		pH		0.2		Bhella & Dawson (1972)
Na	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Na	H ₂ O			Water soluble only	0.9-19,200		Bradford et al. (1971)
Ni	H ₂ O			Water soluble only	0.01-0.09		Bradford et al. (1971)
Ni	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Ni	H ₂ O			Does not reflect uptake by plant			Roth et al. (1971)
Ni	KCl			Does not reflect uptake by plant			Roth et al. (1971)
P	NaHCO ₃				7.5		Carter et al. (1972)
P	HClO ₄	Heat		Total P	423		Sommers & Nelson (1972)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
P	Sodium arsenate		$\text{PO}_4^{=}$, pH, temperature				Barrow (1974)
Pb	HCl			Extractable only. A reference to which the amount of ions absorbed by plant is related.			Lagerwerff (1971)
Pb	H_2O			Water soluble only	0.01-.30		Bradford et al. (1971)
S	CaCl_2 solution			Soluble SO_4			Cowling & Jones (1970)
S	KH_2PO_4 solution			Absorbed SO_4			Cowling & Jones (1970)
Si	H_2O			Water soluble only	0.2-24.0		Bradford et al. (1971)
Sr	NH_4Cl		pH	Does not remove nonexchangeable Sr			Juo & Barber (1970)
Sr	H_2O			Water soluble only	0.1-10.4		Bradford et al. (1971)
Ti	H_2O			Water soluble only	0.1		Bradford et al. (1971)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
V	H ₂ O			Water soluble only	0.01-1.20		Bradford et al.(1971)
Zn	H ₂ O			Water Soluble only	0.01-.40		Bradford et al.(1971)
Zn	H ₂ O	Pressure plate	pH	From shale and spoil material	0.5		Massey & Barnhisel (1972)
Zn	CaCl ₂ solution	Shake	pH	Nutrients available for plant growth	0.1		Lopez & Graham (1970)
Zn	CaCl ₂		pH, clay content, organic matter		0.2		John (1972)
Zn	DPTA-CaCl ₂		pH	Available nutrients	0.4		Lopez & Graham (1972)
Zn	DPTA-TEA*CaCl ₂		pH, clay content, organic matter		1.4		John (1972)
Zn	DPTA-Sodium Acetate-CaCl ₂	Shake	pH, O/R	Labile pool nutrients	0.5		Lopez & Graham (1970)
Zn	DPTA		Soil texture		0.3		Alley et al. (1972); Brown et al. (1971)
Zn	DPTA			Available nutrients	0.2-9.7		Haq and Miller (1972)
Zn	DPTA			Available Zn	0.6-9.0		Follett & Lindsay (1971)
Zn	EDTA			Available nutrients	0.2-9.7		Haq and Miller (1972)
*TEA = Triethanolamine							

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Zn	EDTA		Soil texture		0.2		Alley et al.(1972); Brown et al.(1971)
Zn	EDDHA*			Available nutrients	2.0-7.5		Haq and Miller (1972)
Zn	HCl		Soil texture		0.5		Alley et al.(1972) Brown et al.(1971)
Zn	HCl		time, phosphorus, organic matter	Parent material. Exchangeable Cu and Zn	4.2		Macias (1973)
Zn	HCl			Extractable only. A reference to which the amount of ions absorbed by plant is related.			Lagerwerff (1971)
Zn	HCl		Organic matter content	Available Zn. Varying importance of total nutrient level and pH			Sorenson et al.(1971)
Zn	HCl-H ₂ SO ₄		pH, clay content, organic matter		2.8		John (1972)
Zn	HCl-H ₂ SO ₄		Soil texture		0.6		Alley et al.(1972)
Zn	HCl-H ₂ SO ₄			Available nutrients	0.0-4.4		Haq and Miller (1972)
Zn	HNO ₃ -HF-HClO ₄ -HCl			Total zinc	1.6		John (1972)
Zn	MgCl ₂		pH, clay content, organic matter		1.6		John (1972)

*EDDHA = Ethylene diamine di(O-hydroxyphenyl acetic acid)

INORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Interferences	Comments	Concentration (ppm)	% Recovery	Reference
Zn	NH ₄ OAc		pH, type of clay	Available Zn			Reddy & Perkins (1974)
Zn	NH ₄ OAc		pH, clay content, organic matter		2.0		John (1972)
Zn	NH ₄ OAc		Time, pH, organic matter phosphorus	Parent material. Exchangeable Cu and Zn	1.2		Macias (1973)
Zn	Ammonium oxalate		Time, pH, organic matter phosphorus	Parent material. Exchangeable Cu and Zn	5.3		Macias (1973)
Zn	Citric Acid		Time	Parent material. Exchangeable Cu and Zn	4.2		Macias (1973)
Zn	NaOAc		pH, clay content, organic matter		0.96		John (1972)

TABLE 4-8

INORGANIC ACCUMULATION FROM PLANTS

INORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
Al	HCl	Dry ash first	Plant tissue		28 ppm		Hoyt and Nyborg (1971)
Al	HCl	Ignite sample first	Plants				AOAC 1 (1970)
As	HNO ₃ : H ₂ SO ₄ : H ₂ O: NH ₄ Oxalate		Plants, food				AOAC 1,2 (1970)
As	HNO ₃ : H ₂ SO ₄		Food				AOAC 2 (1970)
B	Ca(OH) ₂ : H ₂ SO ₄	Dry after Ca(OH) ₂ addition	Plants				AOAC 1 (1970)
Ca	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Cd	H ₂ SO ₄ -HNO ₃		Food				AOAC 2 (1970)
Cl	Na ₂ CO ₃	Ignition	Plants				AOAC 1 (1970)
Co	H ₂ O: HClO ₄ , HF	Oven dry: grind: ash dry first	Plants				AOAC 1 (1970)
Cu	HCl	Dry ash first	Citrus roots		10 ppm		Brams and Fiskell (1971)

INORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
Cu	H ₂ SO ₄ -HNO ₃		Food				AOAC 2 (1970)
Cu	HNO ₃	Dry ash first	Grass	pH dependent			Bohn and Aba-Husayn (1971)
Cu	NH ₄ Citrate: NH ₄ OH: Dithi- zone in CCl ₄		Plants				AOAC 1 (1970)
Cu	HNO ₃ : HCl	Dry ash first	Alfalfa plant	Total Cu determin- ed	8 ppm		Baker (1971)
Cu	H ₂ O: HNO ₃ : HClO ₄		Alfalfa plant	No pretreatment necessary	9 ppm	96-100	Baker (1971)
Fe	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Fe	HNO ₃	Dry ash first	Grass	pH dependent			Bohn and Aba-Husayn (1971)
Fl	Ca(OH) ₂	Dry after Ca(OH) ₂ addi- tion	Food				AOAC 2 (1970)
Hg	HNO ₃		Plants and food	Total Hg	0.01 ppb	95-102	Hoover, Melton, and Howard (1971)
Hg	HNO ₃ : HNO ₃ - H ₂ SO ₄		Food	Special apparatus			AOAC 2 (1970)

INORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
K	H ₂ SO ₄ : HCl		Plants				AOAC 1 (1970)
Mg	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Mn	MgO-H ₂ O: HCl	Dry ash after MgO-H ₂ O	Barley, Rape, Alfalfa		47-1658 ppm		Hoyt and Nyborg (1971)
Mn	HNO ₃	Dry ash first	Grass	pH dependent			Bohn and Aba-Husayn (1971)
Mn	HCl	Dry ash first	Wheat, alfalfa, corn, sorghum		.1 ppm	87.0-103.5	Smith and Schrenk (1972)
Mn	HNO ₃ : HClO ₄ : HCl		Wheat, alfalfa, corn, sorghum	Requires extra attention	.1 ppm		Smith and Schrenk (1972)
Mn	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Mo	HNO ₃ : HClO ₄		Plants				AOAC 1 (1970)
Mo	HClO ₄		Clover	pH dependent	0.6 ppm		Ehella and Dawson (1972)
Na	H ₂ SO ₄ :HCl		Plants				AOAC 1 (1970)

INORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
P	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Pb	HCl	Dry ash first	Food				AOAC 2 (1970)
Pb	HNO ₃ : HClO ₄		Corn and alfalfa plants		12 ppm		Lagerwerff, Armiger, and Specht (1973)
S	Na ₂ CO ₃ : H ₂ O: Na ₂ O ₂	Fusion	Plants				AOAC 1 (1970)
Sb	HNO ₃ : H ₂ SO ₄		Food				AOAC 2 (1970)
Se	H ₂ SO ₄ -HNO ₃	Grind sample first	Plants				AOAC 1 (1970)
Se	HgO fixative: H ₂ SO ₄ -HNO ₃		Food				AOAC 2 (1970)
Sn	HNO ₃ : H ₂ SO ₄		Food				AOAC 2 (1970)
Zn	HCl	Ignite sample first	Plants				AOAC 1 (1970)
Zn	HNO ₃ : HClO ₄ : HCl		Wheat, alfalfa, corn, sorghum	Requires extra attention	.03 ppm		Smith and Schrenk (1972)

INORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
Zn	HCl	Dry ash first	Wheat, alfalfa, corn, sorghum		.03 ppm	89.5-106.5	Smith and Schrenk (1972)
Zn	HNO ₃ : H ₂ SO ₄ : HClO ₄		Food				AOAC 2 (1970)
Zn	HNO ₃	Dry ash first	Grass	pH dependent			Bohn and Aba-Husayn (1971)
Zn	HNO ₃ : HClO ₄		Corn and oat grain		10.4 ppm		John (1972)

TABLE 4-9

ORGANIC ACCUMULATION FROM SOIL

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
<u>ACIDIC PESTICIDES</u>						
2,4-D	H ₂ SO ₄ , H ₂ O, ETHYL ETHER	Tumbling	-	.013 to 1.3 ppm	85-100	Wodham, et al. (1971)
PICLORAM	KCl - KOH	Shaking	18 hours equilibrium	-	94-100	Cheng (1971)
PICLORAM	ACETONE - H ₃ PO ₄	Stirring	-	-	-	Biggar and Cheng (1973)
PICLORAM	ACETONE - WATER HCl	Stirring	-	.025 to .1 ppm	75	Metcalf (1972)
2,4,5-T	H ₂ O ETHYL ETHER	Shaking	-	4 ppm	-	Burge (1973)
<u>ANILIDE & ANILINE PESTICIDES</u>						
ALACHLOR	BENZENE	Shaking	-	0.1 to 10 ppm	95	Hargrove and Merkle (1971)
DCA	ACETONE	Blending	-	-	~60	Chisaka and Kearney (1970)
DCA	ETHYL ALCOHOL	Shaking	-	50 ppm	-	Burge (1973)
DCNA	HCl: ACETONE: GLYCOL	Shaking	Sterilize 1-66 days	15.1 ppm	68-76	Greve and Wit (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
PROPANIL	ACETONE	Blending	-	-	>90	Chisaka and Kearney (1970)
PROPANIL	ETHANOL	Shaking	-	100 ppm	97-108	Burge (1973)
PROPANIL	ACETONE: BENZENE	Blending	-	-	90	Kearney, et al. (1970)
TCAB	ACETONE	Blending	-	-	>90	Chisaka and Kearney (1970)
TCAB	ACETONE: BENZENE	Blending	-	-	98	Kearney, et al. (1970)
TCAB	ETHANOL	Shaking	-	.05 ppm	-	Burge (1973)
<u>BENZONITRILE AND AMIDE PESTICIDES</u>						
DICHOLOBENIL	ETHANOL	Boiling	0.5-12 month equilibrium	2.0 ppm	90	Verloop and Nimmo (1970)
DICHOLOBENIL	BENZENE: ISOPROPANOL	Shaking	-	.07-.08 ppm	81-105	Skroch, et al. (1971)
2,6-DCBA	ETHANOL	Boiling	1-6 month equilibrium	2.0 ppm	90	Verloop and Nimmo (1970)
KERB	METHANOL	Soxhlet	34 day equilibrium	20 ppm	38-100	Yih, et al. (1970)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
KERB	H ₂ SO ₄ - METHANOL	-	-	.01 ppm	-	Adler, et al. (1972)
<u>CARBAMATE PESTICIDES</u>						
ALDICARB	ACETONE: METHANOL AND CHLOROFORM: ACETONITRILE	Shaking	0,1,7 day equilibrium	20 ppm	85-100	Bull, et al. (1970)
CARBOFURAN	HCl	Refluxing	-	0.2-100 ppm	71-115	Butler and McDonough (1971)
3-KETO CARBOFURAN	HCl	Refluxing	-	0.1-10 ppm	76-98	Butler and McDonough (1971)
3-HYDROXYCARBO- FURAN	HCl	Refluxing	-	0.2-100 ppm	72-119	Butler and McDonough (1971)
CIPC	ETHANOL	Shaking	-	100 ppm	92-100	Burge and Gross (1972)
EPTC	ISOOCTANE	Steam distill	1 day equilibrium	-	>90	Smith and Fitzpatrick (1971)
IPC	ETHANOL	Shaking	-	100 ppm	94-100	Burge and Gross (1972)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
TRIALATE	BENZENE: ISOPROPANOL	Shaking		-	95	Smith (1971)
TRIALATE	BENZENE: ISOPROPANOL	Shaking	1 day equilibrium	-	>90	Smith and Fitzpatrick (1971)
<u>DIPYRIDINIUM PESTICIDES</u>						
PARAQUAT	H ₂ SO ₄	Refluxing	-	-	>80	Earnest (1971)
<u>MISCELLANEOUS PESTICIDES</u>						
AMITROLE	NH ₄ OH: GLYCOL	Shaking	1 to 17 day equilibrium	2.0 ppm	68-97	Grove and Chough (1971)
TRIFLURALIN	HEXANE: ACETONE	Sonified	-	0.1 ppm	-	Parr and Smith (1973)
TRIFLURALIN	BENZENE: ISOPROPANOL	Shaking	1 day equilibrium	0.1 to 1.0 ppm	89-99	Smith (1972)
TRIFLURALIN	METHANOL	Tumbling	2 day equilibrium	0.1 to 2.5 ppm	61-74	Harrison and Anderson (1970)
TRIFLURALIN	METHANOL	Tumbling	0-7 day equilibrium	6.0 ppm	84-95	Harrison and Anderson (1970)
<u>ORGANOCHLORINE PESTICIDES</u>						
ALDRIN	HEXANE: ACETONE	Shaking	-	.05-.1 ppm	90-100	Saha and Sumner (1971)
ALDRIN-DIELDRIN	HEXANE: ACETONE	Shaking	-	.005 to .015	38-81	McCaskill, et al. (1970)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
ALDRIN-DIELDRIN	HEXANE: ISOPRO- PANOL	Tumbling	-	-	88-102	Onsager, et al. (1970)
ALDRIN-DIELDRIN	ACETONITRILE	Blending	-	-	90-95	Lichtenstein (1970)
ALDRIN, DIELDRIN HEPTACHLOR, HEPTACHLOR EPOX- IDE	HEXANE	Grinding	-	0.1-0.4 ppm	90-105	Grussendorf (1970)
AROCHLOR	ACETONE: PETROLEUM ETHER	Soxhlet	-	-	>80	Duke, et al. (1970)
γ-BHC	HEXANE: ACETONE	Shaking	4 day equilibrium	.001 ppm	87-92	Browman (1971)
γ-BHC	HEXANE: ACETONE	Soxhlet	1.5 hour equilibrium	50 ppm	79-104	Adams and Li (1971)
γ-BHC	HEXANE: ACETONE	Soxhlet	14 days	9-10 ppm	90-99	Guenzi and Beard (1970)
γ-BHC	ACETONE	Polytron	1 month equilib- rium	2.0 ppm	94-99	Johnsen and Starr (1972)
CHLORDANE	ACETONE	Polytron	-	2.0 ppm	93	Johnsen and Starr (1972)
CHLORDANE	WATER: HEXANE- ACETONE	Shaker	-	≈1 ppm	-	Saha (1971)
CHLORDANE	WATER: HEXANE- 2-PROPANOL	Shaker	-	1 ppm	-	Saha (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
CHLORDANE	WATER: METHANOL	Shaker	-	1 ppm	-	Saha (1971)
CHLORDANE	WATER: BENZENE-METHANOL	Shaker	-	1 ppm	-	Saha (1971)
α -, γ -CHLORDANE HEPTACHLOR HEPTACHLOR EPOXIDE	BENZENE: ACETONE	Shaking	-	0.1 ppm	85-94	Dorough, et al. (1972)
p, p' -DDE	HEXANE: ACETONE	Shaking	8 day equilibrium	0.32 ppm	84-99	Browman (1971)
p, p' -DDE; o, p' -DDT; p, p' -DDT	ACETONE	Polytron	1 month equilibrium	2 ppm	92-105	Johnsen and Starr (1972)
p, p' -DDE; o, p' -DDT; p, p' -DDT	HEXANE: ACETONE	Shaking	-	.005-.015 ppm	38-81	McCaskill, et al. (1970)
p, p' -DDE; o, p' -DDT; p, p' -DDT	HEXANE: ISOPROPANOL	Tumbling	-	-	88-102	Onsager, et al. (1970)
p, p' -DDE; o, p' -DDT; p, p' -DDT	HEXANE: ACETONE	Shaking	-	.05 - .1 ppm	90-100	Saha and Sumner (1971)
p, p' -DDT	HEXANE: ACETONE	Shaking	4 day equilibrium	0.1 - 0.2 ppm	88-112	Browman (1971)
o, p' -DDT	HEXANE: ACETONE	Shaking	8 day equilibrium	0.43 ppm	77-111	Browman (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
α, ρ' -DDT ρ, ρ' -TDE	HEXANE: ISOPRO- PANOL	Soxhlet	31 days	1.2 ppm	87-102	Burge (1971)
ρ - ρ' -DDT	BENZENE: ISOPRO- PANOL	Shaking	-	-	90	Swoboda, et al. (1971)
ρ - ρ' -DDT	HEXANE: ACETONE	Soxhlet	30 day equili- brium	-	70-102	Peterson, et al. (1971)
ρ - ρ' -DDT	HEXANE: ACETONE: CH ₃ COONa	Shaking	-	-	93	Willis, et al. (1971)
ρ - ρ' -DDT	HEXANE: ACETONE	Soxhlet	14 day equili- brium	10.0 ppm	95-100	Guenzi and Beard (1970)
DIELDRIN	HEXANE: ISOPRO- PANOL	Soxhlet	-	-	92-95	Caro and Taylor (1971)
DIELDRIN	ACETONE	Polytron	1 month equili- brium	2.0 ppm	92-105	Johnsen and Starr (1972)
DIELDRIN	ACETONE	Polytron	-	0.2 ppm	96±2.2	Johnsen and Starr (1972)
DIELDRIN	HEXANE: ACETONE	Shaking	4 day equilibrium	.04-.08 ppm	87-91	Browman (1971)
DIELDRIN, HEPTACHLOR EPOXIDE	ACETONE	Ultrasound	-	1-2 ppm	92-99	Johnsen and Starr (1972)
γ - and β ENDO- SULFAN	PETROLEUM: ETHER	Heat	-	0.2-0.3 ppm	87-89	Greve and Wit (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
ENDRIN	HEXANE: ISOPRO- PANOL	Shaking	1.5-2 day equili- brium	-	97-99	Saha and Sumner (1971)
HEPTACHLOR	PENTANE: ACETONE	Tumbling	-	-	92-98	McCaskill, et al. (1970)
HEPTACHLOR	HEXANE: ACETONE	Shaking	8 day equilibrium	0.13 ppm	-	Browman (1971)
HEPTACHLOR EPOXIDE	HEXANE: ACETONE	Shaking	8 day equilibrium	0.27 ppm	87-123	Browman (1971)
HEPTACHLOR EPOXIDE	ACETONE	Polytron	1 month equili- brium	2.0 ppm	94-99	Johnsen and Starr (1972)
HEPTACHLOR EPOXIDE- HEPTACHLOR	ACETONITRILE	Blending	-	-	90-95	Lichtenstein (1970)
METHOXYCHLOR	HEXANE: ACETONE	Shaking	8 day equilibrium	2.6 ppm	41-97	Browman (1971)
METHOXYCHLOR	ACETONE	Polytron	8 day equilibrium	2.0 ppm	93±4	Johnsen and Starr (1972)
TDE	HEXANE: ACETONE	Shaking	-	.05-0.1 ppm	90-100	Saha and Sumner (1971)
p,p'-TDE	HEXANE: ACETONE	Shaking	8 day equilibrium	0.6 ppm	89-108	Browman (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
TOXAPHENE	BENZENE: ISOPROPANOL	Shaking	-	-	90	Swoboda, et al. (1971)
TOXAPHENE	HEXANE: ACETONE	Soxhlet	-	-	88±2.9	Veith and Lee (1971)
MIXTURE OF 11 ORGANOCHLORINE PESTICIDES	HEXANE: ISOPROPANOL	Shaking	-	-	61±10	Hannon, et al. (1970)
<u>ORGANOPHOSPHORUS PESTICIDES</u>						
DISULFOTON	ACETONE: WATER	Blending	-	-	85-92	Menzer, et al. (1972)
DURSBAN	ACETONE	Tumbling	-	0.1 ppm	110	Düsch, et al. (1970)
DYFONATE	HEXANE OR CHLOROFORM	Shaking	-	-	86-88	Kiligemani and Terriere (1971lab)
METHIDATHION	ACETONE: CaCl ₂	Shaking	-	10 ppm	97-98	Getzin (1970)
MEVINPHOS	HEXANE: Na ₂ SO ₄	Shaking	-	100 ppm	91	Burns (1971)
PHORATE	HEXANE: Na ₂ SO ₄	Shaking	-	100 ppm	98	Burns (1971)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
PHORATE	ACETONE: WATER	Blending	-	-	85-92	Menzer, et al. (1972)
PHORATE, POA, PSO	HEXANE: ACETONE	Shaking	-	-	94-96	Getzin and Shanks (1970)
PSO ₂ , POASO, POASO ₂	HEXANE: ACETONE	Shaking	-	-	64-77	Getzin and Shanks (1970)
ZINOPHOS	HEXANE	Soxhlet	-	-	86-91	Kiigemani and Terriere (1971lab)
<u>TRIAZINE PESTICIDES</u>						
ATRAZINE	METHANOL	Soxhlet	1 hour mix	8.0 ppm	95	Zimdahl, et al. (1970)
ATRAZINE	ACETONITRILE: WATER	Blending	24 hour equilibrium	.05-1 ppm	71-89	Ott, et al. (1971)
ATRAZINE	ACETONITRILE: WATER	Refluxing	-	.05-2 ppm	80-114	Mattson, et al. (1970)
HYDROXYATRAZINE	HCl: ACETONITRILE: WATER	-	3 day equilibrium	-	89.5 1.5	Hance and Chesters (1970)
PROMETRYNE	TOLUENE	Shaking	-	-	75-80	Walker and Crawford (1970)

