PILOT COMPOSTING FOR BIOREMEDIATION OF A WEATHERED CRUDE OIL

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NOMENCLATURE

API	American Petroleum Institute
BTEX	The group of benzene, toluene, ethylbenzene, and xylenes
CC	Container capacity
CFU	Colony forming units
C/N	Carbon to nitrogen ratio
CO_2	Carbon dioxide
COD	Chemical oxygen demand
FC	Field capacity
FW	Final weight
GC	Gas chromatograph
GC-FID	Gas chromatograph using a flame ionization detector
HC	Hydrocarbons
IR	Infrared
K	Permeability
MAC	Maximum aerobic concentration
MC	Moisture content, % wt
MeCl	Methylene chloride
MNC	Maximum normal concentration
NOM	Natural organic material
O_2	Oxygen
O&G	Oil and grease
OW	Original weight
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
psf	Pounds per square foot
psi	Pounds per square inch
PV	Pore volume
SP	Static pile
SS	Stainless steel
TDR	Time domain reflectometer
TEH	Total extractable hydrocarbons
TKN	Total Kjeldahl nitrogen
TOC	Total organic carbon
TPH	Total petroleum hydrocarbons
VOC	Volatile organic hydrocarbons
v/v	Volume / volume
WR	Windrow

CHAPTER I

INTRODUCTION

Nature and Extent of Crude Oil Contamination of Soils

The petroleum exploration and production industry produces and transports crude oil using vast resources. Production of oil onshore is performed at numerous sites, which may be located closely together in one area or may be spread out at great distance. Production well spacing may be dense, one per acre, or it may be distant, one per 320 acres. In many cases, a single well may exist in an area and be run by a lone operator. Well fields are located in nearly all geographic areas of the world: arctic, coastal, desert, temperate, and tropical rainforest. Additionally, they are found in remote locations as well as in populated areas.

Minimal production facilities include a well, a pump, a short transfer line, and a liquids storage tank. Sites with larger production have larger or additional tanks accompanied by oil-water separation equipment. They also have more valving to accompany more extensive piping for liquids handling. Oil from large production sites is usually transferred into an oil pipeline rather than stored for pickup by a tank truck. An oil pipeline may serve to collect oil from multiple wells in a single field to bring it to a single gathering point. From there, it often enters a much larger pipeline operated at high pressure and may travel to an oil terminal or oil refinery. A single large oilfield can have a network of oil transfer lines or feeder and main trunk lines which total over a hundred miles long.

Oil contamination from these operations occurs because of equipment failure, human error, and acts of nature. Equipment may fail because of age, poor design, or operation above rated capacity. Spills due to human factors include sabotage, loss of well control during drilling which results in blowout, failure to close valves after transfer operations or maintenance procedures, and past production operations. In the past, a common and acceptable practice was to use earthen "blow-down" pits to relieve gas pressure buildup in tanks from produced oil. As the gas was vented, it often carried over some residual hydrocarbon into the pit, which then percolated down through the subsurface. Spills and leaks can also result from earthquakes, lightning, fires, or freezing. With the thousands of surface structures for oil production and refining and thousands of miles of oil pipelines, crude oil contamination to soils is common to the petroleum industry.

Spills from production facilities are most often of short duration and limited extent. Generally, a line or valve rupture is readily detected by instrumentation or visual observation and is controlled within 24 hours, usually much less. This type of spill normally occurs at or near the surface or shallow subsurface with a total spill volume typically less than 100 barrels from production equipment. However, slow seeping leaks in equipment below ground are difficult to detect. These often occur over years or decades and can impact the subsurface to considerable depth. The volume leaked over a long time period can be quite extensive and highly weathered compared to spills caused by ruptures.

The Need for a Low-Cost Remediation Solution

Exploration and production (E&P) operations have generated a tremendous amount of hydrocarbon-contaminated soils and pits worldwide. Because of the geographical extent of petroleum facilities, especially those related to on-shore production and transport, a low cost remediation solution to soil contamination is highly desirable. Transporting these contaminated wastes for off-site disposal consumes landfill capacity and results in the operator losing control of the wastes. Landfill storage can be debated as an incomplete rather than a permanent solution to contamination. However, field experiences during the last 8 to 10 years demonstrate that such contaminated soils and solids can be bioremediated on site, which allows the operator to retain control of the waste (Holliday and Deuel, 1994). Technologies that incorporate high tech skid-mounted processes, which require continuous operation and personnel, are the most costly and must be dedicated to remediating one site at a time. Such technologies as thermal desorption, rotary kiln, or thermal incineration require high energy inputs. Slurry phase bioreactors have reduced through-put capacity and are subject to biological upsets. Soil washing requires chemical surfactants, large amounts of water, and disposal of wash water. All of these options require constant attention from personnel to operate the systems. Furthermore, relatively few plants exist for remediation work. The plants can be set up at a central processing facility or they may be located on the remediation site. In remote areas, operational costs may rise sharply if processing equipment is moved frequently or if contaminated soils are transported to a central facility.

Landfarming or landspreading as well as composting of crude oil-contaminated soils have much to offer the industry for remediating these sites. Because both use biological activity to biodegrade or bioremediate the hydrocarbons, the process can be considered natural and low-impact to the environment. Some consider the process to benefit the fertility and productiveness of the soils (de Bertoldi et al., 1983).

Bioremediation of Petroleum Hydrocarbons

Crude oil is composed of many kinds of organic molecules, each one having a different chemical hydrogen-carbon structure. Since crude oil molecules are hydrophobic or "water-repelling", the rate of biodegradation by microorganisms in oil-saturated media is slow. The rate is limited by the ability of the organism to bring a hydrocarbon molecule across the cell membrane, by the lack of electron acceptors to complete the necessary chemical reaction to release energy to the cell, or by the lack of vital nutrients in the environment, namely nitrogen, phosphorus, and potassium. A microbe most often solubilizes a food molecule, or substrate, in order to bring it into the cell (Oberbremer et al., 1990). It does this by various means, including biologically producing surfactants which facilitate this process. Water aids the solubilization process of breaking down macromolecules (hydrolysis) for individual molecular transport into the cell. Solubilization becomes more difficult as the molecules become larger or more complex. Water also comprises about 80% of the cell mass and its presence facilitates the growth of new cells. Typical electron acceptors for biologically mediated reactions include oxygen, nitrate, sulfate, and carbon dioxide. When oxygen is used as the electron acceptor, the net energy released to the cell is highest, and when carbon dioxide is used, the energy released is lowest (McCarty, 1972). In order to build new or additional cells, key nutrients must be available. Nitrogen comprises 8 to 13% of the dry weight of the cell. Phosphate comprises about 5%, and potassium about 0.5% of the dry weight of a cell. Nitrogen, phosphate, and potassium may not be naturally abundant in the amounts necessary for growth of the biomass.

Composting as a Low-Tech Remediation Option

Composting may be defined as the biological decomposition and stabilization of organic substrates under elevated temperatures as a result of biologically produced heat. The final product is stable and can be beneficially applied to land. Composting is common to the agriculture, landscaping, and municipal waste industries. These industries normally compost their waste products, which include animal manures, plant and tree residues, and wastewater sludge, without adding additional nutrients. Such composting reduces the volume of the initial waste stream and produces a commercially marketable product. Crude oil-contaminated soils, however, are usually nutrient poor. A soil's water-holding capacity may also be reduced as it becomes "oil wetted" from the presence of petroleum hydrocarbons. For these reasons, crude oil-contaminated soils are mixed with bulking agents which retain water, provide nutrients and micronutrients, facilitate air transfer, and increase surface area exposed to air and to microbes. Common bulking agents include rice hulls, cotton trash, straw, wood chips, and cattle manure. The compost mixture may also be augmented with additional nutrients that may be lacking in the soil or bulking agent.

Composting, like land farming, attempts to radically improve the assimilation of hydrocarbons by soil microbes by providing water, oxygen, and nutrients at the appropriate levels for maximum biodegradation rates. Because composting is conducted in biopiles rather than by land spreading, the compost mixture acts as an insulator to retain the heat generated by the improved metabolism. As the heat in the process increases, the biological reaction rate in each microbial cell increases, which produces more heat. Cells multiply rapidly. The process perpetuates itself until it is limited by the availability of substrate or any needed component of the cell or by temperature exceeding the upper limit for biological growth.

Industrial composting is conducted using one of three main designs: windrowing, static pile, and in-vessel composting. The windrow (WR) design is the simplest in structure. The pile is laid out in a long row with a pile height ranging from approximately 3 to 8 ft. The pile is periodically turned with mechanical equipment to aerate the pile and to reduce temperature. Mechanical equipment may include the common front end loader or the specialized Brown Bear® auger or Scarab® composter.

The static pile (SP) incorporates forced-air ventilation and once constructed, it is usually not moved until composting is terminated. Slotted air tubes are imbedded in the biopile and air is moved through the compost using a compressor or blower. The SP may be operated on positive or negative pressure or both during treatment. If emissions are of concern, the pile may be covered and the blower operated on negative pressure to pull vapors out of the pile for treatment in a biofilter (U. S. EPA, 1993a). The SP may be laid out in one or more long rows or in a single rectangular pile. The footprint is usually dictated by the land and equipment available. Pile heights of 8 to 12 ft are common.

In-vessel systems are less common and are usually employed where strict control of process conditions and volatile emissions are required. They seek to optimize kinetics of degradation of xenobiotics or recalcitrant contaminants. There are many in-vessel designs and some include the ability to mix the compost during treatment. These systems may be found in fixed locations at industrial plants and municipal waste treatment facilities and are not feasible for treating most crude oil contamination.

Composting offers some advantages over landfarming for bioremediation of crude oil contamination. Higher crude oil degradation rates in compost systems may be achieved because of increased metabolic activity at higher temperatures. Some have reported near complete degradation in less than two months in compost systems (Fyock et al., 1991). Landfarming is generally labor intensive and requires tilling several times a week or even daily during the initial active period. A compost system can be constructed using forced-air static pile design that requires much less labor during the treatment phase. Landfarming offers less control of contaminant leaching than composting conducted on a prepared surface. It usually requires much more surface area than composting, especially if the amount to be processed is in the hundreds of cubic yards of solids. Furthermore, landfarming may be restricted to a one-time application (as it is in the state of Oklahoma), or it may not be allowed at all where subsurface conditions place fresh water aquifers at risk from potentially harmful leachates. Since water is vital to bioremediation, efficient landfarming can be quite challenging in remote locations that are arid or semiarid. Water must be trucked in and usually hand sprayed almost on a daily basis. Composting, on the other hand, tends to conserve the water initially applied and subsequently generated by cellular respiration. Finally, where odor or volatile emissions are of concern, a forced-air static pile system can be designed so that the pile is covered and emissions collected and treated.

Composting Crude Oil Hydrocarbons is a Recent Development

In the 1970s, Raymond et al. (1976) demonstrated the efficacy of bioremediation of petroleum hydrocarbons in soils. By the late 1970s, more researchers began to investigate the application of biological processes to the degradation of hydrocarbons. Bioremediation studies became more varied and experienced moderate growth during most of the 1980s. Most investigations centered around laboratory bench studies which attempted to isolate specific hydrocarbon-degrading organisms and determine their abilities to assimilate the molecules. However, social, political, and legal pressures on environmental issues did not abound until the late 1980s which then sparked a heightened interest in this area on the part of researchers. In the late 1980s and the early 1990s, investigations into the bioremediation of hydrocarbons began to be applied to such areas as contaminated aquifers using modified pump and treat techniques, bioventing of both aquifers and the vadose zone, slurry phase bioreactors, landfarming, and composting.

The Beltsville composting process for municipal sludge treatment was also developed in the 1970s. Since then, composting of these sludges for waste reduction has developed such popularity that it has become a standard practice for that industry. In composting hydrocarbon-contaminated soils and sludges, this technology is being tested with appropriate modifications. An early work composting crude oil sludge in laboratory reactors was conducted by Dibble (1982) for the American Petroleum Institute (API). Very little work of this nature was conducted for the next several years. Investigations into composting crude oils primarily consisted of other laboratory reactor studies. The earliest published study of pilot- or field-scale composting of crude-oil-based hydrocarbons (HCs) found was conducted by Fyock et al. (1991) in the early 1990s. Many of the laboratory studies were conducted using forced-air reactor systems. However, the field study was conducted using a windrow system. Studies since then have been sparse and no literature has been found reporting pilot or field scale composting of HCs using the static pile configuration.

Thesis Topic: Investigation of Composting Efficacy Using a Weathered Crude Oil

Because of the paucity of scientific literature for pilot- or field-scale composting of crude oil-contaminated soils, little is known about the comparative effectiveness of the static pile method versus the windrow method of composting. Of interest to the oil industry are the answers to several questions.

(1) Will the two methods reach the same end point of hydrocarbon degradation for a given system? If so, at what rates?

(2) How does the ease of operating one system compare to the other?

(3) Which system is more cost effective?

(4) Can the system be initiated in winter conditions?

(5) Can either system be operated without constant monitoring?

(6) Can effective biodegradation of a weathered crude oil be accomplished?

This study attempts to provide answers for these questions.

CHAPTER II

BACKGROUND AND LITERATURE REVIEW

Introduction

Most scientific knowledge and technological applications related to biodegradation of man-made or refined chemical- or hydrocarbon-based materials have been developed in water or soil systems. Such developments have been associated with land farming petroleum-based products, remediating waste streams with industrial bioreactors, and performing in-situ bioremediation of contaminants in groundwater (U.S. EPA, 1993b). Although composting plant and animal wastes is a proven and popular practice, little work has been conducted to study the application and efficiency of biodegradation of hydrocarbons using this technology.

The major parameters involved in all microbial activities include those related to energy transfer and utilization during cellular activity. To achieve optimum performance in any biologically mediated system, an understanding of control of the food or energy source, nitrogen, pH, electron acceptors, heat, and moisture is crucial. Some of the important parameters understood in conventional composting will be discussed and related to the biodegradation of hydrocarbons by microorganisms.

Crude Oil: A Complex and Dynamic Substance

Crude oils are complex mixtures of hydrocarbon molecules of different molecular structures. Each crude has a different mixture of these molecules depending upon its origin

and the effects of weathering. Petroleum hydrocarbon mixtures may contain hundreds or even thousands of individual constituents. These constituents may be grouped into such classes as paraffins, olefins, aromatics, heteroaromatics, and polar compounds (Holliday and Deuel, 1993). They range from simple straight chained alkanes to more complex branched chain and polycyclic carbon molecules.

Weathering changes a crude oil mixture as it is exposed to a new environment. Such changes can be caused by evaporation, dispersion in the environment, photochemical oxidation, chemical degradation, dissolution, water-in-oil emulsification, adsorption, and consumption or biotransformation by microorganisms. Each different molecule has individual chemical and physical properties, such as viscosity, vapor pressure, molecular weight, molecular structure, solubility, etc. Therefore, weathering affects each molecule in a different manner.

The extent of these factors can be observed by comparing a light crude oil with a high API gravity to a heavy crude oil with a low API gravity, or by comparing a fresh oil spill with an aged oil spill. Evaporation is usually the most significant weathering process during the early life of an oil spill. The low-to-medium weight crude oil components with low boiling points are highly volatile and easily lost to the atmosphere. Evaporation can account for the loss of one to two-thirds of an oil spill's mass during this period and is affected by contact with soil or water, wind velocity, air and ground temperature, and intensity of solar radiation (Whiteside, 1993).

Biodegradation of Hydrocarbons

The utilization of hydrocarbon molecules by microorganisms as a food and energy source is well documented in the literature. A review of one computer database (Life Sciences Collection) from January 1978 to June 1989 listed more than 700 citations for microbial degradation of hazardous materials, primarily saturated and substituted hydro-

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carbons (HCs) (Miller, 1990). What makes this attractive for the remediation of hydrocarbon cleanup in the petroleum industry is the potential it provides for the decomposition to innocuous compounds. This process is known as mineralization and is shown in the generic form in Equation 1 below:

$$\text{Hydrocarbon} + \text{oxygen} \text{ ------------> } \text{carbon dioxide} + \text{water} \qquad (1)$$

Specific mineralization of a particular hydrocarbon molecule can be shown stoichiometrically as in Equations 2 and 3 below for hexane and hexadecane.

$$C_6H_{14} + 9.5\ O_2 \text{ -----------> } 6\ CO_2 + 7\ H_2O \qquad (2)$$

$$C_{16}H_{34} + 12.5\ O_2 \text{ ------------> } 12\ CH_2O + 4\ CO_2 + 5\ H_2O \qquad (3)$$

In these reactions, energy is released to the microorganism during aerobic respiration using oxygen as the electron acceptor. When oxygen is limited to the system, other electron acceptors can be used during respiration. These commonly include nitrate (NO_3^-), sulfate (SO_4^{-2}), and carbon dioxide (CO_2) and may require different species of microbes to mediate the biochemical reaction. Most importantly, the alternate electron acceptors provide less net energy to the cell during the process (McCarty,1972) and are usually limited in natural ecosystems. The consequence is that biodegradation using these electron acceptors can be quite slow. For maximum enhancement of biodegradation of hydrocarbons, therefore, oxygen is the most desirable electron acceptor.

Upon the initiation of a biological treatment process, there is usually a small population of microbes in a given system. They may be naturally present, or they may be artificially introduced. The intent of the operator is to maximize the biodegradation rate under suitable treatment parameters. To accomplish this, the microbial population or biomass must be increased. This requires sufficient available nitrogen in the system in addition to the carbon source. Equations 4 and 5, each incorporating a different nitrogen compound, describe mineralization for 2 types of hydrocarbon molecules that may be found in crude oils.

$$C_8H_{12} + 1.3NH_4^+ + 4.83O_2 \text{ ----------> } 1.3C_5H_7O_2N + 1.5CO_2 + 7.1H_2O \quad (4)$$

$$C_7H_8 + 5.31\ NO_3^- + 5.31\ H^+ \text{ ---> } 0.41\ C_5H_7O_2N + 2.45\ N_2 + 4.94\ CO_2 + 5.21\ H_2O \quad (5)$$

Equations 1 through 5 are all mediated by microbes, but 4 and 5 show the formation of new biomass in the commonly accepted molecular form of $C_5H_7O_2N$. In Equation 4, the ammonium ion provides the source of nitrogen, and in Equation 5, nitrate provides the source of nitrogen.

Although simplistic, these equations demonstrate the use of hydrocarbons as an energy source for respiration as well as a carbon source to build new cell material. Biodegradation of complex hydrocarbons is usually carried out in several reactions and by any number of microorganisms. A large molecule may be transformed into a simpler one by a particular microbial species. At this point the new intermediate molecule may be attacked by a different species. The capability to attack and degrade food substrates is initiated by complex intracellular enzyme processes and pathways that differ among microbial species. The number of biochemical reactions for biodegradation of the components in crude oil is almost limitless and constantly changing as molecules are consumed or biotransformed into new compounds.

Utilization of Crude Oil by Microbes

Because of the wide variety in size and structure of petroleum hydrocarbon molecules, it is understandable that some are more easily biodegraded than others. The least toxic and most easily biodegraded HCs are n-alkanes, n-akylaromatics, and aromatics in the C10 to C22 range (Watkinson and Morgan, 1990). C5 to C9 HCs are biodegraded by a narrower range of microorganisms, and due to their nature, these are generally removed more by volatilization than by biodegradation.

Branched alkanes and cycloalkanes sized C10 to C22 are less degradable than n-alkanes and aromatics of equivalent size (Watkinson and Morgan, 1990). Branching hinders beta-oxidation, the primary mechanism for degradation of straight chain HCs; cycloalkane degradation requires the presence of two or more species for complete metabolism.

Due to the complexity of crude oil, no single microbial species is capable of metabolizing all of the different types of hydrocarbon molecules (Whiteside, 1993). For this reason, development of particular oil-degrading microbes to use as inocula in bioremediation projects may be ineffective. In fact, a consortium of species may be considered more useful. Most native soils contain naturally occurring hydrocarbon-degrading populations (Bossert and Bartha, 1984). Hydrocarbon degraders isolated from soils include 22 strains of bacteria and 31 strains of fungi. A number of hydrocarbon-degrading actinomycetes have also been isolated, but do not seem to compete as well as other microbes in HC-contaminated soils. The most commonly isolated hydrocarbon-degrading bacteria in decreasing order of occurrence include: *Pseudomonas*, *Arthrobacter*, *Alcaligenes*, *Corynebacterium*, *Flavobacterium*, *Achromobacter*, *Micrococcus*, *Nocardia*, and *Mycobacterium*. The most commonly isolated hydrocarbon degrading fungi in decreasing order of occurrence include: *Trichoderma*, *Penicillium*, *Aspergillus*, and *Mortierella* (Miller, 1990).

These genera comprise between 1 and 10% of soil microbes in uncontaminated soils and are capable of using hydrocarbon as their sole source of carbon and energy. In chronically polluted systems, such as harbors, hydrocarbon-degrading bacteria comprise 10% or more of the bacterial population. Otherwise, they are less than 1% (Whiteside, 1993). The addition of petroleum hydrocarbons to soil selectively enriches hydrocarbon-degrading microbes. Therefore, most studies of hydrocarbon biodegradation in soil have used the indigenous microbial population rather than seeding with enrichment cultures or commercial products (McMillen et al., 1993a).

Several bioremediation studies have used indigenous populations of microorganisms successfully. Typical observations show that initial populations of 10^4 or 10^5 CFU (colony forming units) per gram of soil increase to 10^9 per gram in a relatively short time period when the process is managed.

Heusemann and Moore (1993) observed hydrocarbon degraders increase from 10^5 to 10^9 in 2 weeks in a managed laboratory study conducted to simulate landfarming conditions. Total heterotrophs measured 10^8 and 10^{11} per gram in all treatment mixtures at 2 and 14 weeks respectively. This compared to 10^4 total heterotrophs measured in clean background Michigan sand after 2 weeks. Total petroleum hydrocarbon (TPH) reductions ranged from 70 to 90% for different treatments.

A laboratory study conducted by McMillen et al. (1993a) to simulate windrow composting used site soil as an inoculum which contained 7×10^6 degraders. In 10 days, the specific hydrocarbon degraders in the compost mixture increased from 5×10^4 to 2.7×10^7 per gram. Total extractable hydrocarbon loss was 92% after 4 weeks.

A study by Francis (1989) which used refinery tank bottoms and refinery desalter sludges demonstrated a rapid microbial growth in all compost reactors. Even though the pH in the systems was slightly acidic (pH 6.0 to 6.2), oil-degrading bacteria increased with time and dominated the population. Few fungi were observed in culture tests, yet microbial numbers climbed from less than 10^4 to more than 10^9 in all reactors during the 6 week study.

Since most soils naturally contain a consortium of microbes, even seeding with hydrocarbon-weathered soils may not be necessary. Whiteside (1993) conducted a bench-scale aerobic biodegradation study in aerated bioreactors which resulted in an overall 60-70% TPH reduction over a 5 week period. He found that the rate of TPH loss did not improve in a nutrient-augmented system seeded with naturally occurring bacteria. McMillen et al. (1993b) conducted two laboratory microcosm studies with clean soils contaminated with 3 different API gravity crude oils and augmented with different levels of nitrogen and phosphorus nutrients. Initial numbers of total heterotrophic bacteria in the 3 uncontaminated soils used were 10^6 CFU per gram. Crude-oil-degrading microbes accounted for 1 to 3% of the total population. After 1 week of exposure to crude oil, these numbers increased to 10^8 and 20%, respectively.

Bulking Agents

In order to increase the accessibility of microbes to oils and greases, the surface area of the composting mixture can be increased by using bulking agents. The selection of bulking agents in remote field operations may be dictated by project economics and area resources. They may range from shredded newspapers, tires, and cardboard to agricultural or animal byproducts. Chevron Production Company conducted field composting on a petroleum production pit sludge in Redwash, Utah using wood chips as a bulking agent (Fyock et al., 1991). Others have used stabilized sewage sludge-derived compost as a bulking agent to biodegrade hydrocarbons and PCBs added to belt filter press waste water sludge cake in laboratory compost reactors (Hogan et al., 1989). Since rice hulls are oleophilic and have been used as an oil absorbent, they were used as the bulking agent in the API compost study on hydrocarbon biodegradation (Dibble, 1982). Another study incorporated leaf compost material to mix with tank bottoms and with oily drill cuttings (Martinson et al., 1993). A Canadian experiment in aerated compost reactors used a slightly acidic peat derived bulking agent (Oclansorb) with refinery tank bottoms and desalter sludges (Francis, 1989). Others have used chicken, cow, or horse manure as both the bulking agent and as a primary or secondary nutrient source (Wickham et al., 1991).

Manure has also been used as a supplemental bulking agent with tree waste (McMillen et al., 1993a).

It is important to understand the moisture retention capacity, the tendency for compaction, and the nutrient levels contributed by the bulking agent. The impact of these factors on the bioremediation process can be established using laboratory studies. Accordingly, bench scale treatability studies are considered part of a three step process by the U. S. EPA for bioremediation of field sites in accordance with CERCLA, 1980 (U.S. EPA, 1993b). Such studies performed prior to conducting field operations have been advantageous for optimizing bulking ratios and nutrient additions, and minimizing compaction in compost piles. Several materials including wood chips, wood shavings, peat moss, sand, vermiculite, sawdust, and cocoa shells were tested with coal tar mixtures by Taddeo et al. (1989) to measure air permeability during compression. Although several bulking agents caused a severe reduction in air permeability above 20% compaction, none of them significantly inhibited the extent of degradation. All experienced 90% degradation of total tar in 80 days. A subsequent pilot-scale test was conducted which resulted in 94% reduction of total hydrocarbons. Chevron conducted bench studies to test nutrient addition and compaction of mixtures. Subsequent field composting tested ratios of 4:1, 2:1, and 1.5:1 dry wt bulking agent to hydrocarbon-contaminated solids (Fyock et al., 1991).

The bulking agent used for this purpose also serves to retain moisture, which is a critical component of the process. It may also be an additional food source for microbes. A nutrient-deficient bulking agent, however, may induce a nitrogen limitation on the biological process, particularly if it is a cellulose-based product. Such a system may require supplemental nitrogen in order to facilitate high rates of metabolic activity required for compost to self-heat. Compost field applications are generally driven by obtaining the lowest cost and regionally supplied bulking agent. Moisture and nutrient parameters are therefore more prone to be externally controlled by the operator rather than by choosing the ideal bulking agent.

Moisture

Moisture is important for removing heat in aerated static pile systems. It also serves to facilitate solubilization of nutrients and target compounds in all treatment systems for intake by microorganisms. Water also provides a medium for transportation, so that microbes can spread throughout the compost pile. Recommendations for initial moisture conditions in static pile composting range from 60 to 75%, but conditions in successful systems have ranged from 25 to 76% (Kreigh, 1993). MacGregor et al. (1981) conducted field composting studies on municipal sewage sludge reduction with wood chips as a bulking agent and used an initial moisture content of 70 to 75% wt. Hogan et al. (1989) used an initial moisture content of 60% in their laboratory study on hydrocarbons. Both studies were conducted where moisture was lost through heat removal during static pile aeration. Moisture contents which are too high can promote clumping of material and interfere with aeration, while moisture values which are too low can inhibit solubilization of substrates and nutrients. Dibble (1982) observed the formation of balls of compost caused by periodic mixing from rolling the 55 gallon compost reactor drums used in the API study. Moisture content varied from about 30 to 60% wt and oil and grease varied from about 5 to 65% wt in the reactors.

Moisture content affects both the air permeability and the solubility of hydrocarbons in the compost. McMillen et al. (1993a) observed a threefold reduction in airflow in treatability studies with a compost mixture of shredded tree, manure, and production pit sludge when increasing the moisture content from 39% wt to saturated conditions of 45%. At low moisture content, solubilization of hydrocarbons is reduced and the biodegradation rate decreases. This is complicated by the fact that the solubility of aliphatic compounds rapidly decreases with increasing molecular weight. To solubilize oils, microorganisms frequently produce biological surfactant molecules as emulsifying agents. These surfactants may operate in water systems to form micelle droplets. Or, they may act directly upon hydrocarbons to produce microdroplets for direct ingestion of oil by microbes (Watkinson and Morgan, 1990).

Highly managed compost studies attempt to maintain pile moisture in a narrow range. However, this may not be necessary or even advantageous to the process. Highly managed systems may require additional capital and personnel expense and must be balanced against the questionable increase in bioremediation efficiency. Additionally, composting appears to be a forgiving process. Fyock et al. (1991) observed a decrease in pile temperature upon drying followed by a temperature increase when remoistening the compost. Furthermore, drier pile conditions may promote biodegradation of certain compounds. Fungi are able to tolerate lower moisture conditions than most other microbes. They have also been shown more capable than other microbes in biodegrading some of the more recalcitrant hydrocarbons. For instance, white rot fungi have been specifically employed to destroy PCBs. Therefore, a range of moisture conditions may enhance a more complete mineralization of crude oils as different microorganisms attack individually favored compounds. Finally, an operator may also desire that the pile dries out for easier handling and lower transport costs at the end of the treatment.

Nutrients

There is evidence that both nitrogen and phosphorus must be readily available to microbes for effective biodegradation. In one study, only 3% of a Swedish crude oil was found to be biodegraded in a freshly collected seawater. Individual nitrate or phosphate addition caused little improvement, but when both were added together, 70% of the crude was biodegraded (Atlas and Bartha, 1972). As shown in Table I, nitrogen comprises 8 to

13% of a cell's organic material structure and is a necessary component for building additional cell material. The carbon to nitrogen, C/N, ratio in the cell is about 5:1. During metabolism, roughly half of the hydrocarbon food source is consumed in catabolism or respiration. The other half is used during anabolism or the construction of new cell matter. As a result, the optimum stable C/N ratio in a soil environment for heterotrophic organisms is considered to be 10:1. However, for composting operations, an initial C/N ratio of 10:1 can lead to high levels of ammonia and odor generated by bacteria during the process. Aside from the odor problems associated with ammonia generation, much of the nitrogen in the system will be lost through vaporization. Therefore, the initial C/N ratio should be higher. As composting proceeds, the ratio will drop toward the 10:1 optimum. An initial ratio of 25:1 is suggested by de Bertoldi et al. (1983), but it may be higher for systems where not all carbon is bioavailable. If the C/N ratio is too high, microbes will have to go through several life cycles before they reduce the ratio enough to eliminate the nitrogen limitation and allow for growth of the biomass. Such a high ratio could then retard composting by creating a delay in the self-heating process. Bench studies may again be helpful to optimize the ratio for a specific system.

TABLE I

COMPOSITION OF THE MICROBIAL CELL
(Stanier et al., 1986)

Element	Percentage of Dry Weight
Carbon	50
Oxygen	20
Nitrogen	14
Hydrogen	8
Phosphorous	3
Sulfur	1
Potassium	1
Sodium	1
Calcium	0.5
Magnesium	0.5
Chlorine	0.5
Iron	0.2
All Others	~0.3

Phosphorus comprises 3% of a cell's total dry weight and is a less important nutrient to control than nitrogen. Phosphorus is less likely to leave the system since it is not found in most volatiles. McMillen et al. (1993b) found that a hydrocarbon:ammonia:phosphate ratio of 100:2:1 with trace minerals in a highly managed microcosm study to simulate landfarming reduced the half-life of a naphthenic crude oil from greater than 2 years to approximately 8 weeks. This compared to a ratio of 100:2.0:0.15 which had only marginal impact on biodegradation.

Nutrient augmentation by investigators has included animal manures, refinery waste activated sludge, municipal waste water sludge, and chemical fertilizers. They have been incorporated as a one-time initial treatment by many investigators, while others have carefully controlled nitrogen and phosphorus throughout the entire composting process. Although some add micronutrients to their systems, most investigators consider them available in excess in the soil and therefore supplement with nitrogen and phosphorus only (Bossert and Bartha, 1984). Taddeo et al. (1989) found that nitrogen and phosphorus decreased to the point of requiring supplemental nutrients in the coal tar compost in their study. In Chevron's pilot compost study with production pit sludge, an initial nitrogen concentration of 500 ppm nitrogen (0.089 M nitrogen, 40% moisture) was used. Subsequent additions were made to maintain minimum concentrations of 50 ppm nitrogen and 20 ppm phosphorus (Fyock et al., 1991). The follow-up field scale project used an initial dose of 230 ppm nitrogen (0.066 M nitrogen, 25% moisture) but also employed manure for 15% of the bulking agent volume. Minimum levels of 50 ppm nitrogen and 20 ppm phosphorus were desired, but were difficult to maintain, and required the addition of up to 230 ppm nitrogen and 150 ppm phosphorus twice a week. McMillen et al. (1993a) used an initial nitrogen concentration of 300 ppm urea (0.110 M nitrogen, 39% moisture) and 217 ppm phosphate plus trace elements with subsequent augmentation for composting sludge from a production site. Reduction of total extractable hydrocarbons (TEH) was reported at 92% after 4 weeks. Dibble (1982) used waste activated sludge without additional nutrients in drum reactor tests. Heusemann and Moore (1993) tested various combinations of cow manure, chemical fertilizers, and waste activated sludge as nutrients when composting 5 to 15 year weathered Michigan crude oil-contaminated soil. The combination of all 3 produced the greatest reduction in oil and grease, 58%, and the fastest rate. Chemical fertilizer and cow manure, when used individually, resulted in approximately equal reductions (50%). However, the cow-manure-augmented system experienced a 22 week lag in activity. The waste activated sludge augmented system showed no significant reduction in oil and grease.