ORGANIC ACCUMULATION FROM SOIL

Accumulant	Extractant	Special Procedures	Comments	Concentration	% Recovery	Reference
PROPAZINE	TOULENE	-	-	-	75-80	Walker and Crawford (1970)
<u>URACILS</u>						
BROMOCIL	DIL NaOH	Shaking, blending	-	80 ppm	87	Zimdahl, et al. (1970)
TERBACIL	DIL NaOH	Shaking, blending	-	8 ppm	87	Zimdahl, et al. (1970)
TERBACIL	ETHYL ACETATE	Shaking	-	0.4-0.45 ppm	80-112	Skroch, et al. (1971)
<u>NON-PESTICIDES</u>						
POLYCYCLIC AROMATIC HYDROCARBONS	METHANOL: BENZENE	Soxhlet	-	~8-3600 ppb	39-88	Giger and Blumer (1974)

TABLE 4-10

ORGANIC ACCUMULATION FROM PLANTS

ORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
<u>BENZONITRILE PESTICIDES</u>							
KERB	H ₂ SO ₄ - METHANOL	homogenize first	CROPS	-	.01 ppm	-	Adler, et al. (1972)
<u>MISCELLANEOUS PESTICIDES</u>							
ORYZEMATE	ACETONITRILE: n-HEXANE	filtration	RICE PLANT	-	-	-	Uchiyama, et al. (1973)
<u>ORGANOCHLORINE PESTICIDES</u>							
CYCLODIENE PESTICIDE RESIDUES	HEXANE-ACE- TONE-MENTHA- NOL-H ₂ O	SOXHELET	SOYBEAN PLANTS	2-ENDRIN and 2-HEPTACHLOR RESIDUES	-	-	Nash and Beall (1971)
ORGANO- CHLORINE PESTICIDE RESIDUES	ACETONITRILE	chopped and mixed first	KALE, COLLARDS AND POTATO	7 PESTICIDES and RESIDUES	0.2-20 ppm	56-107	Burke, et al. (1971)
ORGANO- CHLORINE PESTICIDE RESIDUES	H ₂ O- ACETONITRILE	chopped and mixed first	KALE, COLLARDS AND POTATO	7 PESTICIDES and RESIDUE. FOR DEHYDRATED SAMPLE	-	96-108	Burke, et al. (1971)

ORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
ORGANO-CHLORINE PESTICIDE RESIDUES	ETHYL ETHER- HEXANE- Na ₂ SO ₄	grind sample first	WHEAT	17 PESTICIDES and RESIDUES RAPID TECHNIQUE	.01 ppm	82-95	Levi, et al. (1972)
<u>ORGANOPHOSPHORUS PESTICIDES</u>							
HINOSAN	ACETONITRILE	macerate and homogenize first	RICE PLANT	-	-	-	Ueyama, et al. (1973)
HINOSAN	WATER-TOLUENE	pulverize first	RICE GRAIN	-	-	-	Ueyama, et al. (1973)
KITAZIN P	ACETONITRILE	macerate and homogenize first	RICE PLANT	-	-	-	Yamamoto, et al. (1973)
KITAZIN P	ACETONITRILE: H ₂ O-TOLUENE	-	RICE GRAIN	-	-	-	Yamamoto, et al. (1973)
ORGANO-PHOSPHOROUS PESTICIDE RESIDUE	ACETONITRILE	chop and blend first	CROPS AND FRUITS	41 PESTICIDES and RESIDUES	.02 ppm	81-116.	Storherr, Ott, and Watts (1971)
ORGANO-PHOSPHOROUS PESTICIDE RESIDUES	ACETONITRILE	chop and mix first	KALE, COLLARDS AND POTATO	4 PESTICIDES and RESIDUES	8-16.3 ppm	88-107	Burke, et al. (1971)

ORGANIC ACCUMULATION FROM PLANTS

Accumulant	Extractant	Special Procedures	Medium	Comments	Concentration	% Recovery	Reference
ORGANO-PHOSPHOROUS PESTICIDE RESIDUES	ETHYL ACETATE- Na_2SO_4	ice bath	FOOD AND CROPS	-	-	-	AOAC 3 (1970)
<u>MISCELLANEOUS NON-PESTICIDES</u>							
CH_3I	METHANOL	ice cold	SORGHUM AND RICE PLANT AND GRAIN	-	6.7 ppm	75.3-100	Rangaswamy, et al. (1972)

ACCUMULATION FROM SOLIDS
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SECTION FIVE

BIOACCUMULATORS

5.0 General Discussion

Radioactive isotopes, heavy metals, and organic compounds (including chlorinated organics) are all accumulated in food chains. Particular plant and animal species will selectively accumulate one or more of these substances. Due to the biochemistry of particular organisms, individual species will be able to selectively resist accumulation of substances which are not required by particular cells for metabolism. The availability of other accumulated substances in high concentrations may limit the accumulation of a given compound.

A survey of the recent literature of biological accumulation was performed in order to determine whether any plant or animal species are satisfactory accumulators of chemical compounds and ions for quantitative analysis. This literature search included the available literature on the following types of bioaccumulation:

1. biogeochemical techniques
2. microbial accumulation
3. radioisotope accumulation
4. accumulation of trace metals by animals and plants

5. accumulation of pesticides by animals and plants
6. plants as indicators of chemical equilibria and air and water pollution.

The bioaccumulator literature falls generally into two categories: laboratory experimental studies and field studies. Laboratory studies allow a given organism to grow in a medium while maintaining precise control over ambient concentrations of the accumulant being studied. At predetermined time periods after the onset of the experiment the amounts accumulated by the organism are analytically measured, allowing measurement of accumulation factors on a time horizon. Graphical relationships and regression functions can be calculated for accumulation factor vs. time and accumulation factor vs. ambient concentration.

Field studies are most useful in identifying the suitability of particular species as indicators of ambient concentrations of metals and ions. Field studies yield quantitative data on the distribution of plant species and on the accumulated levels of metals, compounds, and ions in plants and animal organs, but do not allow a presentation of quantitative data on lengths of exposure time or on ambient concentration levels.

In order to assess the utility of bioaccumulators in the accumulation and monitoring of substances present in

the ppb and ppt range, chemical and biological accumulating systems which accumulate the same molecules or ions in the same media were compared. Concentration factors and accuracy of concentration were compared. The only bio-accumulators of potential importance for quantitative studies are listed in section 5.3.

5.1 Characteristics of Bioaccumulator Systems

Organisms accumulate chemical substances in three ways. Some substances are taken up actively by cellular biochemical transport processes; this is the familiar process of active transport. In this process, termed active transport, organisms expend energy in order to maintain certain substances at high concentrations in the cell. Generally, substances concentrated in active transport are trace metals and compounds which are important to the cell metabolism. These include S, Fe, and P.

Other substances are accumulated by absorption onto tissue or through cell walls. A characteristic of accumulation by this mechanism is that accumulation factors and accumulation time will be similar for both live and dead tissue. Absorption and the speed of concentration

will be greater in dead tissue for those organisms whose biochemical mechanisms actively exclude the compounds or ions being absorbed. Pesticides and other organics are accumulated by this mechanism.

A third method of concentration is that of food chain concentration. Chemical substances which are consumed during metabolism will not be concentrated along a food chain; however, compounds and ions which are not employed in metabolism will be concentrated by the higher members of a food chain, which eat producers and first level predators with low concentrations of non-metabolized substances in their systems.

Since biochemical mechanisms are generally molecule-specific and enzyme-specific, it is not surprising that each biological accumulator is selective as to the substances which are accumulated. While chemical accumulator systems will generally accumulate a class of compounds with similar chemical behavior (i.e., one accumulator system may accumulate As, Sb, and Bi; another may accumulate Ga, In, and Pt; and another may accumulate Ti, (II) and Va (III)), in general biological accumulators will be more selective. A particular organism may accumulate Pb but not Sn or may accumulate P and not Sb.

Biological accumulators are not selective, however, for substances passively absorbed. In accumulation of chlorinated organics, for example, one organism may accumulate a wide range of chlorinated organics such as DDT, DDD, DDE, aldrin and dieldrin.

Bioaccumulator species generally exhibit variability in the ability of individuals of the species to accumulate a substance. This generally makes organisms unsatisfactory for quantitative analytical work, especially when compared to chemical or physical accumulation systems. It can be confidently predicted that 73% of a particular organic compound will be recovered by using a carbon filter. For a biological accumulator such confidence is not generally justified. Accumulation factors vary with the following:

1. temperature variations
2. pH variations
3. age of organism
4. daily oscillations (endogenous rhythms)
5. seasonal and growth period oscillations
6. latitudinal day length variations
7. variations due to location of sample leaf on plants

8. variations due to the ambient concentration level
9. variations due to the part of the body samples
(such as digestive system, gills, bones)
10. variations due to different soil moistures and
different precipitation levels.

The bioaccumulation changes encountered for different environmental conditions make the use of biological accumulators for analytical work impossible. For example, Cearley (1973)¹ found that the southern naiad (*Najas quadulepensis*) accumulated cadmium with an accumulation factor varying from 6,000 to 40,000. The factor of accumulation varied with the ambient concentration of the cadmium ions. There is no simple functional relationship relating concentration of cadmium in the southern naiad to ambient concentrations. Wilkes and Weiss² (1971) found that dragonfly nymphs (*Tetragonuria*) accumulate DDT with an accumulation factor varying from 250 to 2,700. The accumulation factor changes with the length of exposure time. Thus, the accumulator system can be used for analytical assessment of ambient concentration only if the time of exposure is accurately known.

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1. Cearley, J.E. and R.L. Coleman, "Cadmium Toxicity and Accumulation in Southern Naiad," Bull. of Environmental Contamination and Toxicology, 9: 100-101 (1973).
 2. Wilkes, F.G. and C.M. Weiss, "The Accumulation of DDT by the Dragonfly Nymph, *Tetragoneuria*," Trans. Amer. Fish. Soc., (No. 2), (1971).

Organisms such as bacteria, plankton, yeasts, and other microorganisms are generally more useful for analytical accumulation than are higher organisms such as fungi, invertebrates, vascular plants, and higher animals. More advanced organisms contain many organized biochemical systems and biological subsystems. Microbial organisms display a much simpler biological organization and are regulated by a limited number of chemical systems. It is therefore possible to characterize analytically the relationship between accumulation factors and ambient concentrations of chemicals, and to specify constraints within which the microbial systems must be grown to achieve analytical accumulation.

More highly organized biological systems are characterized by complex biochemical systems and organs that permit them to adapt to adverse conditions; they can become specialized for survival in hostile niches and in adverse environments to which microbial systems are not specifically adapted. Therefore, species distribution of organisms such as vascular plants, insects, and animals can indicate differences in environ-

mental constraints and can serve as indicators of the chemical concentrations found in different environmental media.

For example, trees, bushes, and understory plants serve as excellent indicators of mineral concentrations in the ground water. The presence of plants which are particularly adapted to assimilating high concentrations of particular ions, or which are adapted to resisting the effects of particular ions, is useful in locating ore deposits. Certain species grow while others are absent in the vicinity of aluminum ore deposits. Chemical extraction and analysis of ions present in plant tissues is used to further verify the presence of an ore deposit.

Tissue analyses of certain plants can be used to characterize ground water chemistry without direct sampling of the ground water. Some of the plant species are particularly useful because of their deep root systems. These plants accumulate dissolved substances from subsurface soil and ground water which is filtered through rock formations, allowing the convenient monitoring of ground water at depths which can only be directly measured by the use of expensive and time-consuming drilling technologies.

5.2 Bioaccumulator Methods

The accumulation of particles from air takes place through respiration or by absorption onto surface tissue. Only a few organisms have been successfully used as indicators of particulate levels in the air. There has been no successful quantitative study of the relationship of particle concentrations in tissue as indicators of the concentrations in the air. Human blood levels of cadmium and lead have been employed as indicators of dangerous levels of these compounds for industrial workers who are exposed to those metals in the factory air. Plants such as spanish moss and rice have been used as indicators of the aerial dispersion of compounds. The growth distribution of lichens has been used as an indicator of air particulate levels.

A wide spectrum of plants, which have been used as accumulation systems for measuring substances in the soil, is recorded in the literature. Many plants accumulate pesticides and provide a monitor on the pesticide concentrations residing in the food chain. Others accumulate metals such as nickel, copper, aluminum, and tungsten. These accumulators, as summarized in the accompanying tables, are plants which serve as indicators of metal concentrations in the soil.³