Aeration

It is important to supply an adequate volume of air during composting in order to provide oxygen to microbes and to control the heat generated by them. Field composting may be conducted in windrows and manipulated by machinery or in static piles where air is drawn through the piles with blowers under positive or negative pressure. Successful composting of hydrocarbons conducted by Chevron was conducted using the windrow method. Oxygen data were not reported for the study (Fyock et al., 1991). Although a 97% reduction in hydrocarbons was reported, it is possible that the concentration of oxygen in the piles may have been low at times and that the rate of biodegradation may have been reduced. Finstein (1992) found that temperature and oxygen levels 0.6 meters into a windrow pile ranged from 45°C and 19% O_2 soon after manipulation, to 55°C and 2.5% O_2 10 hours later, and 79°C and 10% O_2 35 hours later. The rise in oxygen at 35 hours suggested that the microbes had become debilitated, allowing the oxygen concentration to recover. de Bertoldi et al. (1983) also claim that pile manipulation does not maintain oxygen above their recommended 18%. A satisfactory proper oxygen level may be less than this. Epstein et al. (1976) observed highest compost pile temperatures when oxygen ranged between 10 and 13% using a blower and therefore suggest that oxygen be maintained between 5 and 15% for the Beltsville process.

Regardless of this debate, aeration is probably more important for heat control than it is for supplying oxygen. Static pile forced aeration offers this advantage over that of windrow composting. Eighty to ninety percent of the heat in a biopile can be removed by water vaporization from aeration. Moreover, the biologically generated heat drives 95% of this vaporization (Finstein, 1992). The remainder of the heat loss is caused by dry air convection. Heat losses due to radiation and conduction are negligible for these systems. Since approximately nine times more air is needed for heat removal than for oxygenation in static piles, maintaining high oxygen levels in forced-air systems is an artifact of good heat control.

During composting, heat production increases with microbial activity and is accompanied by increased CO_2 production and O_2 consumption. When conducting compost tests in drum bioreactors which were allowed to self-heat, Dibble (1982) observed a direct inverse relationship between oxygen measured in the exit streams and temperatures of the compost mixtures. Additionally, microbial activity can be evaluated by measuring these gases when temperature is held constant. When conducting compost respirometry tests on weathered Nigerian Brass crude oil at constant temperature, Dibble observed an increase in CO_2 production over a 0 to 10% wt crude oil loading rate and a subsequent decrease in CO_2 from 10 to 25% loading rates.

Oxygen and CO_2 measurements are generally the keystones of laboratory treatability testing for optimizing bioremediation systems including compost mixtures. Respirometry has been used by some to measure either CO_2 production or O_2 consumption or both in bioremediation studies. Marks et al. (1988) conducted respirometry studies on liquid slurries of oilfield production pit sludges and on oily drill cuttings to investigate the efficacy of bioremediating these materials. Investigated were various sources of hydrocarbon substrates, solids loading rates, use of various inocula and measuring oxygen uptake to measure respiration and determine specific growth rates of microorganisms. Kreigh (1993) used respirometry with a production pit sludge to investigate the effects of compaction, moisture content, temperature, nutrient addition, and hydrocarbon concentration on compost mixtures. This study was aimed at developing a Tier 1 protocol for optimizing the selection of parameters for Tier 2 and 3 testing (U. S. EPA, 1993a).

Temperature

As microorganisms metabolize food substances, they generate heat as a result of respiration. The compost pile by nature can act as an insulator and allow heat to build up faster than it is dissipated.

During development of the Beltsville process for composting municipal waste water sludge, temperatures were monitored over a 30 day treatment period at low (40 cm from base), medium (150 cm from base), and high (300 cm from base or 10 cm from top) positions in a static pile (Epstein et al., 1976). Ambient conditions during the study ranged from -2 to 10°C. Boundary and insulating effects were seen by differences throughout the pile. The lowest pile temperatures were measured at 40 cm (16 in.) from the bottom and 30 cm (12 in.) from the outer surfaces. Middle and high positions experienced higher temperatures, up to 78°C (172°F), which were sustained for about 1 week. One mixture showed a decline in the temperature profile at the high position before such a decline occurred at the middle position. The lowest temperature at the bottom center may be explained by the bottom surface acting as a heat sink. The middle position had the best insulation, while the top position had the advantage of rising heat. Temperatures were highest when oxygen ranged from 10 to 13%. Oxygen was lowest (approximately 5%) when an exponential rise in temperature was observed.

As the temperature rises, two categories of microorganisms are activated at different temperature ranges. Mesophilic organisms function at temperatures up to about 45°C before they become self-limiting. Above this temperature, thermophilic microbes begin to metabolize food more efficiently until they become self-limiting at temperatures above 65°C (Finstein, 1992). It is important for the operator to understand the class of organisms needed to meet the objectives of the process. Pile temperatures should be maintained near the optimum, but below the self-limiting temperature. Composting of sewage sludge requires higher operating temperatures to kill pathogens that are a threat to public health. Additionally, composting sometimes seeks high temperatures in order to kill unwanted seeds. However, composting hydrocarbons do not require these high temperatures unless raw sewage sludge is used as a bulking agent.

Biological kinetics, as described by van't Hoff's equation (6), shows that cellular metabolism is roughly doubled for every 10°C rise in temperature (Giese, 1973).

$$Q_{10} = (k_2/k_1)^{10/(t_2-t_1)} \tag{6}$$

where Q_{10}, the temperature coefficient, is the ratio of the rate of activity at a given temperature to the rate at a temperature 10°C lower, k_2 is the rate of the reaction at temperature t_2, and k_1 is the rate at t_1. This equation is more convenient to use in the form of equation (7):

$$\log Q_{10} = 10 / (t_2 - t_1) \log (k_2 / k_1). \tag{7}$$

From this perspective, biodegradation of hydrocarbons should occur more rapidly at thermophilic temperatures. This rule may be generally observed for microbial utilization of hydrocarbons within each category of temperatures up to the upper limit of each range. However, there is indication that highest overall rates of biodegradation occur in the mesophilic temperature range. In the study by McGregor et al. (1981), sewage sludge was composted with wood chips at temperatures of 45, 55, and 65°C using temperature feedback to control forced aeration. The greatest microbial activity occurred at the 45 degree pile temperature. Dibble (1982) reasoned from rates of CO_2 production in weathered crude in composting respirometry studies that the highest biological activity was at 35°C followed by 45°C. CO_2 production at 25 and 55°C were similar and lowest. The laboratory compost reactor study by Hogan et al. (1981) was conducted at both 35 and 50°C using 6000 ppm hydrocarbons mixed with sewage sludge as a bulking agent. After 35 days, the reduction was greater for all compounds at the lower temperature. At 35 and 50° respectively, these reductions were: straight-chain aliphatic 1-octodecene 98.0 and 95.0%, saturated branched-chain 2, 6,10,15,19, 23-hexamethyltetracosane 75.1 and 50.0%, phenanthrene PAH 99.7 and 97.0%, fluoranthene PAH 94.4 and 90.0%, and pyrene PAH 93.0 and 87.1%. Johnson et al. (1994) found that temperature had an inverse effect on the disappearance of BTEX compounds when measuring headspace gases in laboratory studies. At 25°C, a 93% reduction of BTEX was measured, while at 60°C, there was 65% reduction. The field study conducted by Fyock et al. (1991) using the windrow method of composting, resulted in a 98.8% reduction in hydrocarbons at the 4:1 bulking agent to HC ratio, in which the pile temperature was maintained at 43°C.

The API study by Dibble (1982) experienced thermophilic conditions in some aerated drum reactors in 1 to 3 days, depending upon the HC source and the loading rate, when composted with fresh rice hulls. Loading rates up to 15% wt of heavy-fuel tank bottoms did not affect self-heating or oxygen uptake with fresh rice hulls as the bulking agent. A slight temperature increase was typically observed after the drums were periodically rolled to mix the compost. The importance of nutrients was seen with very limited self-heating when the bulking agent consisted of previously composted and recycled material. Only the disposal pit sludge self-heated to a significant and measurable degree during a second incubation cycle when a small amount of fresh rice hulls (6-17%) was added to the drums. A third cycle showed very little temperature increase. Dibble (1982) concluded that petroleum biodegradation alone is insufficient to reach thermophilic temperatures.

Bioremediation as Determined by Hydrocarbon Analysis

Bioremediation is difficult to measure. Because of the broad spectrum of petroleum field environments, hydrocarbon source materials, remediation resources and options, and the metrics used to measure process changes, a standard protocol for conducting tests and measuring endpoints has not been derived by the bioremediation research community. The difficulty in so doing is readily observed in Table II which shows a summary of some of the solid phase bioremediation studies mentioned in this discussion. One can easily notice a wide range of test conditions conducted with different petroleum hydrocarbon derivatives. Tests have been conducted with fresh crude oil, refinery sludges from pits, tank bottoms, and desalter units, oily drill cuttings, and reserve and production pit sludges. They have been conducted in beakers, bottles, flasks, drums, carboys, and PVC pipes at different temperatures and with or without various amendments. Some were actively aerated; some were not. Tests lasted from 5 days to 1 year. It is not likely that a rigid protocol will be established in the future. Bioremediation of hydrocarbons is a new technology and new methods are constantly being attempted.

Biological activity is monitored by measuring temperature, oxygen depletion, carbon dioxide production, and changes in microbial populations. Temperature and gas measurements are rather straightforward. On the other hand, microbial analyses include some variation and use liquid and solid culturing methods such as most probable number (MPN) and plate counts, respectively. These may measure broad groups of microorganisms such as total heterotrophs, or they may measure specific categories such as HC degraders. Although there are differences in these analyses, they are generally used as indicators of biological activity. The method of choice can be used meaningfully as long it is used consistently during a study.

However, a debate exists regarding the analysis of hydrocarbons. The primary reason for this is that hydrocarbon reduction is the purpose and measure for success in

TABLE II

SOLID PHASE REMEDIATION STUDIES

Investigator	Type of Test	Hydrocarbon	Duration	HC Reduction	Analysis Type
Marks et al. (1988)	31 respirometry runs	Diesel drill cuttings; 32 yr old production pit sludge, 16 API grav.	5 days	Up to 70% v/v depending on system parameters.	O&G Std. Method 503 A with $CHCl_3$ extractant
Heusemann and Moore (1993)	Bench study to simulate landfarming	Michigan crude oil	52 weeks	70-90% of TPH, 97% of saturates, 79% of aromatics, up to 88% of heavy HCs w/C > 44; C_0 = 2.45% O & G / 2.11% TPH	O & G 413.1 (grav.) TPH 418.1 (IR)
McMillen et al. (1993a)	Lab compost, passive aeration; shredded tree waste and manure	Production pit sludge	4 weeks	92% of TEH (5.95 to 0.47%), 97% of saturates, 86% of aromatics; 2-, 3-, and 4-ring PAHs extensively biod. TEH (Total extractable hydrocarbons).	TEH (grav.) by Soxlet with 9:1 methylene chloride: methanol; subsequent chrom. column separation and GC/MS using hopane as internal std. and biomarker.
McMillen et al. (1993b)	Lab microcosm	3 crudes: 21.6 API @ 60° 28.7 API @ 60° 31.1 API @ 60°	8 to 52 weeks	15 to 25% of TEH for minimal to moderately managed samples; 80% of TEH for highly managed sample.	TEH (grav.) by Soxlet with 9:1 methylene chloride: methanol; subsequent chrom. column separtaion and GC/MS using hopane as internal std. and biomarker.
Dibble (1982)	Respirometer compost study @ 23 and 56°C; rice hulls; no nutrient augmentation	Weathered Nigerian Brass crude	48 days	Not measured	Solvent extraction (grav.)
Dibble (1982)	Respirometer compost study @ 25, 35, 45, and 55°C; waste act. sl.:oil:wood chips @ 2:1:6 vol.; no nutrient augmentation	Refinery disposal pit sludge (7.5% O&G), separator sludge (2% O&G), tank bottom sludge (13% O&G)	40 days	Not measured	Solvent extraction (grav.)

TABLE II (Continued)

Investigator	Type of Test	Hydrocarbon	Duration	HC Reduction	Analysis Type
Dibble (1982)	55 gal. drum compost reactor; rice hulls bulking agent; no nutrient augmentation; heat into thermophilic range	Weathered Libyan Amna crude	29 days	Approximately 50%	Solvent extraction (grav.)
Dibble (1982)	55 gal. drum compost reactors; rice hulls; no nutrient augmentation; 3 incubation cycles	Disposal pit sludge, separator sludge, tank bottom sludge	21 to 30 days	Disp. pit--76% Sep. sludge--76% T. bottoms--24%	Solvent extraction (grav.)
Whiteside (1993)	Crude oil weathering	S. Arabian crude	58 days	Most significant weathering loss during first 50 hours; 36.5 to 29.1 API grav. reduction during test	Gravimetric
Whiteside (1993)	Aerated bioreactors (not composted), seeded with nat. occ. bacteria	Relatively high API grav. S. Arabian crude	5 weeks	70% of TPH in best system, $C_0 =$ 1689 TPH	TPH-IR (freon extraction)
Fyock et al. (1991)	Field pilot compost windrowing 330 yd^3; wood chips:HC sludge @ 4:1, 2:1, and 1.5:1; nutrient managed	Production pit sludge	38 days	98.8% of TPH in 4:1 w. chips:HC sludge	TPH freon extr. by 418.1; GC type analysis by EPA Method 8100
Francis (1989)	Aerated lab compost reactors; Oclansorb peat; sludges pretreated with permangenate as preoxidant to complex aromatics; nutrient augmented	Refinery tank bottoms, 2 @ desalter sludges	6 weeks	38-55% of TEH	Dean Stark solvent extraction (Soxhlet)
Martinson et al. (1993)	Case study of pilot SP and WR composting; 1:2 leaf:diesel solids	Diesel solids	45 days	12,000 to 100 ppm (WR) 12,000 to 36 ppm (SP)	TPH-GC
Martinson et al. (1993)	Bench compost; 1:1 sludge:straw-manure	Reserve pit sludge	83 days	~60,000 to 1000 ppm	TPH-GC
Hogan et al. (1989)	Aerated composting bioreactors; HCs added to belt filter press w-water sludge cake; this mixture: stabilized sl.-derived compost @ 1:1 w/w. Inoculum added. 35 and 50 C. tests.	Aliphatics: 1-octadecene, and 2, 6,10,15,19, 23-hexamethyltetracosane. PAHs: phenanthrene, fluoranthene, pyrene, chrysene. PCB Aroclor 1232 in a separate reactor at 50°only.	35 days	35°C: 98% of 1-octadecene, 75.1% of hexam., 99.7 phen., 94.4% fluor., 93.0% pyrene; 50°C: 95.1, 50.9, 97.0, 90.0, 87.1. No disapp. in chrysene at either temp. 81.4% of Aroclor at 50°	Soxhlet extraction with methylene chloride,pre and post extracts dried with anhydrous salts; GC/MS

bioremediation of petroleum-contaminated systems. Two general categories of measuring HC reduction are oil and grease extraction (O&G) and total petroleum hydrocarbon (TPH) analyses. Each of these is usually modified by investigators for HC analysis in soils or solids. Method 413.1 developed by the U.S. EPA (1979a) uses solvent to extract and weigh HCs gravimetrically to determine the concentration of residuals on the solids. Modifications include the type of solvent or solvent mixture used in the extraction, extraction apparatuses, and the use of drying agents to negate the effects of water in samples. Pure solvents used include freon, light alkanes, methylene chloride and other chlorinated solvents. Mixtures such as 9:1 methylene chloride:methanol (McMillen et al., 1993a) and 1:1 hexane:toluene (Dibble, 1982) were utilized in other studies. Extraction is conducted in separatory funnels, rotating shakers, or in Soxhlet type systems. Some have used drying agents such as diatomaceous earth or anhydrous magnesium sulfate and anhydrous sodium sulfate (Hogan et al., 1989) to sequester moisture in samples and reduce interferences in the extraction process.

Some consider O&G less reliable than TPH measurement because of extraction of polar compounds that are formed by cellular synthesis (Heusemann, 1993). TPH may be considered an extension of the O&G method whereby polar compounds are removed by chromatographic separation, usually with packed columns. The U. S. EPA Method 418.1 (1979b) serves as the basis for most TPH analyses. However, the EPA methods were developed to measure petroleum HCs in water and require liquid-liquid separation. This is inappropriate for matrices containing solids (Holliday and Deuel, 1993). Therefore, Method 418.1 is also usually modified for solid phase extraction by researchers. Removal of polar compounds is sometimes conducted in alumina or silica gel slurries. Types and quantities of adsorptive materials and solvents have a bearing on the quality of separation as do flow and retention rates. TPH extraction can be quantitated gravimetrically or by analysis using instruments. TPH-IR (infrared) measures one entire peak while TPH-GC measures concentration by individual peak analysis. Positive biases in IR analyses can result from IR dispersion and absorbance of clay particles, and measurement of natural organic matter (NOM) and extraneous plant residues not removed during silica gel cleanup. Negative biases can result from poor extraction efficiency of high molecular weight compounds, loss of hydrocarbons during silica gel cleanup, differences in IR absorbance between standards and samples, and preferential biodegradation of n-alkanes which primarily leave aromatics that are not sensitively measured by IR at 2930 nm. TPH-IR, therefore, is best used only to estimate crude oil concentration in soils (Holliday and Deuel, 1993).

TPH-GC using flame ionization detection (FID) has gained favor over TPH-IR in recent years since it discriminates individual peaks. Additional steps may be taken to separate hydrocarbon saturates, aromatics, and polar compounds (Heusemann and Moore, 1989) or saturates, aromatics, nitrogen/sulfur/oxygen compounds, and asphaltenes from each other (McMillen et al., 1993a). Such separations are also complicated by the previously mentioned factors. Because of the usual high numbers of peaks in crude oils, specific ones must be selected for quantification. An additional complication inherent with individual peak analysis using chromatography is properly establishing a baseline to the peaks. Peak area integration from a baseline common to all peaks can yield very different results than when baselines are established for individual peaks. Quantification is therefore more laborious for TPH-GC analysis. The precision of TPH-GC has been exploited by some researchers to demonstrate bioremediation in their studies. Refractory compounds, ones recalcitrant to bioremediation, may be found in petroleum hydrocarbons. The peaks from such "biomarkers" may be compared to those of other compounds over the lifetime of a study to indicate degradation. Comparisons to C_{30} -hopane have been made to adjust the final reduction in TPH. Ratios of C17/pristane and C18/phytane were also used to demonstrate bioremediation (McMillen et al., 1993a and 1993b).

Although researchers frequently document successful bioremediation from TPH reduction, enhancements such as dispersants, surfactants, and compost can interfere with

TPH analysis. Still, Holliday and Deuel (1993) recommend TPH by GC/FID as the preferred method for monitoring TPH bioremediation. Furthermore, integrating by using the total peak area gives a better indication of bioremediation than by using the total chromatographic area (common baseline).

Although comparisons of n-alkane to pristane or phytane have been made since the 1970s (Bossert and Bartha, 1984), the O&G method has the advantage of simplicity over other methods. It requires no sophisticated separation or analytical methods or instrumentation. For this reason, it is faster, and more replicate samples can be analyzed to improve statistical analysis. Analysis of replicate samples is very important, particularly in field trials where high spatial variability in HC concentrations often occurs. The method has the disadvantage of extracting natural organic material (NOM) along with HCs. This may be corrected by extracting uncontaminated background soil whenever possible. Finally, since extracted HC intermediates contain oxygen, the added weight of oxygen causes some underestimation of actual HC biodegradation (Bossert and Bartha, 1984).

McMillen et al. (1993b) suggests that using any one measurement of HC biodegradation provides an incomplete view of the extent and rate of biodegradation and that a better view of is made by comparing the results of several analytical methods. Such a statement is hardly debatable, but may be impractical for many studies.

Numerous authors have observed oil biodegradation by essentially indirect methods, such as an increase of oil-degrading microorganisms after a pollution incident or a characteristic change in oil composition, such as a decrease of the n-alkane ratio as compared to the branched pristane or phytane. While circumstances sometimes make such indirect approaches unavoidable, there are advantages to direct measurement using a solvent extraction method for gravimetric quantification of HCs. The solvent extract contains not only polluting fossil hydrocarbons, but also lipids, waxes, and hydrocarbons of nonfossil origin. Therefore, uncontaminated soil controls need to be extracted and used for correction whenever residual weight is used as a measure of fossil HC biodegration. HC loss may also occur by abiotic mechanisms, primarily by evaporation. Weight changes due to oxygen addition and evaporation of light HCs need to be included in the assessment of overall HC biodegradation. Bossert and Bartha (1984) recommend using sterile controls if possible. This is usually possible in laboratory studies, but not in large-scale studies.

Composting Effects on Heavy Metals

There is some indication that composting can reduce the leachability of heavy metals. Dibble (1982) observed a decline in arsenic, chromium, manganese, nickel, and zinc, but not in barium and iron after three incubation cycles from sludges mixed with rice hulls. McMillen et al. (1993a) observed low leachable metals in finished compost except for barium. Francis (1989) noted that heavy metals did not accumulate in compost reactor leachate and reasoned that they were retained in the peat matrix. Although these studies did not focus on these effects, they indicate a potential for the humic matter generated by composting to act as a chelating agent to complex heavy metals.

Hydrocarbon Composting Costs

Due to the scarcity of case histories on pilot or field scale composting, good estimates of costs are uncertain. However, the cost per cubic yard of sludge treated in Chevron's pilot demonstration was less than $30 (Martinson et al., 1993). This cost was based on materials, site labor, chemical analyses, and project management and excluded the cost of the prepared facility.

Summary

Although composting is not a new treatment for breaking down plant and animal waste products, its application in biodegrading hydrocarbons is very recent. Furthermore, the majority of the techniques used in the process have been developed for treatment of sewage sludge. Because of their nature, hydrocarbons present different challenges in composting than plant and animal byproducts. Hydrocarbons may present a broad spectrum of molecules that need to be biodegraded. Furthermore, those excavated from various depths in contaminated soils may not provide a homogeneous feedstock for a pile mixture. In some cases, volatile hydrocarbon emissions from the pile may require control.

The bench scale treatability study is an important element of any program designed to implement composting on undefined hydrocarbon sources. By using it as a precursor to field implementation, the critical elements of surface area, HC concentration, aeration, moisture, nutrients, and temperature can be studied as independent variables to help optimize full-scale conditions. Finally, chemical, physical, and biological parameters need to be studied collectively throughout the composting process. Toxicity and bioeffects tests should be run on mature composted material to ensure that recalcitrant and toxic daughter compounds pose no risk to the environment.

Further research needs to be conducted using compost systems for petroleum-hydrocarbon bioremediation. Relatively few such studies have been reported. They consist of a handful of laboratory-scale and even fewer pilot-scale experiments. The diversity in crude and refined petroleum-hydrocarbons provides a large arena for such studies. Of significant interest are the comparative abilities of static pile and windrow systems to facilitate bioremediation of target contaminants. Depending upon the particular petroleum contaminant of interest, the performance of one system may not be superior to the performance of the other system in all cases. Since petroleum-contaminated soils often consist of highly weathered crude oil sludge, a comparison of these two composting methods using such a sludge is of special interest.

CHAPTER III

EXPERIMENTAL MATERIALS AND METHODS

General Overview

A sludge containing approximately 4% O&G was obtained from an excavated production pit in southern Oklahoma and mixed with straw at a ratio of 2:1 straw:sludge v/v and composted for 121 days. Two compost pile methods were used: the forced-air static pile (SP) and the windrow (WR) methods. The common agricultural fertilizers urea and superphosphate were added to the initial compost mixture in a water solution to provide nitrogen, phosphorus, and water to the compost. Static pile conditions were monitored by continuous data collection of temperature measurements at 40 locations in the pile. Oxygen, carbon dioxide, and volatile organic hydrocarbons were usually measured four times a week. Attempts were made to control temperature and oxygen in the SP during the test by operating the blower at various rates. The windrow pile conditions were monitored by manual probing for temperature and gas levels. These measurements on the WR were also usually conducted four times weekly. Once the windrow pile began to self-heat, it was turned regularly with a front-end loader once a week for the first nine weeks of the test. This was later modified. Multiple samples were collected from each pile on a predetermined schedule and O&G, moisture, pH, nitrogen, phosphorus, and microbial counts were measured. A companion study (Lautzenheiser, 1994 - in preparation) was conducted using laboratory reactors using the same compost mixture under aerated conditions at constant temperature.

Compost Facility

The pilot compost tests were conducted at an outdoor facility that consisted of a 50 ft wide by 75 ft long concrete slab which was covered by a metal roof supported by 8 steel beams. The roof provided a working clearance of 10 ft under the steel cross beams for heavy equipment. The slab was crowned in the center. Drains were located around the perimeter of the slab just inside concrete curbing and connected to a leachate collection system. A small office in one corner of the slab was used to house the data acquisition system for the static pile. Ambient weather conditions were monitored by a Climatronics® weather station 200 ft south of the compost facility.

Petroleum-Contaminated Soil Feedstock

A petroleum-contaminated soil feedstock was identified and obtained from an accidentally discovered production pit in southern Oklahoma. Initially, two sites were visited and oil-contaminated soils were gathered and analyzed in order to select a feedstock for the study. Sludge from an abandoned tank battery site consisted of extremely weathered crude oil and appeared very matted and tar-like in nature. This sludge was rejected for the study. The production pit at the second site had been closed for perhaps 15 to 20 years by filling it in with trees and covering it with top soil. Upon excavation, the depth of contamination was found to be 3 to 5 ft beneath the surface. The lateral extent of the contaminated area as judged by visible hydrocarbons in various trenches, was approximately 50 ft wide by 250 ft long. Upon observing the soil profiles of several trenches made at the site, the contaminated interval exhibited a clayey texture composed of several colors. Table III shows a mineralogy analysis of a solvent-extracted sample of the feedstock used in the pilot test. From these data, the soil consisted of about 30% clays with a large fraction composed of highly swelling and exchangeable smectite.

TABLE III

X-RAY DIFFRACTION MINERALOGY ANALYSIS OF SLUDGE FEEDSTOCK

Mineral Constituents	Percentage
Quartz	66
Plagioclase feldspar	Trace
K-feldspar	Trace
Calcite	1
Dolomite	Trace
Hematite	1
Goethite	2
Kaolinite	1
Chlorite	2
Illite/Mica	8
Mixed-layer Illite/Smectite	19
Total	100

The production pit sludge was then extracted with methylene chloride (MeCl) solvent, and the extract was analyzed on a GC to produce a hydrocarbon fingerprint of the oil. Subsequent trips were made to the site to obtain additional feedstock for other tests and to obtain a sizable quantity of material for the pilot test. Treatability tests were performed on a combined sample of 30 gallons. The sludge was collected from the production pit site and mixed on the concrete slab at the pilot test facility using a TroyBilt® 7 HP garden rototiller. The mixed sludge was returned to 5 gallon plastic buckets and sealed with lids. The O&G of the mixed soil measured 10.14% wt. Treatability tests included laboratory compaction tests, nutrient loading tests using lab respirometry, and compost mixing by rototilling. The moisture content of the sludge was measured at 16.89% wt on a Denver moisture balance which dried the sample to constant weight at 105°C. A dry bulk density of 1.427 g/ml was calculated by weighing a 100 ml volume of sludge and subtracting the moisture content of the sample.

A third trip was made after the conclusion of treatability testing to acquire a large quantity of feedstock for the pilot test. In early November 1993, approximately 13 cubic yards of this material was transported to the test site. The sludge was mixed with a BEFCO Series G, side-shift rotary tiller (Model 266-231) with a 66 in. working width and adjustable to a working depth of 8-1/2 in. The tiller was attached to the power takeoff (PTO) of a 50 HP diesel Ford 445 tractor/front end loader with a 1/2 cubic yard shovel. After mixing for approximately 8 hours, 6 samples with 2 replicates each were analyzed and yielded 3.27% dry wt O&G. A concentration between 5 and 10% in the feedstock was desired, so the pile was covered with plastic sheeting until additional material was added. In mid-December 1993, 1 cubic yard of material was excavated from a highly contaminated zone of the production pit and transported to the project site in sealed drums.

In January of 1994, the covered sludge was spread out on the concrete pad with the front end loader and tilled to even the height of the sludge to approximately 6 in. The drums were opened and the contents were distributed about the pile by hand in 2 to 3 gallon increments. The sludge was then mixed with the tractor-tiller for at least 4 hours to homogenize the soil (Figure 1). Fourteen discrete grab samples were obtained for analysis, and the mean O&G was 3.77% dry wt with a standard deviation of 0.056 (4.25% inert solids wt; SD 0.075). This was still below the original O&G level desired for the test, but a decision was made to use the sludge as it was. Its moisture content was measured at 14.56% wt.

Bulking Agent

Straw was selected as the bulking agent for the study since it is almost universally available throughout the United States and much of the world. The straw consisted of stems and some seed heads of harvested wheat. The straw contained 4592 ppm total

Figure 1. Photo of Tractor-Tiller Mixing Final Sludge Feedstock

Kjeldahl nitrogen (TKN). The moisture content of straw was measured at 15.06% wt on the Denver moisture balance. Ten bales of the straw were measured and weighed which produced a dry bulk density of 0.1045 g/ml in bale form. This value would later be used for compaction tests and the companion lab reactor study. It was assumed that upon mixing with sludge and water, and after compaction in the pilot compost pile, the straw under packed conditions would be more similar to that of straw in bale form than in uncompressed and fluffed form.

Treatability Tests

Treatability tests were conducted on the compost materials to determine the ability to mix materials, and the degree or effects of water retention, compaction, and nutrient utilization of the compost. These tests included compost tilling tests, container capacity tests, compaction tests, and respirometry tests.

Compost Tilling Test

Preliminary mixing tests were conducted using the TroyBilt® garden tiller to mix whole straw into a small plot of pretilled earth. This simulated the use of a larger tiller for mixing straw and sludge on a concrete surface. Tilling depth was set at about 5 in.

Container Capacity Test

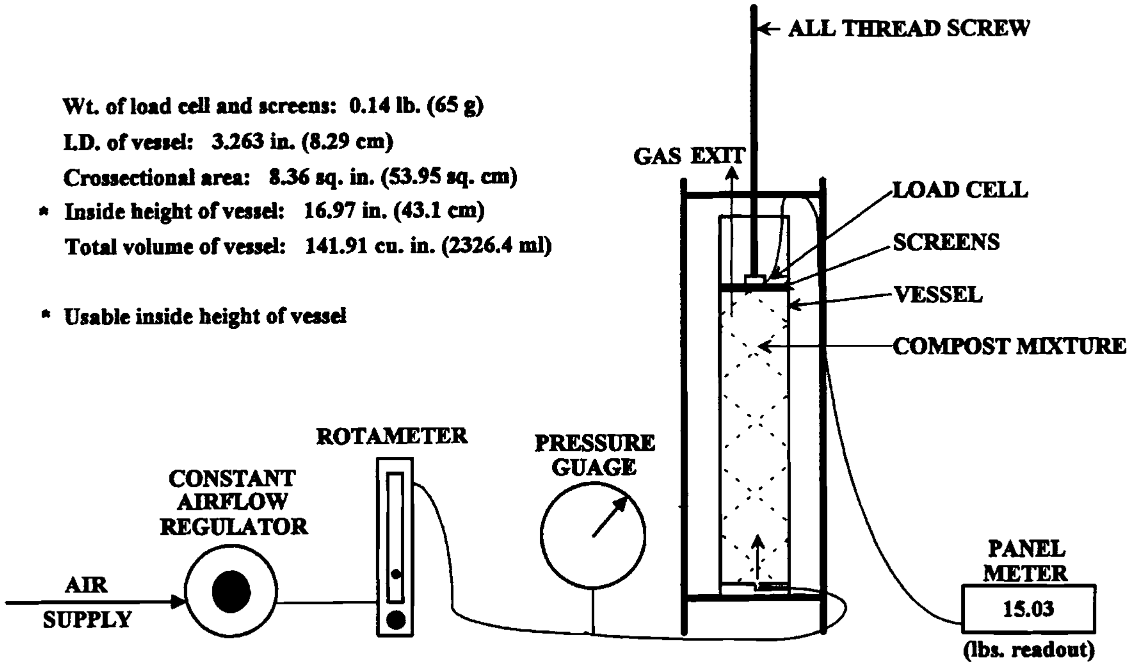
In order to determine the moisture holding capacity in a compost mixture, its container capacity (CC) should be measured. This term is used for moisture holding capacity when measuring soil or solid media in an ex-situ measurement (Kriegh, 1993). This is roughly equivalent to the moisture condition for an in-situ measurement known as field capacity (FC) (Brady, 1990). FC is the percentage of water remaining in soil two or three days after being saturated and after free drainage has practically ceased. It was previously noted that biodegradation rates are higher when the moisture content is less than container capacity.