3. See Tables 5-1 to 5-3. Cannon, H.L., Science, 132: 591 (1960).

TABLE 5-1
PROSPECTING BY PLANT ANALYSIS

Locality	Metal sought	Element used	Plant sampled	Results*	Reference
Australia	U	U	<i>Xanthostereum paradoxus</i>	Good correlation	(57)
Canada	Cu, Zn	Cu, Zn	Birch	Good correlation	(58)
British Columbia	Cu	Cu/Zn	Sagebrush, juniper	Good correlation	(37)
British Columbia	Zn	Zn	Silver birch	Good correlation	(34)
British Columbia	Zn	Zn	Alder	Good correlation	(59)
British Columbia	Mn	Mn	Hemlock	Good correlation	(34)
British Columbia	Au	Au	Horsetails, trees	Good correlation	(60)
British Columbia	Ag	Ag	Horsetails, trees	No correlation	(60)
British Columbia	Ni	Ni	Fir, cedar	Good correlation	(61)
British Columbia	Mo	Mo	Balsam	Good correlation	(34)
British Columbia	Cu	Cu/Zn	Pine, fir	Good correlation	(38)
Eastern Canada	Cu, Zn	Cu/Zn	Alder, maple, birch, willow	Used in prospecting	(62)
Quebec	Cu	Cu	Balsam twigs	Anomalies discovered	(35)
Cornwall, Wales	W	W	Heather	Anomalies discovered	(4)
Cornwall, Wales	Sn	Sn	Heather	Anomalies discovered	(4)
Cuba	Ni	Ni	Vegetation	Good correlation	(63)
Estérel (Pyrenées)	U	U	Vegetation	Good correlation	(64)
Far East	As	Fe	Grass	Veins defined	(3)
Finland	Ni	Ni	Birch	Good correlation	(6)
Finland	Cu	Cu	Vegetation	Correlations at low concentrations, not at high	(36)
Germany	Ni	Ni	Birch, spruce, pine	Good correlation	(65)
Greece	Cr	Cr	Vegetation	Good correlation	(8)
Japan	U	U	Cypress, pine	Good correlation	(66)
Nigeria	Pb-Zn	Pb	Savannah trees	Good correlation	(42)
Norway	Cu	Cu	Birch, willow	Too erratic to be useful	(7)
Sweden	V	V	Birch, pine	V-shale discovered	(4)
Sweden	Pb, Zn	Pb, Zn, Cu	Birch, pine	Good correlation	(8)
Sweden	Pb-Ag	Pb-Ag	Tree cover	No correlation	(8)
Sweden	Mo	Mo	Tree cover	No correlation	(8)
Sweden	W	W	Tree cover	No correlation	(5)
United States					
Arizona	U	Alpha count	Oak	Good correlation	(67)
Arizona	Cu	Cu	Oak, mesquite	Good correlation	(68)
Arizona	Cu	Cu	Cresote bush, oak	Good correlation	(69)
Calif., Nevada	Ba	Ba	Fir, manzanita	Good correlation	(70)
Idaho	Zn, Pb, Cu	Zn, Pb, Cu	Fir, pine, spruce	Good correlation Zn, Pb, poor correlation Cu	(50)
Missouri	Zn	Zn	Oak	Good correlation	(71)
New Mexico	U	U	Juniper, pine	Anomalies discovered	(72)
New York	Zn	Zn	Willow	Defined Zn area	(26)
New York	Pb, Zn	Zn	Birch, maple, hemlock	Good correlation	(73)
Pennsylvania	Pb-Zn	Pb, Cu/Zn	Birch	Good correlation	(39)
Pennsylvania	Zn	Zn	Vegetation	Good correlation	(74)
Tennessee	Mn	Ni	Oak	Good correlation	(75)
Utah	U	U	Juniper	Anomalies discovered	(31)
Utah	U	U	Juniper, pinyon	Anomalies discovered	(76)
U.S.S.R.	Cu, Mo	Mo	Legumes	Two major Cu discoveries	(77)
U.S.S.R.	Cu, Fe	Fe	Birch, fir	Outlined Cu ore	(78)
U.S.S.R.	B	B	Phreatophytes	Good correlation	(79)
U.S.S.R.	Ni	Ni/Cu	Grasses, herbs	Good correlation	(12)
U.S.S.R.	Co	Co	Grasses, herbs	Good correlation	(80)
U.S.S.R.	Cu	Ni/Cu	Grasses, larch	New Cu discoveries	(12)
U.S.S.R.	Cr	Cr	Grass	Good correlation	(12)
U.S.S.R.	Pb	Pb	Vegetation	Good correlation	(12)
U.S.S.R.	Mo	Mo	Vegetation	Good correlation	(12)

* "Correlation" signifies correlation between plant content and soil content over known mineralization.

Source: H.L. Cannon, "Botanical Prospecting for Ore Deposits," Science 132 (1960), p. 595. (Used with permission)

Table 5-2
PLANTS THAT HAVE BEEN USED AS INDICATORS
IN PROSPECTING

Universal (U) or local (L)	Family	Genus and species	Common name	Locality	Reference
<i>Bitumen</i>					
L	Goosefoot	<i>Anabasis salsa</i>		Caspian Sea	(20)
L	Goosefoot	<i>Salsola spp.</i>	Saltwort	Caspian Sea	(20)
L	Lily	<i>Allium sp.</i>	Onion	California	(44)
<i>Boron</i>					
L	Goosefoot	<i>Salsola nitraria</i>	Saltwort	U.S.S.R.	(24)
L	Goosefoot	<i>Eurotia ceratoides</i>	Winter fat	U.S.S.R.	(24)
L	Plumbago	<i>Limonium suffruticosum</i>	Statice	U.S.S.R.	(24)
<i>Copper</i>					
U	Pink	<i>Gypsophila patini</i>	Karum	U.S.S.R.	(13)
L	Pink	<i>Polycarpea spirostylis</i>	Pink	Australia	(45)
U	Mint	<i>Acrocephalus roberti</i>		Katanga	(16)
L	Mint	<i>Elsholtzia haichowensis</i>	Elsholtzia	China	(15)
U	Mint	<i>Ocimum homblei</i>	Basil	Rhodesia	(17)
U	Moss	<i>Merceya latifolia</i>	Copper moss	Sweden and Montana	(14)
L	Poppy	<i>Eschscholtzia mexicana</i>	Calif. poppy	Arizona	(46)
L	Plumbago	<i>Armeria maritima</i>	Thrift	Scotland	(47)
<i>Gypsum</i>					
L	Buckwheat	<i>Eriogonum inflatum</i>	Desert trumpet	Western U.S.	(18)
L	Loasa	<i>Mentzelia spp.</i>	Blazing star	Western U.S.	(18)
<i>Iron</i>					
L	Birch	<i>Betula sp.</i>	Birch	Germany	(48)
L	Guttiferae	<i>Clusia rosea</i>	Copey clusia	Venezuela	(49)
<i>Lead</i>					
L	Grass	<i>Erianthus giganteus</i>	Beardgrass	Tennessee	(50)
<i>Phosphorus</i>					
L	Morning-glory	<i>Convolvulus althacoides</i>	Bindweed	Spain	(48)
<i>Selenium</i>					
U	Legume	<i>Astragalus bisulcatus</i>	Poison vetch	Western U.S.	(51)
U	Legume	<i>Astragalus racemosus</i>	Poison vetch	Western U.S.	(51)
U	Legume	<i>Astragalus pectinatus</i>	Poison vetch	Western U.S.	(51)
U	Sunflower	<i>Oenopsis spp.</i>	Goldenweed	Western U.S.	(51)
U	Sunflower	<i>Aster venustus</i>	Woody aster	Western U.S.	(51)
U	Mustard	<i>Stanleya spp.</i>	Princessplume	Western U.S.	(51)
<i>Selenium and Uranium</i>					
U	Legume	<i>Astragalus pattersonii</i>	Poison vetch	Western U.S.	(18)
L	Legume	<i>Astragalus preussi</i>	Poison vetch	Western U.S.	(18)
L	Legume	<i>Astragalus sp.</i>	Garbancillo	Andes	(19)
<i>Silver</i>					
L	Buckwheat	<i>Eriogonum ovalifolium</i>	Eriogonum	Montana	(47)
<i>Zinc</i>					
U	Violet	<i>Viola calaminaria (hitea)</i>	Zinc violet	Belgium and Germany	(52)
L	Saxifrage	<i>Philadelphus sp.</i>	Mock orange	Washington	(53)

Source: See Table 5-3.

Table 5-3
PHYSIOLOGICAL AND MORPHOLOGICAL CHANGES IN PLANTS
DUE TO METAL TOXICITIES

Element	Effect	Reference
Aluminum	Stubby roots, leaf scorch, mottling	(54)
Boron	Dark foliage; marginal scorch of older leaves at high concentrations; stunted, deformed, shortened internodes; creeping forms; heavy pubescence; increased gall production	(54) (24)
Chromium	Yellow leaves with green veins	(31)
Cobalt	White dead patches on leaves	(30)
Copper	Dead patches on lower leaves from tips; purple stems, chlorotic leaves with green veins, stunted roots, creeping sterile forms in some species	(55) (16)
Iron	Stunted tops, thickened roots; cell division disturbed in algae, resulting cells greatly enlarged	(55) (56)
Manganese	Chlorotic leaves, stem and petiole lesions, curling and dead areas on leaf margins, distortion of laminae	(54)
Molybdenum	Stunting, yellow-orange coloration	(55)
Nickel	White dead patches on leaves, apetalous sterile forms	(30)
Uranium	Abnormal number of chromosomes in nuclei; unusually shaped fruits; sterile apetalous forms, stalked leaf rosette	(28) (32) (31)
Zinc	Chlorotic leaves with green veins, white dwarfed forms; dead areas on leaf tips; roots stunted	(31) (55)

Source: H.L. Cannon, "Botanical Prospecting for Ore Deposits," Science 132 (1960), p. 593. (Used with permission)

Vascular plants, fish and invertebrates accumulate many substances from fresh water. Transition metals, radioactive isotopes such as Cr^{51} and Se^{75} , pesticides, are all mentioned in the literature as substances which are accumulated by plants and fish. Plants provide accurate monitoring when employed as monitors of long-term ambient concentrations because of their slow growth rate. Fish and invertebrates generally have widely varying concentration factors depending upon which parts of the body are measured.

Fungi, bacteria, yeast, algae, and invertebrates such as daphnia, are accumulators which have been studied under laboratory conditions. A functional relationship between organism concentration and ambient concentration can often be determined for these species. Accumulation usually requires 8 to 90 hours, and in some cases organisms can accumulate ions in the ppb range.