Container capacity (CC) tests were conducted on the 30 gallon sludge sample with 10.14% wt O&G in order to determine the amount of moisture retained by compost mixtures. CC tests were conducted in perforated 1000 ml plastic beakers at room temperature. These consisted of preliminary tests on mixtures using shredded straw and sludge at 3:1 and 2:1 ratios and a final test using unshredded straw at the 2:1 ratio only. The shredded straw was prepared using a Goossen Bale Chopper®. Both the preliminary and final tests investigated the effect of the order of wetting the materials on the CC. Therefore, each mixing ratio was subdivided into three tests: (1) where the straw was moistened before mixing in the sludge, (2) the sludge was moistened before mixing in the straw, and (3) the straw and sludge were moistened and mixed at the same time. The procedure for conducting these tests is described in Appendix A.

Compaction Tests

Compost compaction testing at 2:1 and 3:1 straw to sludge mixing ratios was conducted using the test stand configuration shown in Figure 2. The tests were run in order to determine the impact of compaction on air flow expected during the pilot test from an eight foot tall compost pile. Compost mixtures were made in the laboratory at room temperature with shredded straw and the 10.14% wt O&G sludge simultaneously mixed together with water at moisture contents (MCs) near CC to simulate worst case conditions (lowest permeabilities). An MC value of 60% wt of the compost components was used to approximate the CC. Later calculations showed the MCs of the 2:1 mixture to be 32.2% wt and the 3:1 mixture 51.7% wt. The MC of the 2:1 mixture was significantly lower than the mixture's CC. However, the 3:1 mixture was at its CC for the test.

The compost mixture was placed in the laboratory test vessel. A 1.25 in. diameter Omega load cell (Model LCG-250) was attached to the center of a set of three stainless steel 50 mesh screens and the assembly was placed on top of the compost. The screens' diameters were slightly less than the diameter of the vessel in order to avoid friction which would artificially raise the load measured by the load cell. An all-thread screw mounted on the top of the vessel holder allowed a load to be placed on the compost mixture to simulate conditions at the bottom of the pile caused by compaction. Air from a Moore Products Nullmatic® constant airflow regulator (Model 40-200) was delivered to an Omega 0-150 standard ml/min rotameter (Model FL-3861G-HRV) which allowed the air flowrate to be carefully controlled and measured. The pressure drop across the compost was measured by a Wallace and Tiernan 0-4.5 psi pressure gauge (Model 62A-4A-0005, Series 1500 High Performance) on the upstream side and was assumed to be 0 on the broad open top on



Wt. of load cell and screens: 0.14 lb. (65 g)
I.D. of vessel: 3.263 in. (8.29 cm)
Crossectional area: 8.36 sq. in. (53.95 sq. cm)
*** Inside height of vessel: 16.97 in. (43.1 cm)**
Total volume of vessel: 141.91 cu. in. (2326.4 ml)

*** Usable inside height of vessel**

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Figure 2. Compaction Test Stand Diagram

the downstream side. The load cell measurement was read on an Omega strain gauge indicator (DP41-S High Performance).

Respirometry Tests

Bench scale treatability studies included tests on 2:1 and 3:1 straw:sludge compost mixtures to measure oxygen uptake as an indicator of biological activity. These tests were conducted using a computer automated respirometer (N-CON System, Inc., pneumatic respirometer; 12 reactor/12 reference chamber, pressure differential detection) which combines oxygen delivery with "real-time" data collection. Tests on compost mixtures were conducted in 500 ml glass reactor bottles for 300 hours. The sludge used was from the original 30 gallon sample with 10.14% wt O&G. The samples were prepared with MC at 80% of CC. Nutrient additions to the mixtures were varied in order to help determine the amount to be used in the pilot test. These included a baseline condition in which no nutrients were added, 0.27 N and 0.54 N nitrogen conditions using urea as the nitrogen source, and a final condition using 0.5 N nitrogen in Evans mineral medium. The Evans mineral medium consists of a macronutrient solution augmented by Drew's trace element solution is added. These solutions are shown in Table IV and Table V, respectively.

TABLE IV

EVANS MINERAL MEDIUM FOR HYDROCARBON-DEGRADING BACTERIA
(Rosenberg and Gutnick, 1981)

Compound	Grams/Liter
$(NH_4)_2SO_4$	1.00
K_2HPO_4	1.00
$MgSO_4{\bullet}7H_2O$	0.30
$CaCl_2{\bullet}2H_2O$	0.132
$FeSO_4{\bullet}7H_2O$	0.02

Add 1 ml of Drew's solution to Evans mineral medium as a growth stimulant.

TABLE V

DREW'S TRACE ELEMENT SOLUTION

Constituent	(FW)	Grams/L	M
$MnSO_4{\bullet}H_2O$	(169)	0.10	591
$CoCl_2{\bullet}6H_2O$	(237.83)	0.02	84
$CuSO_4{\bullet}5H_2O$	(249.6)	0.01	40
$NaMoO_4{\bullet}2H_2O$	(241.94)	0.01	41
$ZnSO_4$	(161.43)	0.011	69
$LiCl_2$	(77.84)	0.009	120
$SnCl_2{\bullet}2H_20$	(225.59)	0.005	22
H_3PO_4	(97.97)	0.016	162
KBr	(119.0)	0.02	168
KI	(166.0)	0.02	120
EDTA		8.00	

The above formulation used in the respirometry test is a tenfold concentration of the original Drew's formulation, as set forth in Reichenback and Dworkin (1981).

Compost Mixing

In mid January 1994, compost mixing operations began. A 2:1 straw:sludge ratio required 28 yds^3 straw for the 14 yds^3 sludge available. From volume measurements averaged on 10 bales of straw, 123 bales were required for the compost. Because of the limitations of the usable area of the concrete pad for mixing the compost, 5 batches of compost were prepared using the tractor-tiller. The 14 yds^3 of sludge was divided into 5 sections, each containing 2.8 yd^3 of sludge. A decision was made to use 25 bales of straw per batch of compost by rounding off the calculation. Based upon the final CC measurement made in the laboratory on the 2:1 mixture with whole straw, the CC value for the mixture was rounded to 90%. The MC of the initial compost mixture was established at 60% of the CC based upon literature values established for bioremediation. After accounting for the moisture content in the raw straw and sludge, 1600 gallons of water were required to raise the MC to this point. This equalled 320 gallons per batch. Based upon previous respirometry work (Kreigh, 1993) and the respirometry testing in this study, the nitrogen level for the mixture was established at 0.25 N. A 4:1 nitrogen:phosphorus ratio was also selected based upon the literature. A total of 30 lbs urea, $(NH_4)_2CO$, and 17.7 lbs superphosphate, P_2O_5, was used in each batch of compost. Laboratory analyses of the compost stock indicated that the starting material had a C:N:P ratio of 18.3 : 1 : 0.24. Appendix B shows the calculations made for nutrient and water additions.

Nutrients for each batch were mixed in a 100 gallon cylindrical composite tank with an Eastern mixer (Model 35E371-1172) mounted on top. The tank was filled with water to which the nutrients were slowly added while dissolving. The urea dissolved readily, but the superphosphate was much less soluble and gave the appearance of being partially in suspension. About 2 hours mixing time was required to dissolve the nutrients in the cold conditions. Hot water was obtained for some of the batches to facilitate their preparation. The pH of these mixtures was measured at less than 2.0 with a Beckman Φ^{TM} 11 pH meter, a dual junction pH electrode and a plug-in thermometer for temperature correction. The pH of each batch was raised from about 2 to about 7 with approximately 1500 g of NaOH crystals. Fresh water was added to raise the volume of solution to 100 gallons. Final pH values of each batch are listed in Table VI.

TABLE VI

FINAL PH'S OF NUTRIENT SOLUTIONS FOR COMPOST BATCHES

Batch	Final pH of Nutrient Solution
1	6.10
2	6.58
3	6.70
4	7.10
5	6.70

After a 1/5 portion of sludge was taken from the feedstock pile, it was spread out in a long, narrow row approximately 10 ft wide by 50 ft long by 5 in. high with the tractor-tiller (Figure 3). During preparation of the first batch, the bales were wet with the nutrient solution before they were added to the sludge and mixed with the tiller. A jet pump, operated by a 3/4 HP electric motor and attached to one of the two mixing tanks or the larger Nalgene tank, was used to apply and measure liquids to the compost. Although provisions were made for adding nutrients to the piles during the test, none were added after the initial application.

Static Pile Forced-Air System

Figure 4 shows a schematic of the side view of the static pile aeration system. An EG&G Rotron regenerative blower (Model DR505AS72M) was mounted on top of a wooden box which served as manifold housing for the air ducts to the static pile. The box was constructed of 3/4 in. plywood with outside dimensions of 42-1/2 in. H x 41 in. W x 27 in. D. A Wood's E-TRAC AC Inverter speed controller (Model WFC-2005OC) was

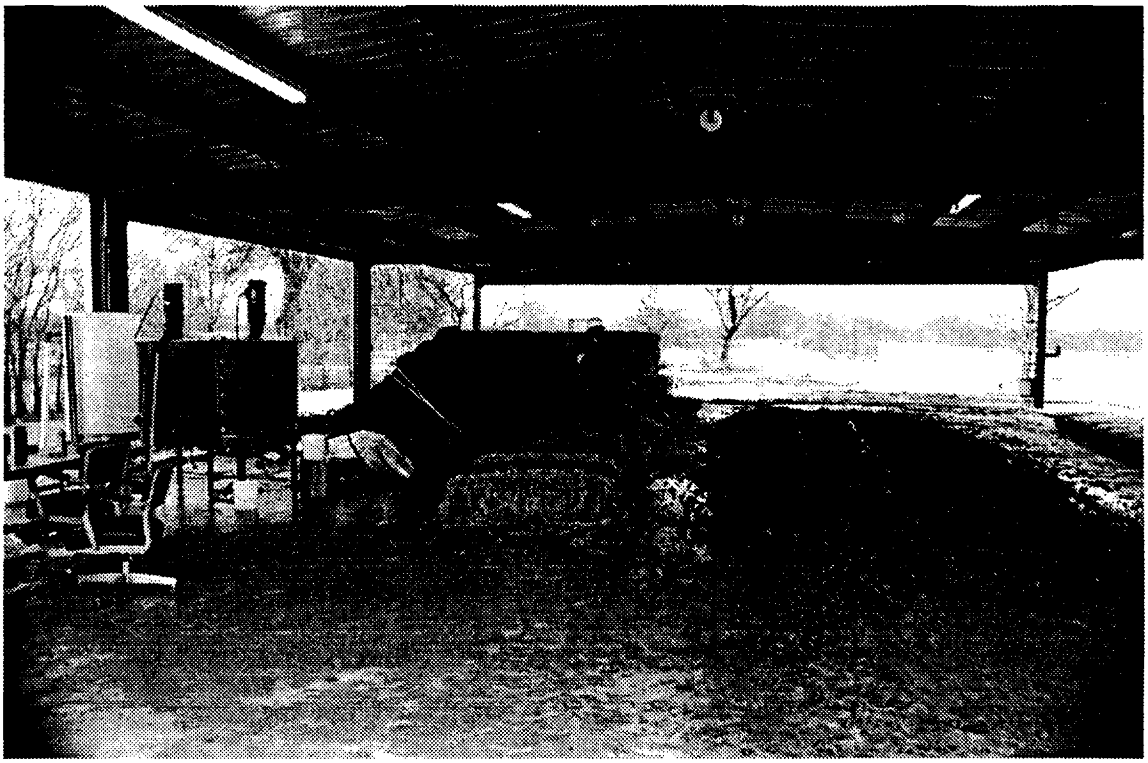

Figure 3. Photo of Sludge Spread Out Ready for Straw Addition

mounted on the side of the plenum box and was wired to the blower to control its speed and direction. Using the controller, the blower could be operated at any speed in either direction to supply air pressure or vacuum to the pile.

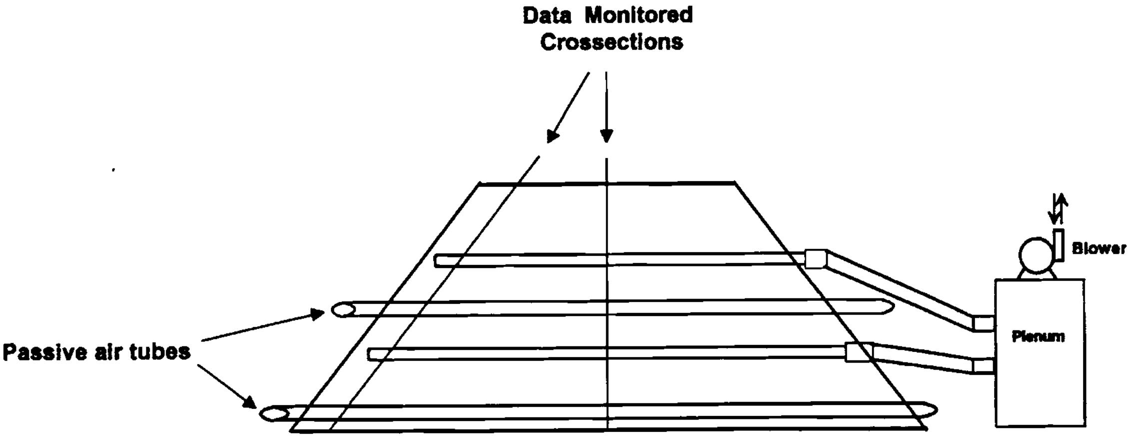

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Figure 4. Diagram of Side View of SP Aeration System

Figure 5 shows the air distribution system with 13 ports available for operation. Each port consisted of 3 in. ID PVC tubing filled with plastic drinking straws held in place with a 20 mesh stainless steel screen. A 1/4 in. hole was drilled into each PVC tube immediately downstream of the screen. This design allowed the insertion of the probe of an air flow velocity meter (TSI VelociCalc Plus Portable Air Velocity Meter, Model 8360) to any depth in the tubing cross section to read the same airflow velocity without being affected by tubing friction losses. Of the 13 ports, 10 were used in the pilot test by attaching 3 in. black ABS, corrugated, flexible plastic tubing. This tubing is commonly used for French drains. Solid, unslotted tubing from the plenum box to the static pile was joined to slotted tubing inside the pile. Since the lengths of the tubing inside the static pile and the resistance due to compaction were different depending upon location, the airflow in the tubes required individual adjustment in order to properly balance overall airflow throughout the pile.

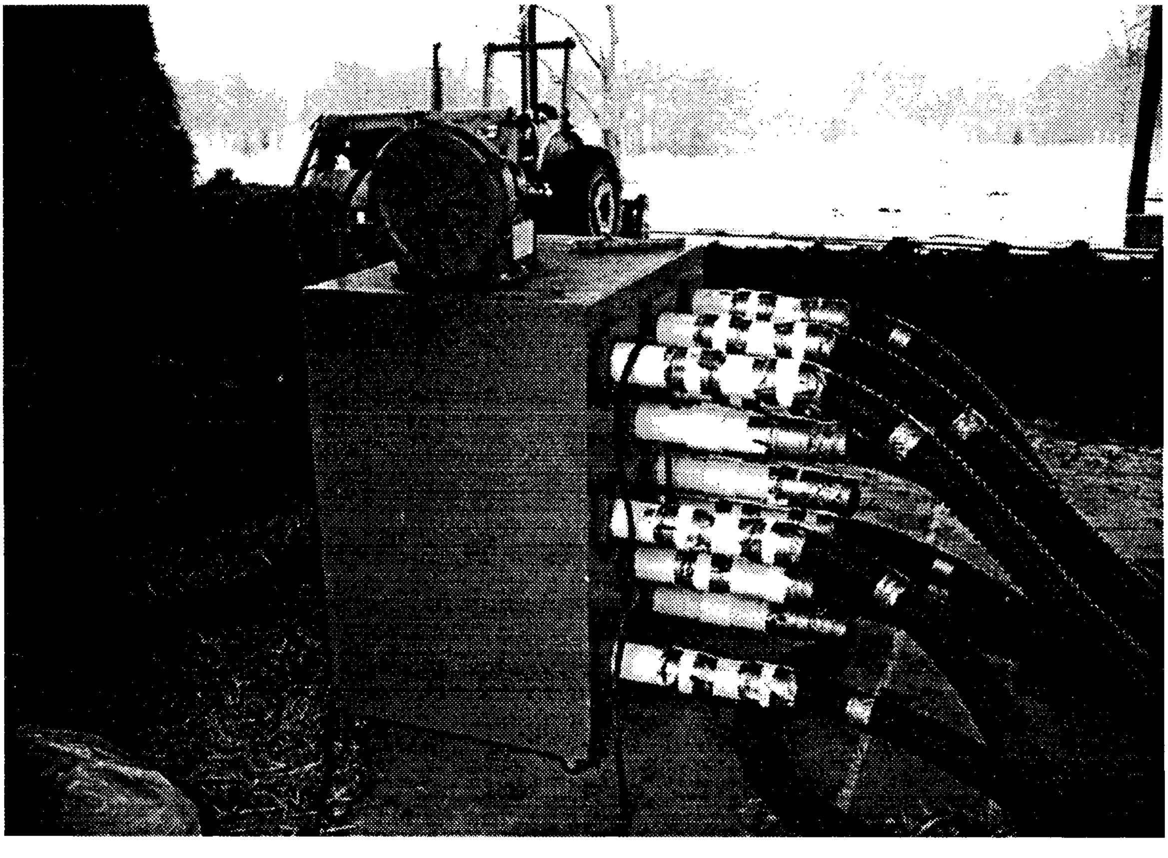


Figure 5. Photo of Plenum Box and Tubing

Butterfly valves were constructed of 1/8 in. aluminum plates to fit the inside diameter of the tubing. A small handle attached to one edge of each valve was used to adjust the degree of opening, and a set screw opposite the handle allowed the valve to both pivot and be locked into place. Balancing the airflow was an iterative process, especially at low flow rates, since adjustment of one tube affected all others.

Figure 6 shows the cross section of the airflow tubing. Tubes at the ground surface and at 4 ft elevation were passive tubes and protruded beyond the edge of the pile about 2 ft. Tubes at the 2 and 6 ft elevations were forced-air tubes which were connected on one end to the solid tubes from the plenum box and were capped off 1 ft from the edge of the pile on the other end.

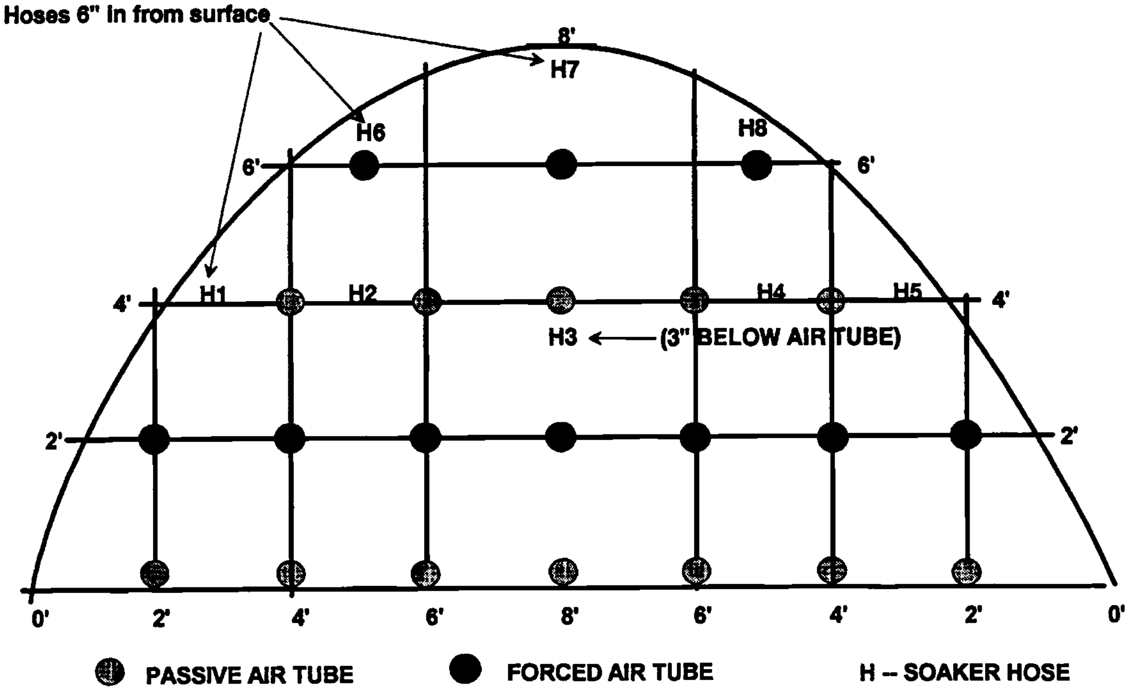


Figure 6. SP Cross Section Showing Aeration Tubing and Soaker Hose Placement

Static Pile Construction

On January 31, work began on constructing the static pile (SP). As the pile was built, the portion of the prepared compost to be added was refluffed with the tractor-tiller.

The intent of this was to break up any clumps that may have formed due to compaction during batch preparation and storage. During construction, a number of instruments and data sampling ports were installed in one of two cross sections. One cross section was in the center of the pile and was perpendicular to the ground plane. The other cross section was located on the end opposite of the blower and lay parallel to the pile surface and 1 ft from it. Figure 4 shows the orientation of the cross sections. Twenty thermocouples (Type T installed in 6 in. long x 1/8 in. dia. SS sheath) attached to Type T thermocouple wire were installed in each of the cross sections and were attached to an automated data collection system (ACUREX AutoCalc® Programmable Data Acquisition System). Thermocouples T0 through T19 were located in the center cross section, and thermocouples T20 through T39 were located in the end cross section. They were positioned in the centers of the grid patterns shown in Figure 7. The 40 thermocouples had been previously bundled and placed in a bucket of icewater for calibration. Ambient temperature at the facility during this procedure was 22°F. Their temperature readings were individually corrected by making

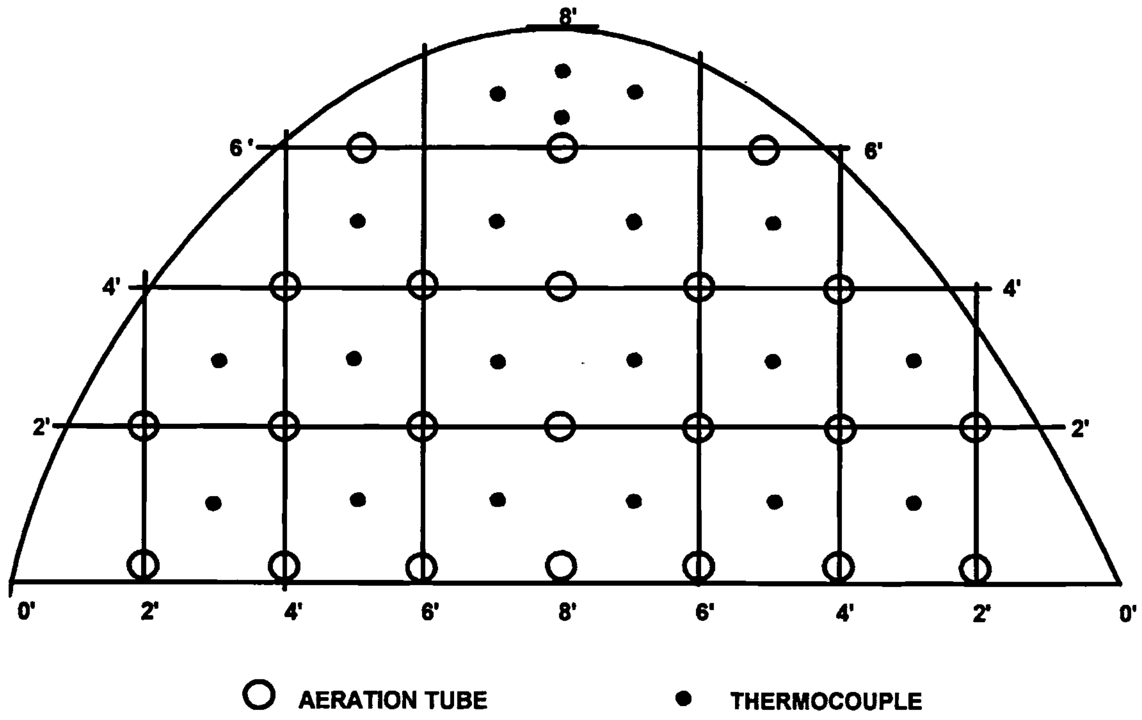


Figure 7. Placement of Thermocouples in SP Cross Sections

appropriate adjustments in the data collection system. The thermocouples were later removed from the icewater and allowed to stabilize at ambient temperature. At the later date, the readings ranged from 40.8 to 42.5°F. A Black Box™ Code Operated Matrix Switch (Model SW541-C) was used to connect the data collection system to an IBM PC compatible computer for programming or to a Black Box™ 2400 baud serial modem for remote data acquisition and program control. Figure 8 also shows the location of 14 waveguides, W1-W14, which were installed to measure the moisture content of the SP. Each buriable waveguide (Model 6005L2), shown in Figure 9, was attached to a 5 meter extension cable. The cable could be connected to a Soil Moisture Trase® System 1 Time Domain Reflectometer (TDR) (Model 6050XI), for moisture measurement of the compost. The waveguides were installed in the center of the SP, but their cross section was offset from that of the thermocouples by 1 ft to avoid electronic interference. Soaker hoses H1-H8 (5/8 in. black porous, connected to reinforced nylon 3/4 in. hose) were installed

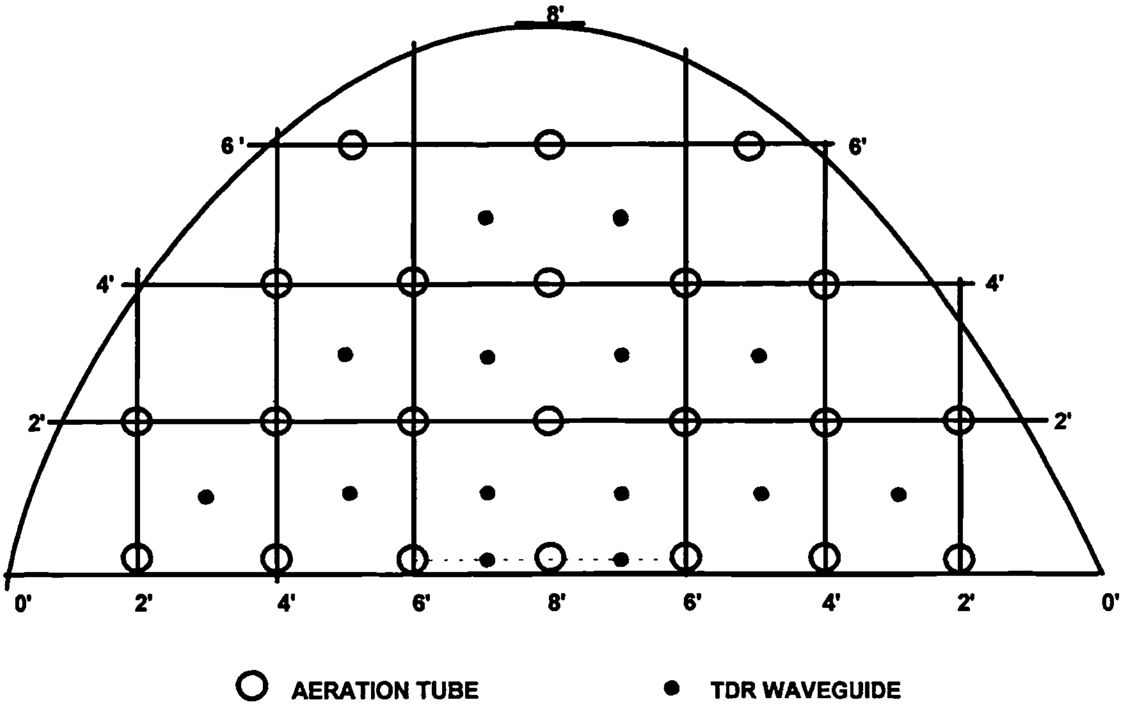


Figure 8. Placement of TDR Waveguides in SP Center Cross Section

inside the pile and along its length to apply moisture or nutrients during the test if necessary. These were connected to a valved manifold system for liquid distribution.

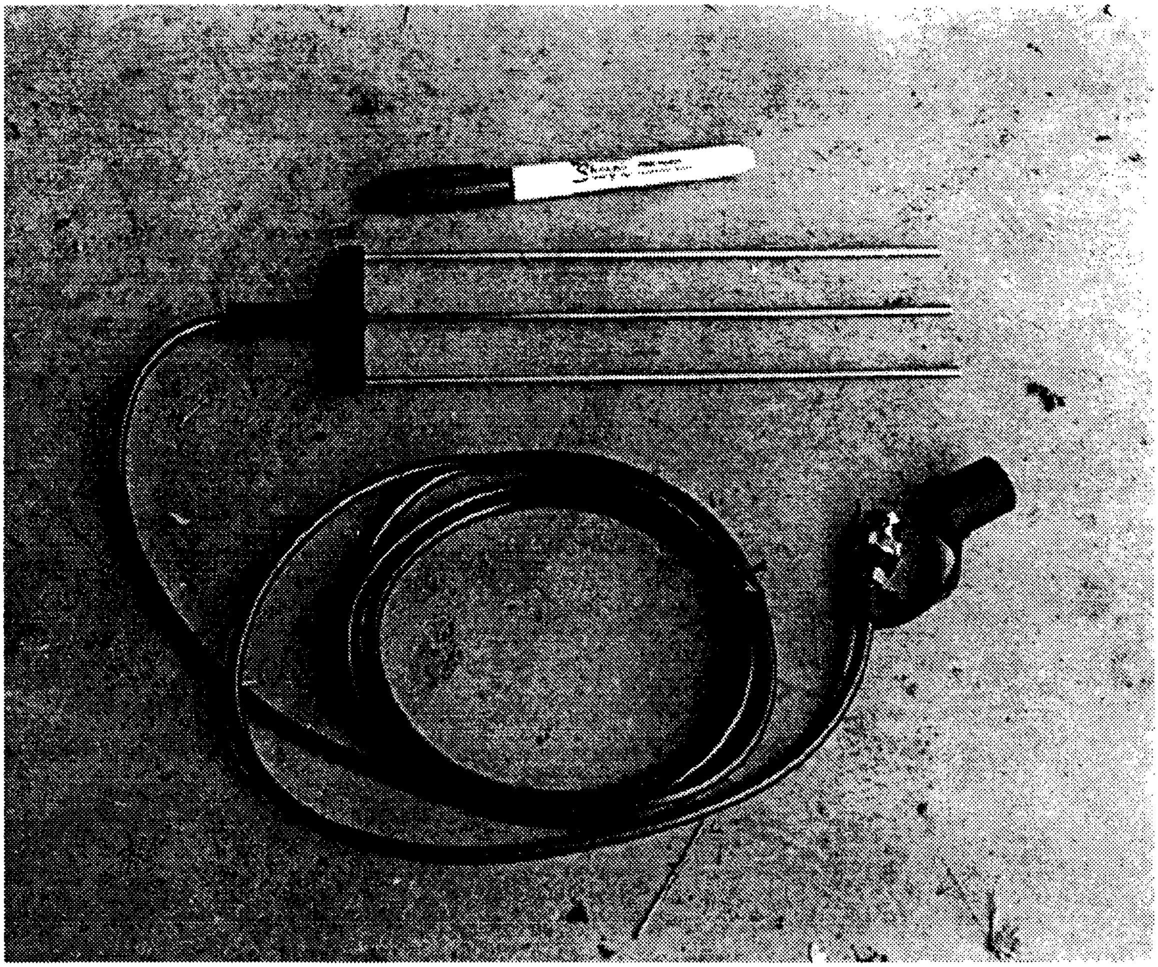


Figure 9. Photo of Waveguide

Seventy-eight porous filters (Porex N125FF) were attached to 1/8 in. teflon tubing and were installed in the two cross sections for sampling gases in the pile. Each filter had been modified with several perforations with a 1/16 in. drill bit to preclude it from plugging off with sludge. When not in use, the tubing was capped with 1/8 in. electrical wire nuts to prevent fouling. Double-faced, aluminum tags were used to mark and identify individual cables, wires, and porous tubing in the SP. The center cross section, shown in Figure 10, provided a high density placement of gas sampling nodes to measure the efficiency of aeration during the test and to identify dead spots. The end cross section, shown in Figure 11,