Mollusks, polychaete worms, and algae are all mentioned in the literature as accumulators of substances from salt water. Transition metals such as Cd, Cu, Mn, Fe, Hg, Pb, radioactive transition metals such as Co^{58} , Co^{60} , Cr^{37} , K^{40} , Fe^{59} , Mn^{54} , Mg^{55} , and pesticides such as DDT, DDE, DDD, Dieldrin, Parathion and toxaphene are all accumulated.

The concentration factors achieved by mollusks vary widely with the season, the biological process involved (for example, the unique accumulation rate during the spawning season) and with the section of the body analyzed. For example, the gonads of Milittus edulus accumulate Co⁵⁸ with a concentration factor of ten, while the stomach accumulates Co⁵⁸ with a concentration factor of 1,000, and the bones and gills in concentrations of 100 over the ambient level. Accumulation by mussels requires a time period of approximately one to two months, and it is not therefore a good laboratory system for water sampling.

Mammals act as food chain accumulators from tissues of fish, plants, insects, animal invertebrates, and other vertebrates. Few experimental determinations of accumulant concentration factors for food chains are recorded in the literature. Thus, analysis of the metal, radioactive metal, and pesticide content of mammal tissues can at present only be used as rough indicators of the occurrence of toxic substances within a food chain.

5.3 Presently Most Suitable Bioaccumulators

Three recommendations can be made for further study of bioaccumulators:

- 1) Yeast, fungi, and several microbial systems including bacteria are the accumulation systems

which give the most accurate quantitative indication of environmental contamination levels. In particular, yeast accumulation from water follows a direct functional relationship between ambient and tissue concentration.

- 2) Aquatic organisms accumulate selenium from fresh water organisms such as algae, daphnia, and other microinvertebrates. Selenium is present in forms such as seleno-methionine and selenite. The importance of these bio-accumulators is that selenium is difficult to accumulate using physical-chemical systems.
- 3) Most other bioaccumulator systems are useful as indicators of pollution levels rather than for the analysis of environmental levels of toxic substances. This includes the use of mollusks as indicators of metals, mammals as indicators of pesticides and radioactive isotopes, and plants as indicators of metal deposits.

TABLE 5-4

BIOACCUMULATION OF TOXIC SUBSTANCES FROM AIR

BIOACCUMULATION OF TOXIC SUBSTANCES FROM AIR

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cd	Human blood	Air	Indicator, no quantitative relationship		Unknown	Unknown	Cernik (1974)
Cd	Human urine	Air	Indicator, no quantitative relationship		Unknown	Unknown	Cernik (1974)
Pb	Tillandsia usneoides (Spanish Moss)	Air	Indicator		Indicator	Unknown	Martinez, Nathany, and Dharmarajan (1971)
Pb	Human blood	Air	Indicator	Unknown	Indicator	Unknown	McNeil and Plasnik (1974)
O-ethyl S,S-Diphenyl-Phosphorodithiolate (Hinosan)	Rice Plants	Air	(.001-.003 ?)	40 ppm (?)	Laboratory results differed from field results	4 weeks	Takase, Tan, Ishizuko (1973)

TABLE 5-5

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cl	Citrus seedling tissue	Soil	Cl ⁻ uptake depends on root surface area and is independent of Cl ⁻ concentration		Cl ⁻ uptake, independent of high Cl ⁻ concentration	(?)	Attman (1972)
Hg	Milfoil, wormwood, other plants	Soil	30-100		Unknown	Unknown	Bol'shakov, K'yakora, Plushko, and Sheherbakov (1969)
N1	Eucalyptus lesoveffi	Soil					Cole (1973)
N1	Eucalyptus salvbris	Soil					Cole (1973)
N1	Malaleuca shethiana	Soil					Cole (1973)
N1	Hybanthus floribundis	Soil					Cole (1973)
p ³²	(Spruce needles) Picera excelsa	Soil	85		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
p ³²	(Spruce wood) <i>Picea excelsa</i>	Soil	135		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
p ³²	<i>Acer campestre</i> (Hedge maple)	Soil	Wood 35 Shoots 170		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
p ³²	<i>Populus nigra</i> (Black poplar)	Soil			Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
p ³²	<i>Thuja</i> (Arbor Vitae)	Soil			Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
Pb	<i>Spartina alterniflora</i>	Soil	Unclear		Unclear	Months (?)	Banus (1974)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Sr ⁹⁰	(Spruce needles) <i>Picea excelsa</i>	Soil	85		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
Sr ⁹⁰	(Spruce wood) <i>Picea excelsa</i>	Soil	135		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
Sr ⁹⁰	<i>Acer campestre</i> (Hedge maple)	Soil	Wood 85 Shoots 170		Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
Sr ⁹⁰	<i>Populus nigra</i> (Black Poplar)	Soil			Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)
Sr ⁹⁰	<i>Thuja</i> (Arbor Vitae)	Soil			Accumulation varies with season, chemical composition of soil	4 weeks dependent on season	Gagnaire (1962)

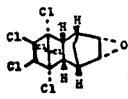
BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
W	Nathofagus menziesii (Silver Beech)	Soil	22.2-79.5	.9 - 270 ppm	Good functional relationship between accumulation and concentration in soil. Linear relationship for beech seedling leaves	8 weeks	Quin, Brooks, and Reay (1972)
DDT	Aphanonyces euteides	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
DDT	Fusarium solani	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	24 hours	Ko and Lockwood (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
DDT	<i>Pythium utimum</i>	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
DDT	<i>Rhizoctona solani</i>	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
DDT	<i>Streptomyces albus</i>	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
DDT	<i>Streptomyces aureofaciens</i>	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content & pH	24 hours	Ko and Lockwood (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
DDT	Streptomyces griseus	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content and pH	24 hours	Ko and Lockwood (1968)
DDT	Streptomyces viridochromogenes	Soil	1.8	294 ppb	Concentration factor varies with plant density in soil and with soil moisture content and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Cotton plants	Soil	(80?)(leaves)		Unknown	21 days	Kavadia (1972)
	Aphanomyces euteiches	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture, and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Fusarium solani	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture, and pH	24 hours	Ko and Lockwood (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	Pythium ultimum	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Rhizoctonia solani	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Streptomyces albus	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Streptomyces aureofaciens	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
Dieldrin	Streptomyces griseus	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content & pH	24 hours	Ko and Lockwood (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	Streptomyces viridochromogenes	Soil	2.4	250 ppb	Varies with density of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
N-1-papthyl-phtalamic acid (Naphtalan)	Phaseoleus vulgaris (Bean plants)	Soil					Devlin and Yaklich (1972)
PCNB (pentachloronitrobenzene)	Aphanomyces euteiches	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachloronitrobenzene)	Fusarium solani	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachloronitrobenzene)	Pythium ultimum	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SOIL

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
PCNB (pentachlorobenzene)	Rhizoctonia solani	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachlorobenzene)	Streptomyces albus	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachlorobenzene)	Streptomyces aureofaciens	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachlorobenzene)	Streptomyces griseus	Soil	7	250 ppb	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)
PCNB (pentachlorobenzene)	Streptomyces viridochromogenes	Soil	7	250	Varies with concentration of plant growth, soil moisture content, and pH	24 hours	Ko and Lockwood (1968)

TABLE 5-6

BIOACCUMULATORS OF TOXIC SUBSTANCES FROM FRESH WATER

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cd	Najas quadulepensis Spreng (Southern Naiad)	Fresh water	6000-40000		Accumulation factor may be second order function of ambient concentration	11 - 21 days	Cearley (1973)
Cr ⁵¹	Anguilla anguilla	Fresh water				20 days	Descamps (1973)
Cu	Ictalurus nebulosus (Brown bullhead)	Fresh water	not > 2		not > 60%	30 days	Brungs, Leonard, McKim (1973)
Cu	Lymnaea stagnatis	Fresh water				4 hours	Spronk, Tilders, Van Hook (1973)
Pb	Lolium perenne (Rye grass)	Fresh water (may also accumulate from soil)	203-240 roots 124-266 shoots	375-1500 ppb	se= .3 ppm shoots .04 ppm roots under laboratory conditions. Concentration factor varies with medium concentration	21 days	Jones, Clement, Hopper (1973)


BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Se ⁷⁵ (from selenomethionine. <small>CH₃SeCH₂CH₂CH₂COOH</small>)	Scenelesmus dimorphus (algae)	Fresh water		.8 ppb		24 hours	Sandholm, Oksan Pesonen (1973)
Se (from selenite)	Daphnia pulex	Fresh water		.8 ppb		24 hours	Sandholm, Oksan Pesonen(1973)
Se (from selenite)	Daphia magna	Fresh water		.8 ppb		24 hours	Sandholm, Oksan Pesonen(1973)
Se (from selenite)	Cyclops serrulatis	Fresh water		.8 ppb		24 hours	Sandholm, Oksan Pesonen(1973)
Zn	Lepomis macrochirus (Bluegill sunfish)	Fresh water	<.25 gill <.1 bone		±50%	96 hours	Cairns et al. (1971)
Zn	Phaeodactylum tricornutum	Fresh water					Davies (1972)
Zn	Neocosmospora vasinfecca (fungi)	Fresh water culture	5.6 (pH 6.5)	1.6 ppm	Concentration factors are accurate if pH, temperature, and metabolic conditions are held constant	60 minutes	Paton and Budd (1972)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Zn ⁶⁵	Dunaliella tertiolecta (algae)	Fresh water	Unknown	600 ppb	Accumulation varies with temperature and light	4 hours	Parry and Hayward (1973)
DDT	Tetragonuria (Dragonfly nymphs)	Fresh water	250-2700 (varies functionally)	3.5-20 ppb	Concentration factor varies with plant density in soil and with soil moisture content, and pH	.5-6 days	Wilkes and Weiss (1971)
DDT	Daphnia Magna	Fresh water	16000-23000		Accumulation factor varies with background concentration	26 hours	Crosby and Tucker (1971)
DDT	Gambusia affinis	Fresh water			Accumulation varies with temperature		Murphy and Murphy (1971)
DDT	Fish	Fresh water	.88 1870	.11 ppm .002 ppm	Unknown	Unknown	Vrochinskii (1971)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dichlobenil	Potamogeton	Fresh water	1.3	1 ppm	Unknown, time of measurement critical	2 days	Walsh, Miller, Heitmuller (1971)
Dichlobenil	Poecilia labipinna	Fresh water	4.2	1 ppm	Unknown, time of measurement critical	2 days	Walsh, Miller, Heitmuller (1971)
Dichlobenil	Gambusia affinis	Fresh water	6.6	1 ppm	Unknown, time of measurement critical	2 days	Walsh, Miller, Heitmuller (1971)
Dichlobenil	Orthemis	Fresh water	1.6	1 ppm	Unknown, time of measurement critical	2 days	Walsh, Miller, Heitmuller (1971)
Dichlobenil	Plankton	Fresh water	2.9	1 ppm	Unknown, time of measurement critical	2 days	Walsh, Miller, Heitmuller (1971)
α-hexachlorocyclohexane 	Chlorella pyrenoidosa (algae)	Fresh water	200	(153-267) variance	(153-267) variance	15 minutes	Canton and Greve (1974)
α-hexachlorocyclohexane	Daphnia Magna	Fresh water	60 (350)	(11.05 ppm) (.8 ppm)	±20% ±20%	3 hours (1/8 hours)	Canton and Greve (1974)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
α -hexachlorocyclohexane	Leloistes reticulatus ("guppy")	Fresh water	140 250	(.01, .05 ppm) (.8 ppm)	±20%	3 hours 48 hours	Carton and Greve (1974)
HgCH ₃	Lepomis macrochirus (Bluegill)	Fresh water	<270		No	100 hours	Burrows and Krenkel (1973)
HgCH ₃	Salmo gardner (Rainbow trout)	Fresh water	1471 gill 106.5 liver 35.0 stomach 57.9 intestine 111.2 heart 334.8 kidney 8.7 muscle 297.4 blood	.275 ppb	Unknown	24 hours	Olson, Bergman, and Fromm (1973)
HgCH ₃	Hemibarbus barbus (Japanese Barbel)	Fresh water	~100,000	<1 ppm	Unknown	Unknown	Kakizawa, Kosaka, Sugai, Sasagawa, Sekiguchi, Minagua(1972)
HgCH ₃	Triblodon halconesis (Dace)	Fresh water	~100,000	<1 ppm	Unknown	Unknown	Takizawa, Kosaka, Sugai, Sasagawa, Sekiguchi, Minagua(1972)

BIOACCUMULATION OF SUBSTANCES FROM FRESH WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
HgCl ₂ (Hg ⁺⁺)	Carassius auratus (goldfish)	Fresh water	32-250	Range 1 ppb- 100 ppb	Accumulation factor different for different ambient concentrations:	81 hours	McKone, Young, Bradie, and Lisle (1971)
HgCl ₂ (Hg ⁺⁺)	Salmo gardneri (Rainbow trout)	Fresh water	102.1 gill 9.4 liver 8.5 stomach 44.5 intestine 9.4 heart 16.8 kidney .58 muscle 55.92 blood	.275 ppb	Unknown	24 hours	Olson, Bergman, and Fromm (1973)
HgCl ₂ (Hg ⁺⁺)	Chorella (Algae)	Fresh water	Unknown	Unknown	Varies with concentration of algae and temperature. There is less accumulation at lower temperature and higher algae density	Varies with algae concentration and temperature, 20 minutes and longer	Shieh and Barber (1973)

TABLE 5-7

BIOACCUMULATION OF FOUR (4) TOXIC SUBSTANCES
FROM SALT WATER

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cd	Mytilus galloprovincialis	Salt water	~80	50 ppb	Unknown	4-8 days	Majori, Petronio (1973)
Co ⁵⁸	Mytilus edulis (Mussel)	Salt water	95 gill 1000 stomach 10 gonad 100 bone	(?)	Varies with environmental gradients	21 days	Pentreath (1973)
Cs ³⁷	Mytilus edulis (Mussel)	Salt water	<1		Unknown	24 hours	Lee, Saverheloan, Benson (1972)
Cu	Nereis diversicolor (Polychaete worm)	Salt water	.38-.68	(for ~50 ppb)	Variable accumulating factor	Unknown (weeks?)	Bryan and Hummerstone (1971)
Cu	Mytilus galloprovincialis (Mussel)	Salt water	172	55 ppb	Unknown	8 days	Majori, Petronio (1973)
Co ⁶⁰	Fucus inflatus (Brown algae)	Salt water	100-510		Accumulation factor varies with age, temperature, season. Measures are ±8%	Growth season	Buyanov and Boyko (1972)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Co ⁶⁰	Fucus serratus	Salt water	100-510		Accumulation factor varies with age, temperature, season. Measures are $\pm 8\%$	Growth season	Buyanov and Boyko (1972)
Co ⁶⁰	Fucus vesiculosus	Salt water	340-5700		Accumulation factor varies with age, temperature, season. Measures are $\pm 8\%$	Growth season	Buyanov and Boyko (1972)
Co ⁶⁰	Mytilus edulis	Salt water	12 mantle 40 gills 15 adductor muscle 330 liver 1500 byssus 72 gonad 20 viscera 64 all soft parts		Agrees with other similar experimental-ly calculated concentration factors	40-80 days	Shimizu, Kajihara, Suyama, and Hiyama (1971)
Co ⁶⁰	Tapes japonica	Salt water	36 shell		Unknown	(40-80 days) (?)	Kameda, Shimizu, Hiyama (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Hg	Anguilla anguilla (Eel)	Salt water	8 days at .2ppm: 157 gills 54.5 kidneys 62.5 spleens 46.0 brains 48.0 livers		Unknown	Gills: 5 hrs Rest of organs: up to 8 days	Bouquegneau (1973)
K ⁴⁰	Fucus inflatus (Brown algae)	Salt water	16-41		±8% and accumulation factor varies with age, season, and temperature	Growing season	Buyanov and Boyko (1972)
Mn	Nereis diversicolor (Polychaete worm)	Salt water	.01-.16		Variable accumulation factor	Unknown (weeks?)	Bryan and Hummerstone (1971)
Fe	Nereis diversicolor (Polychaete worm)	Salt water	.01-106		Variable accumulation factor	Unknown (weeks?)	Bryan and Hummerstone (1971)
Fe ⁵⁹	Mytilus edulis (Mussel)	Salt water	950 gill 5000 stomach 100 gonad 100 bone	1.23 ppb	Varies with environmental gradients	21 days	Penttreath (1973)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Mn ⁵⁴	Mytilus edulis (Mussel)	Salt water	200 gill 200 stomach 70 gonad 60 bone	1.66 ppb	Varies with environmental gradients	21 days	Pentreath (1973)
Pb	Nereis diversicolor (Polychaete worm)	Salt water	.01-.13		Variable accumulation factor	Unknown	Bryan and Hummerstone (1971)
Pb	Mytilus galloprovincialis (Mussel)	Salt water	394-1080	5-100 ppb	Accumulation proportionate to concentration in water	Unknown	Majori and Petronio (1973)
Zn	Nereis diversicolor (Polychaete worm)	Salt water	.09-1.78		Variable accumulation factor	Unknown	Bryan and Hummerstone (1971)
Zn ⁶⁵	Mytilus edulis (Mussel)	Salt water	260 gill 460 stomach 100 gonad 30 bone	25.6 ppb	Varies with environmental gradients	21 days	Pentreath (1973)
Zn ⁶⁵	Mytilus edulis (Mussel)	Salt water	400 shell		Agrees with other experimental work done by same experimenters	40-80 days	Kameda, Shimizu, Hiyama (1968)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Zn ⁶⁵	Tapes japonica	Salt water	50 shell		Agrees with other experimental work done by same experimenters	40-80 days	Kameda, Shimizu, Hiyama (1968)
Zn ⁶⁵	Fucus spiralis (Brown seaweed)	Salt water	Unknown		Unknown	Unknown	VanWeers(1973)
DDD (DDT)	Crassostrea virginica (Oysters)	Salt water	280-980		Variable and also varies with time, season, and biological function	24 weeks	Lowe, Wilson, Rick, and Wilson (1971)
DDE (DDT)	Crassostrea virginica (Oysters)	Salt water	21,000-31,000		Varies with time, season, and biological function	24 weeks	Lowe, Wilson, Rick, and Wilson (1971)
DDT	Crassostrea virginica (Oysters)	Salt water	45,000-69,000		Time dependent on season and biological function	24 weeks	Lowe, Wilson, Rick, and Wilson (1971)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM SALT WATER

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	Rangia cuneata (Mollusc)	Salt water	850-2000	.8 ppb	Varies with season	72 hours	Petrocelli, Hanks, and Anderson(1973)
Parathion	Crassostrea virginica (Oysters)	Salt water	240-360		Varies with time, season, and biological function (i.e. spawning)	24 weeks	Lowe, Wilson, Rick, and Wilson (1971)
Toxaphene	Crassostrea virginica (Oysters)	Salt water	23,000-30,000		Varies with time, season, and biological function (i.e. spawning)	24 weeks	Lowe, Wilson, Rick, and Wilson (1971)

TABLE 5-8

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE
(THAT IS, FOOD CHAIN ACCUMULATION)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cd	Halichoerus grypus (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Cd	Phoca vitulina (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Co ⁶⁰	Ondatra zibethicus (Muskrat)	Tissue					Kaye and Dunaway (1962)
Co ⁶⁰	Peromyscus leucopus (White footed mouse)	Tissue					Kaye and Dunaway (1962)
Co ⁶⁰	Peromyscus nuttalli (Golden mouse)	Tissue					Kaye and Dunaway (1962)
Co ⁶⁰	Sigmodon hispidus (Cotton rat)	Tissue					Kaye and Dunaway (1962)
Co ⁶⁰	Microtus pinetorum (Pine mouse)	Tissue					Kaye and Dunaway (1962)
Co ⁶⁰	Sylvilagus floridanus (Cotton tail rabbit)	Tissue					Kaye and Dunaway (1962)
Cs ¹³⁷	Ondatra zibethicus (Muskrat)	Tissue					Kaye and Dunaway (1962)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Cs ¹³⁷	Peromyscus leucopus (White footed mouse)	Tissue					Kaye and Dunaway (1962)
Cs ¹³⁷	Peromyscus nuttalli (Golden mouse)	Tissue					Kaye and Dunaway (1962)
Cs ¹³⁷	Sigmodon hispidus (Cotton rat)	Tissue					Kaye and Dunaway (1962)
Cs ¹³⁷	Microtus pinetorum (Pine mouse)	Tissue					Kaye and Dunaway (1962)
Cs ¹³⁷	Sylvilagus floridanus (Cotton tail rabbit)	Tissue					Kaye and Dunaway (1962)
Cu	Phoca vibulina (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Cu	Halichoerus grypus (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Hg	Chickens	Tissue (fishmeat)	<1 in tissue and eggs		Unknown	<1 day	Campbell, L.D. et al. (1973)
Hg	Phoca vibulina (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Hg	Halichoerus grypus (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Pb	Halichoerus grypus (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Pb	Phoca vibulina (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Ru ¹⁰⁶	Ondatra zibethicus (Muskrat)	Tissue					Kaye and Dunaway (1962)
Ru ¹⁰⁶	Peromyscus leucopus (White footed mouse)	Tissue					Kaye and Dunaway (1962)
Ru ¹⁰⁶	Peromyscus nuttalli (Golden mouse)	Tissue					Kaye and Dunaway (1962)
Ru ¹⁰⁶	Sigmodon hispidus (Cotton rat)	Tissue					Kaye and Dunaway (1962)
Ru ¹⁰⁶	Microtus pinetorum (Pine mouse)	Tissue					Kaye and Dunaway (1962)
Ru ¹⁰⁶	Syvilagus floridanus (Cotton tail rabbit)	Tissue					Kaye and Dunaway (1962)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Se ⁷⁵ from selenite	Puntias arulios	Tissue				24 hour	Sandholm, Oksan, Personen (1973)
Sr ⁹⁰	Ondatra zibethicus (Muskrat)	Tissue					Kaye and Dunaway (1962)
Sr ⁹⁰	Peromyscus leucopus (White footed mouse)	Tissue					Kaye and Dunaway (1962)
Sr ⁹⁰	Peromyscus nuttalli (Golden mouse)	Tissue					Kaye and Dunaway (1962)
Sr ⁹⁰	Sigmodon hispidus (Cotton rat)	Tissue					Kaye and Dunaway (1962)
Sr ⁹⁰	Microtus pinetorum (Pine mouse)	Tissue					Kaye and Dunaway (1962)
Sr ⁹⁰	Syvilagus floridanus (Cotton tail rabbit)	Tissue					Kaye and Dunaway (1962)
Zn	Halichoerus grypus (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)
Zn	Phoca vibulina (Seal)	Tissue (fish)	Unknown (Indicator)		Varies with age	Unknown	Heppleston and French (1973)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIA	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Zn ⁶⁵	Ondatra zibethicus (Muskrat)	Tissue					Kaye and Dunaway (1962)
Zn ⁶⁵	Peromyscus leucopus (White footed mouse)	Tissue					Kaye and Dunaway (1962)
Zn ⁶⁵	Peromyscus nuttalli (Golden mouse)	Tissue					Kaye and Dunaway (1962)
Zn ⁶⁵	Sigmodon hispidus (Cotton rat)	Tissue					Kaye and Dunaway (1962)
Zn ⁶⁵	Microtus pinetorum (Pine mouse)	Tissue					Kaye and Dunaway (1962)
Zn ⁶⁵	Sylvilagus floridanus (Cotton tail rabbit)	Tissue					Kaye and Dunaway (1962)
DDD (DDT)	Mink adipose tissue	Tissues, animal and plant	2.46		±2.18% deviation. No data on efficiency of mechanism	<4 weeks	Aulerich et al. (1972)
DDT	Mink adipose tissue	Tissues, animal and plant	3.95		±2.75% deviation. No data on efficiency of mechanism	<4 weeks	Aulerich et al. (1972)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM TISSUE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	Mink adipose tissue	Tissues, animal and plant	8.4	4 weeks	±74% deviation for cited experiment, no data on efficiency of mechanism	<4 weeks	Aulerich et al. (1972)
α-hexachlorocyclohexane	Lebistes reticulatus	Tissue (daphnia)	3-4		±35%	24 hours	Canton and Greve (1974)

TABLE 5-9

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER
CULTURE (THAT IS, ACCUMULATION FROM AGAR AND NUTRIENT MEDIA)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
DDT	Mucor ramannianus	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	Glomerella cingulata	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	Trichoderma viride	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	Streptomyces lavendulae	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	Streptomyces griseus	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
DDT	<i>Streptomyces venezuelae</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	<i>Bacillus subtilus</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	<i>Serratia marcescens</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	<i>Agrobacterium tumefaciens</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
DDT	<i>Anacystis nidulans</i>	Fresh water	849 ± 232	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester(1969)
DDT	<i>Scenedesmus obliquus</i>	Fresh water	626 ± 134	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester(1969)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
DDT	<i>Euglena gracilis</i>	Fresh water	99 ± 22	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester(1969)
DDT	<i>Paramecium busaria</i>	Fresh water	264 ± 21	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester(1969)
DDT	<i>Paramecium multimicronutleatum</i>	Fresh water	964 ± 16	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester(1969)
Dieldrin	<i>Mueor ramannianus</i>	Fresh water (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Glomerella cingulata</i>	Fresh water (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Trichodema viride</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	<i>Streptomyces lavendulae</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Streptomyces griseus</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Streptomyces venezuelae</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Bacillus subtilus</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)
Dieldrin	<i>Serratia marcescens</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood(1967)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Dieldrin	<i>Agrobacterium tumefaciens</i>	Fresh water culture (may also accumulate in soil)	Unknown	.1-1 ppm	Accumulation factor varies with pH	24 hours	Chacko and Lockwood (1967)
Parathion	<i>Anacystis nidulans</i>	Fresh water	50 ± 3 ppm	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester (1969)
Parathion	<i>Scenedesmus obliquus</i>	Fresh water	72 ± 7 ppm	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester (1969)
Parathion	<i>Euglena gracilis</i>	Fresh water	62 ± 2 ppm	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester (1969)
Parathion	<i>Paramecium busaria</i>	Fresh water	94 ± 2 ppm	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester (1969)
Parathion	<i>Paramecium multimicronutleatum</i>	Fresh water	116 ± 2 ppm	1 ppm	As noted under laboratory conditions	7 days	Gregory, Reed, and Priester (1969)

BIOACCUMULATION OF TOXIC SUBSTANCES FROM FRESH WATER CULTURE

ACCUMULANT	ACCUMULATOR	MEDIUM	ACCUMULATION FACTOR	APPLICABLE CONCENTRATION	ACCURACY & CONFIDENCE	TIME REQ. FOR ACCUM.	REFERENCE
Mn	Bacillus subtilis W23	Fresh water		55 ppm	Complex active transport mechanism	2 minutes-4 hours	Fisher, Buxbaum, Toth, Eisenstadt, Silver (1973)

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16. ABSTRACT This report contains a discussion of the techniques which are currently available for the concentration of trace pollutants prior to their analysis. Methods for the accumulation of metals and organic compounds from air, water, and solids are covered as well as a review of recent literature on bioaccumulation. Each section includes tables in which concentration methods, and the accumulated materials are listed along with the pertinent literature references.
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