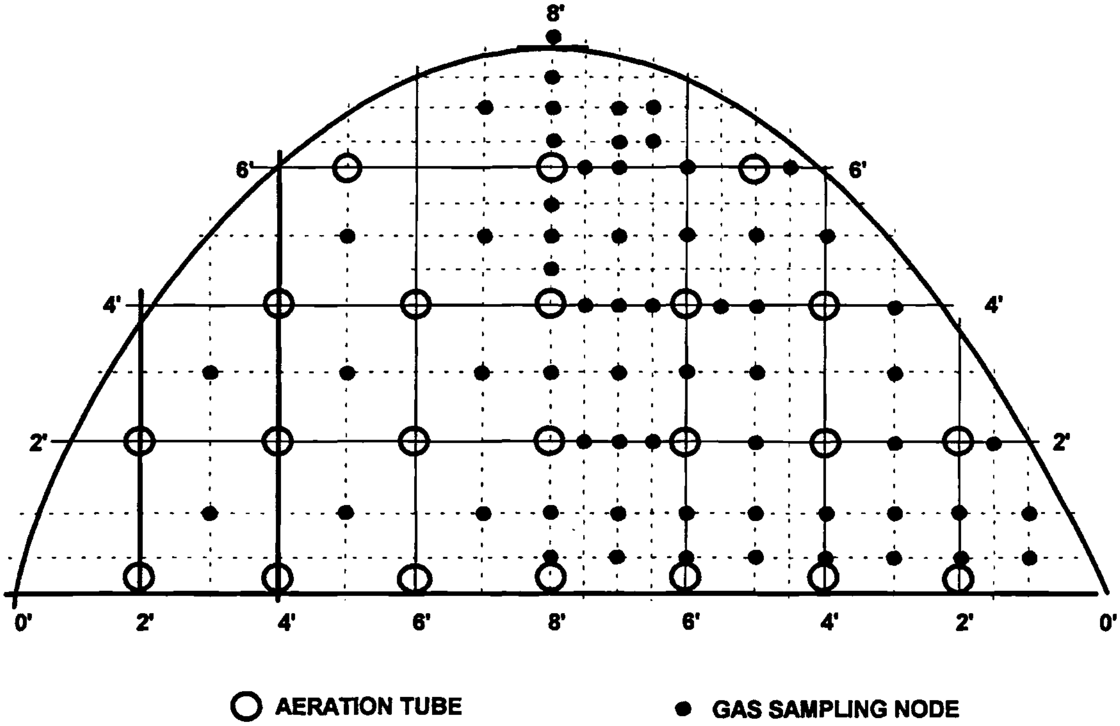


Figure 10. Placement of Gas Sampling Nodes in SP Center Cross Section

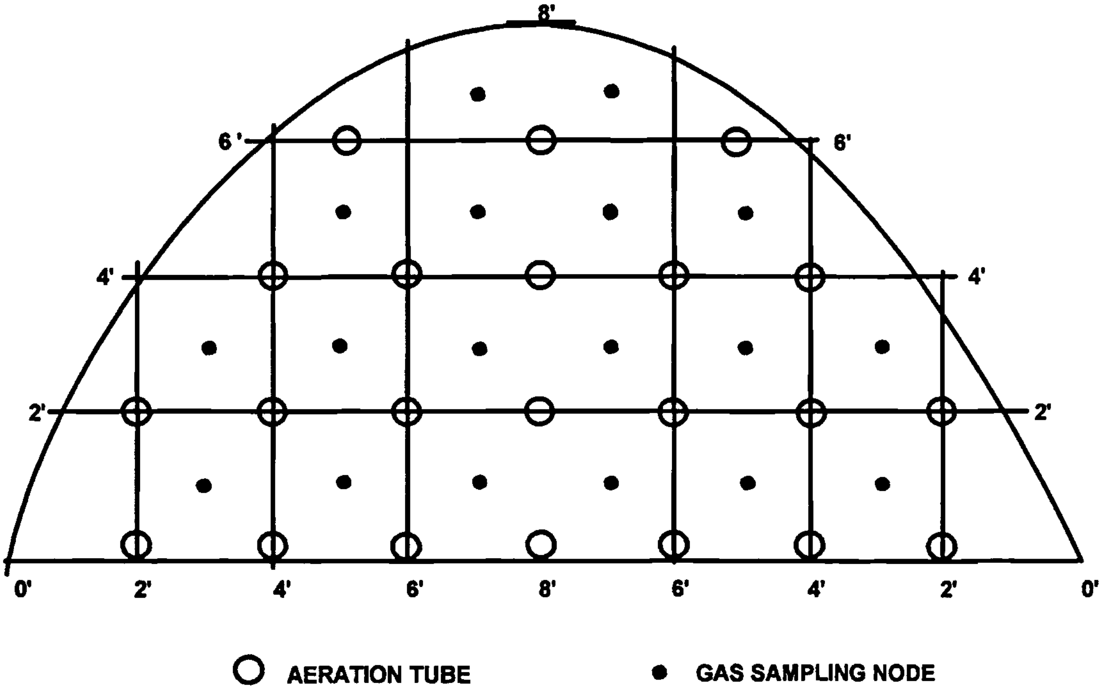


Figure 11. Placement of Gas Sampling Nodes in SP End Cross Section

used the same placement of sampling points as with the other instruments. Figure 12 shows an early stage of the pile construction and porous filter tubing in the center cross section. Figure 13 shows the SP near the latter stage of construction with the passive air flow tubing protruding from the pile and the bottom row of forced-air tubing capped off.

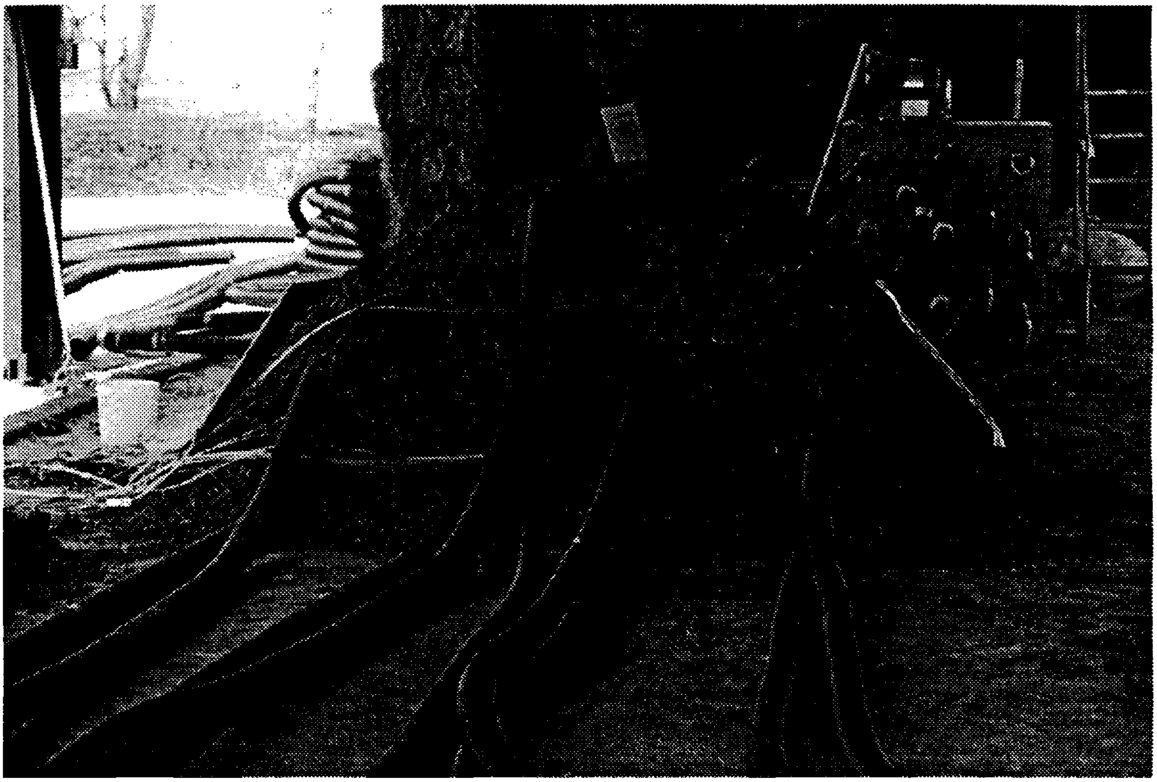


Figure 12. Photo of Early Pile Construction with Porous Filter Tubing in Center

Because the front end loader on the tractor could not reach all the way to the top of the pile, which was 8 ft from the side and 8 ft high, the pile had to be built from side to side rather than completely across the full width. The full height of the pile had to be built by hand by tossing the compost to the peak from a standing position in the raised front-end loader, which slowed construction. Upon completion of construction, the pile was covered with 8 mil black visqueen plastic to retain heat to facilitate the compost startup. The final pile dimensions were 16 ft L x 16 ft W x 8 ft H. Figure 14 shows an end view of the SP after construction was completed.

Figure 13. Photo of SP Near Completion with Forced-Air Tubing Capped Off

Figure 14. Photo of Finished Static Pile

Windrow Pile Construction

Upon completion of the static pile, the windrow was quickly constructed using the remainder of the compost and the front end loader. Pile dimensions were approximately 20 ft L x 5 ft W x 4 ft H.

Compost Pile Monitoring

Oxygen and carbon dioxide levels in the two compost piles were measured using a GasTech CO_2/O_2 gas detector (GasTechtor Model 32520X). A calibration test kit with 2.5% CO_2 in nitrogen and a regulator were used to calibrate the meter on sampling dates. To measure hydrocarbon vapors, a GasTech total hydrocarbon analyzer (Model TraceTech-Tor) was used. This was also calibrated daily with 4400 ppm hexane in air standard. All VOC measurements and calibrations were conducted with the meter set for "No Methane Response." The gas detectors were attached to the porous filter tubes in the SP and to a stainless steel sampling probe used in the WR. Gas measurements were made on the two piles at least 4 times a week.

The WR was scheduled to be turned weekly once it began to self heat. Therefore, instruments were not installed in the pile. For temperature measurements, a stout Type J temperature probe (1/4 in. OD x 10 ft long SS sheath) was used to measure temperatures at several locations in the pile. The probe was connected to a Fluke hand-held digital thermometer (Model 52 K/J). Temperatures were also measured at the same time gases were measured on the WR. A stainless steel gas sampling probe was constructed with a porous tip made of 100 mesh screen, 6 in. long x 3/8 in. diameter and attached to 1/4 in. x 6 ft long SS tubing. The probe was used to measure gases at several locations in the pile.

Compost Sampling

An initial baseline sampling of the compost was made on January 29 from the compost feedstock resulting from the 5 mixed batches. This consisted of 14 discrete grab samples taken about the pile. After the construction of the SP and WR piles was completed and the test begun, grab samples of the compost material were collected from each pile on a scheduled basis. These were placed in 1/2 pint glass Mason jars, labelled, and then stored in an incubator at 4°C for later analyses. Table VII shows the sampling schedule for the two piles. A dozen or more samples from the SP were collected during each sampling event. Initially, 4 or 5 samples were collected from the WR. These consisted of a composite sample from each quadrant of the WR. The sampling event on February 11 included a composite sample of the compost on the surface of the WR pile. The sampling intervals for the WR were established at one week for the first 8 weeks of the study. The sampling intervals for the SP were more frequent than those for the WR because the biodegradation rate of O&G was expected to be higher in the SP. The frequency of sampling the SP was lowered gradually after the fourth week until the end of the test.

Sampling of the SP could not be accomplished using a spade or shovel, as it could be done on the WR, without seriously disturbing the pile and the instrumentation. This was especially true for taking samples from the center of the SP. Therefore, a coring assembly was designed and fabricated to facilitate compost sampling. As shown in Figure 15 and Figure 16, the assembly consisted of a 9-ft long piece of stainless steel (SS) seamless tubing, a pointed plunger tip attached to a 1/2 in. aluminum rod, a tube-plunger driver, and a coring tool attached to the end of a 1/2 in. stainless steel rod. Each rod was outfitted with Delrin bushings to keep it centered in the SS tube. The SS tube was beveled on the edge contacting the compost. The coring tool was both beveled on the edge and notched to form cutting teeth. To drive the SS tube to the desired location, the plunger was inserted into the SS tube and the driver was attached to the plunger with a connecting pin. The tube was

TABLE VII

COMPOST SAMPLING SCHEDULE

Date	Day	Static Pile (No. of Samples)	Wind Row (No. of Samples)
Jan 29	0	14	14
Feb 11	6	12	5 (composites)
Feb 17	12	14	4 (composites)
Feb 22	17	13	Not sampled
Feb 25	20	14	8
Mar 1	24	12	Not sampled
Mar 4	27	12	8
Mar 10	33	Not sampled	8
Mar 11	34	12	Not sampled
Mar 17	40	Not sampled	8
Mar 18	41	12	Not sampled
Mar 24	47	Not sampled	8
Mar 31	54	Not sampled	8
Apr 4	58	12	8
Apr 22	76	12	8
May 13	97	12	8
Jun 6	121	12	8

driven into the side of the pile to the desired depth by a hammering motion of the plunger against the SS tube. The tube penetrated quite easily in most cases. After compaction had occurred, later in the test, driving was more difficult at the 1 ft elevation after penetrating more than 5 ft into the side of the pile. However, one person was always able to remove the tool without mechanical help throughout the test. Eight feet of penetration using this tool seemed to be a good working maximum. Once the SS tube was emplaced, the plunger-driver was removed and the coring tool was inserted. A 1/2 in. drive drill motor was attached to the shaft of the coring tool and was used to cut the straw of the compost and collect the sample. Once the coring tool penetrated a full cup length, it was withdrawn

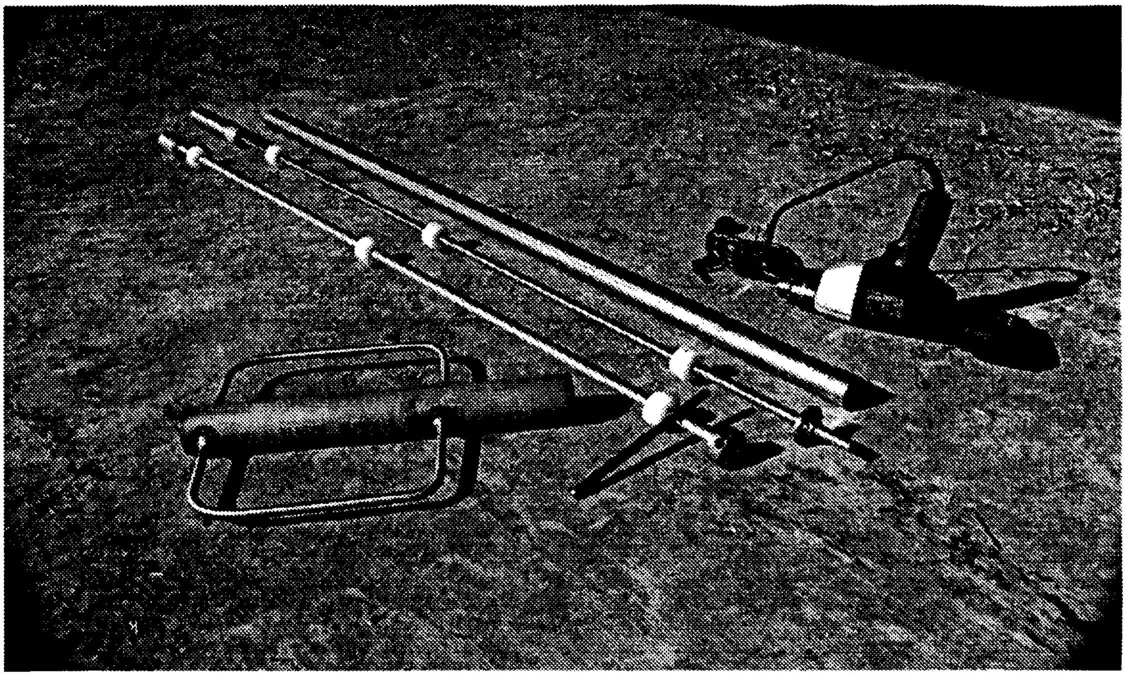


Figure 15. Coring Assembly with Drill Motor

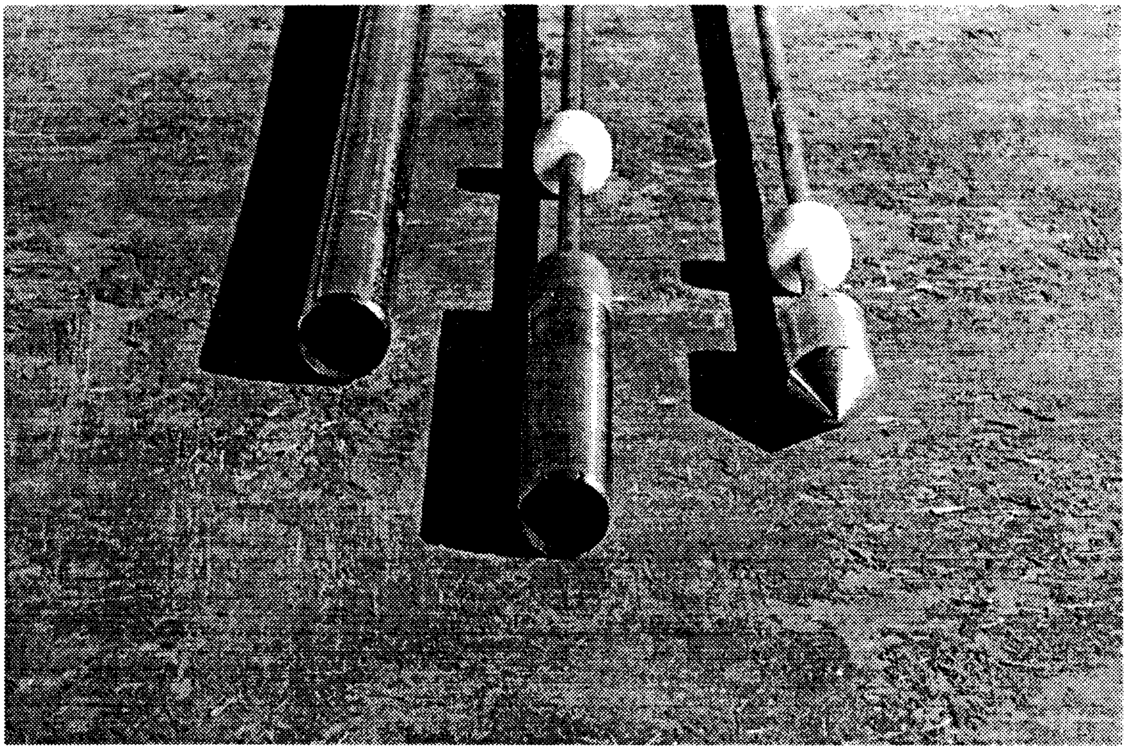


Figure 16. Close-Up of Coring Assembly

and the compost sample was transferred directly into a Mason jar using a spatula. It was discovered that the cutting edge and the notches had to be maintained before or after each sampling event. The tool worked extremely well during the first sampling event on February 11. However, the next time it was used, several samples failed to remain in the tool after they were cored. Even though the cutting edge looked sharp, the tool had to be dressed with a steel file to restore the cutting edge and teeth. This was accomplished quickly and the tool once again performed as it did originally. Appendix C includes a series of photographs showing the use of the coring assembly.

Early in the test, cores were collected from all quadrants of the SP during sampling events. However, some of the lines to the various monitoring devices in the pile were cut by the coring tool. Although attempts were made to avoid cutting them, predicting their exact locations was difficult because of settling and compaction of the pile. Therefore, sampling was largely restricted to the half of the pile closest to the blower which did not have instruments. Complete random sampling was not possible in order to miss air tubes. Cores were then at low, medium, and high elevations and at different depths of penetration in the SP. Survey flags were put in the holes created by the SS tube once it was removed, and the hole was collapsed by hand at the pile surface.

Compost Analyses

O&G Measurement

Several analyses were conducted on the compost samples. The most significant of these was the analysis used to measure the disappearance of the hydrocarbons. U.S. EPA Method 413.1 (1979a) (modified), similar to Method 5520E (1989), was used to measure HCs gravimetrically in compost extracts. This method was chosen because it was simpler and much quicker than TPH and biomarker methods. A larger number of samples could be analyzed with the O&G method than with the other methods, which should have ensured better statistical results. This was especially important for the pilot test with large compost piles which had the potential of large spatial variability in HC concentration.

Compost samples were ground with mortar and pestle with anhydrous magnesium sulfate on a 1:1 w/w basis to sequester moisture and eliminate interference of water with the solvent extraction procedure. The resultant mixtures were placed in extraction thimbles on a Soxtec® extractor (Soxtec system HT2 1045 Extraction Unit connected to a Soxtec System HT Service Unit). The thimble was lowered to the boil position for 30 minutes and then raised to the rinse position for another 5 hours. The service unit was operated at 105°C to heat the methylene chloride extraction solvent. After 5-1/2 hours total extraction time, the solvent was evaporated from the extraction beaker at 60°C until it just disappeared. The extraction beakers and the extracts were placed in a vacuum oven for a minimum of 24 hours at 40°C and full vacuum to evaporate any water that might have invaded the samples during extraction. After being removed from the vacuum oven, the beakers were placed in desiccators for cooling and storage prior to being weighed. The extracts in the beakers were weighed and the O&G was calculated on a dry wt basis by subtracting the MC from the sludge. A detailed procedure of the analysis method is provided in Appendix G.

GC Type Analysis

Weighed O&G extracts were reconstituted with methylene chloride to dissolve the HCs. The dissolved extracts were then analyzed by GC-FID to produce fingerprints or type analyses of the samples' HC components. Although not quantitative, the fingerprints were used to help determine degradation of the HCs. The equipment and the GC method used for the type analysis are described in Appendix D.

BTEX

Composites from the discrete samples were also prepared as before, but instead of being dried for inorganic analyses, they were kept in the incubator at 4°C for other analyses. BTEX was measured on selected composite samples by Soil Analytical Services, Inc. (SASI), College Station, Texas, using Method 8020 with GC-IR in accordance with Test Methods for Evaluating Solid Waste, EPA SW-846 (U.S. EPA, 1986).

Moisture Content

The moisture content (MC) of the materials used or sampled for the study was measured using a Denver Instrument Company IR-100 Moisture Analyzer. The analyzer was programmed to dry a sample placed on its weighing pan at 105°C until it reached a constant weight. The moisture content was calculated as shown in Equation 8.

$$MC = [(\text{Initial wt} - \text{Final wt}) / \text{Final wt}] \times 100 \tag{8}$$

pH

The pH of the compost samples was measured on composites made from the discrete grab samples taken during each sampling event using a method described by Deuel and Holliday (1994). The composite samples were first dried in a drying oven at 105°C to constant weight. Each sample was then run through a soil grinder (Straub Model 4E Grinding Mill) to break down the straw in the compost sample and also to break up the dirt clods formed during drying. The plates were set at a distance large enough to avoid pulverizing the minerals in the soil which could cause an undesirable change in the pH measurement. The ground sample was then mixed by hand and placed in a clean Mason jar. Before measuring pH, a portion of the sample was passed through a 2 mm sieve to separate out large straw and dirt particles. A 1:1 solution was made using 5 grams of the sieved sample and

5 ml of DI water. The sample was stirred briskly by hand with a stainless steel spatula and set aside for 10 minutes. A 2:1 solution was also made at the same time using 5 grams sample and 10 ml of DI water. This 2:1 mixture was not necessary, but it was very easy to prepare and measure at the same time as the 1:1 mixture. After the 10 minute waiting period, the pH electrode was inserted into the mixture and the value read after 10 seconds. The pH electrode and meter were the same ones used to adjust the pH of the nutrient solution added to the compost. They were calibrated using calibration standards at pH 4.0 and pH 7.0.

Nitrogen, Phosphorus, and Total Organic Carbon Analyses

The dried and ground composite samples were sent to Soil Analytical Services, Inc. (SASI), College Station, Texas for nitrogen, phosphorus, and total organic carbon (TOC) analyses. Nitrogen analyses included total Kjeldahl nitrogen (TKN), ammonium-nitrogen, and nitrate-nitrogen. Phosphorus analyses included total phosphorus, EDTA phosphate-phosphorus, and soluble phosphate-phosphorus. TOC was performed by dry combustion after acid digestion to eliminate inorganic carbon from being measured. The methods used in these analyses were in accordance with EPA SW-846 (U.S. EPA, 1986).

Microbial Analyses

Refrigerated composite samples were sent to Pace Inc., Lenexa, KS for microbial analyses. A few selected samples were sent to check the populations to ensure that there was no inhibition of microbial activity. Measurements of total heterotrophs were made by standard plate count. Specific HC degraders were also measured.

Standard plate counts were conducted in accordance with Standard Method 9215B (1989) modified by substituting 1 g of solid matrix sample for the liquid matrix sample. Specific HC degraders were evaluated also in accordance with Standard Method 9215B and modified by substituting a prepared nutrient solution for the specified deionized (DI) water. Solid matrix sample was also substituted for the required liquid matrix sample. The nutrient solution was prepared by combining 50 ml of salt solution A, 50 ml of salts solution B (SSB), 1.0 ml of trace element solution (TES), and 0.1 ml of motor oil. The resulting nutrient solution was autoclaved and then tempered to 40°C before being added to the plate-count agar. Salt solution A was prepared by dissolving 0.2 g of $CaCl_2 \cdot 2H_2O$ in DI water to make 1 liter of solution. Salts solution B and the trace element solution were also prepared with DI water to make 1 liter solutions each. Quantities of specific salts in SSB and TES solutions are listed in Table VIII and Table IX.

TABLE VIII

COMPONENTS OF SALTS SOLUTION B

Salt	gram/liter
$(NH_4)_2SO_4$	2.0000
$MgSO_4 \cdot 7H_2O$	0.1300
KH_2PO_4	3.0000
K_2HPO_4	4.0000
$FeSO_4 \cdot 7H_2O$	0.0060
$MnSO_4 \cdot H_2O$	0.0045

TABLE IX

COMPONENTS OF TRACE ELEMENTS SOLUTION

Salt	gram/liter
H_3BO_3	0.0100
$BaSO_4$	0.0700
MoO_3	0.0100

CHAPTER IV

RESULTS AND DISCUSSION

Treatability Tests

Treatability tests were conducted on the compost materials to determine the ability to mix materials, and the degree or effects of water retention, compaction, and nutrient utilization of the compost. These tests included compost tilling tests, container capacity tests, compaction tests, and respirometry tests.

Compost Tilling Test

There was some concern about what would occur if a tiller was used to mix the straw and sludge together to form the compost. It was thought that the straw might wrap and build up around the tiller blades and make mixing with this equipment ineffective. At first, a Goossen Bale Chopper® was tested and considered for shredding the straw to precondition it. However, shredding was considered an extra step that would add more time and cost to compost preparation and was not performed for the pilot test. The shredded straw was later used in the laboratory treatability studies.

After loosening a plot of soil with the TroyBilt® tiller, a bale of straw was broken open and applied to the tilled earth. The straw and loosened soil blended together well without the straw binding up in the hand tiller. The loamy soil appeared moderately moist, though soil moisture was not measured. Ambient temperature was about 85°F. After observing these encouraging results, the much larger BEFCO tiller was purchased for pilot scale mixing. The BEFCO tiller allowed higher capacity and more vigorous mixing of the compost than the smaller tiller.

Container Capacity Testing

Preliminary CC tests were conducted on 2:1 and 3:1 shredded straw:sludge mixtures in 1000 ml plastic beakers at room temperature. The tests were conducted at these ratios because the composition of the pilot mixture had not yet been established. Ratios of 1:1 and 4:1 were not tested since the former was considered to cause a substantially lower permeability and the latter required an excessive amount of straw. Kriegh (1993) noted that the CC of mixed compost was significantly less than that of the bulking agent alone. He reasoned that the O&G in the sludge inhibited absorption of water by the bulking agent. Consequently, tests were performed to determine the effects of wetting order on the CC of the compost at the 2:1 and 3:1 ratios. Results of these tests were used when mixing the compost for the pilot test. The procedure used in these tests is described in Appendix A. Results are shown in Table X. The CC for each mixture was calculated using Equation 9 while the moisture content (MC) was obtained directly from measurements of the saturated and drained mixtures on the Denver moisture balance. Thus, CC is calculated as:

$$CC = (FW - OW) / OW \tag{9}$$

where: FW is the final weight of the saturated and drained mixture and OW is the original weight of the sludge and bulking agent less their initial moisture contents. Several observations were made from this test:

- The CC ranges for the 2:1 ratio (90-100.8%) and the 3:1 ratio (109.3-121.3%) suggest that moisture retention for this system was not greatly influenced by the wetting order of the components.

- When wetting and mixing the straw and soil at the same time, a 50% total MC provided a very even mixture without clumps or excess water.
- For this system, about 33% total MC was required to obtain a good workable paste when the soil was wetted first. This was observed when 25% wt of additional water was added to the sludge having 16% MC.
- For soil wetted first, the 2:1 ratio tended to clump more than the 3:1 ratio when no additional water was used to raise the MC from 33%.
- When the straw was wetted first (by soaking and then draining), additional water was required upon adding the sludge in order to achieve good mixing. This 2 step wetting process requires additional effort for pilot and field scale mixing.
- 3:1 mixtures had higher average CCs than 2:1 mixtures (114.1% vs. 94.8%).
- Mixtures with straw wetted first had the most compaction.
- Mixtures with sludge wetted first had the lowest CCs within each mixing ratio.

TABLE X

RESULTS OF PRELIMINARY CONTAINER CAPACITY TESTS

Sample	CC%	MC, % wt	Compost Vol, ml	Total Density
2:1 Straw wetted first	100.80	55.50	500.00	1.03
3:1 Straw wetted first	111.75	54.30	620.00	0.71
2:1 Sludge wetted first	90.01	47.50	650.00	0.75
3:1 Sludge wetted first	109.32	47.30	720.00	0.60
2:1 Both wetted together	93.65	49.20	650.00	0.77
3:1 Both wetted together	121.32	53.10	675.00	0.67

Compaction tests were conducted following the preliminary CC tests. Based upon those results, a 2:1 straw:sludge mixing ratio was selected for the pilot study. Therefore, a final CC measurement at the 2:1 ratio was made using whole straw, which was also planned for the pilot, instead of shredded straw. The same three wetting methods were again investigated. Moisture contents of the whole straw used and the sludge were measured for this test and were 15.06% and 13.22%, respectively. Appendix A describes the procedure used to measure the CC for this test. Table XI lists the results of the test.

TABLE XI

FINAL 2:1 CC TEST WITH WHOLE STRAW

Sample Wetted First	Dry wt of Straw, g	Dry wt of Sludge, g	Dry wt of Compost, g	Drained wt of Compost, g	CC, %
Straw	35.67	243.61	279.28	532.63	90.72
Sludge	35.67	243.61	279.28	495.98	77.59
Both	35.67	243.61	279.28	536.46	92.09

A comparison of the data in Tables X and XI show the same trend for the three wetting methods. Wetting the straw first and wetting the straw and the sludge simultaneously produced similar and higher CCs than when wetting the sludge first. The differences between the preliminary and final 2:1 ratio tests were probably due to the shredded straw being able to absorb more water than the whole straw.

Compaction Tests

The test was initiated without a load placed on the compost mixture. Compaction was determined by the decrease in the original column height as the all-thread was turned to compress the mixture. The compost column height, percent compaction, load, air flowrate, pressure drop, and permeability data are shown for each mixture in Table XII and Table XIII. Pressure drops for both mixture ratio tests showed little increase until excess water was observed during compaction. Upon draining the excess water from the lower fitting of the vessel and resuming the tests, the pressure drop decreased substantially in each instance.

TABLE XII

3:1 SP:SLUDGE COMPOST COMPACTION TEST

Col. Ht.	Compaction	Load, lbs	Load, psf	Q, ml/min	P Drop, psi	K, Darcy	Note
431	0.00%	0	0.0	69	0	Inf.	Original compost column uncompacted
381	11.60%	4.56	78.5	74	0	Inf.	
281	**34.80%**	**19.26**	**331.7**	**74**	**0.016**	**10.75**	**Test load greater than load at bottom of 8 ft pile**
273	36.66%	23.5	404.7	74	0.06	2.79	Bubbling of liquid in compost
253	41.30%	27.8	478.7	67	0.05	2.81	
252	41.53%	29.1	501.1	67	0.04	3.50	
251	41.76%	32.5	559.7	67	0.05	2.79	
247	42.69%	37.3	642.3	67	0.06	2.29	
241	44.08%	40.7	700.9	67	0.08	1.68	
240	44.32%	43.9	756.0	67	0.12	1.12	
217	49.65%	66	1136.6	67	0.35	0.35	
217	49.65%	66	1136.6	67	0.015	8.01	Response after 40 ml drained

Some of the key observations of this test are summarized as follows:

- At the maximum flow rate delivered by the rotameter, neither mixture ratio showed a pressure drop when uncompacted. This suggests that air was flowing through the mixture without any restriction. This might be similar to conditions at the very top of a compost pile.

- There was only a slight increase in pressure drop in the 3:1 mixture up to about 45% compaction. The load on the compost at this point was greater than 756 lbs/sq ft. By comparison, the force at the bottom of an 8 ft pile with an initial compost density of 40.52 lbs/ft^3 is 324.2 lbs, or less than half the load under this amount of compaction.

TABLE XIII

2:1 STRAW:SLUDGE COMPOST COMPACTION TEST

Col. Ht.	Compaction	Load, lbs	Load, psf	Q, ml/min	P Drop, psi	K, Darcy	Note
431	0.00%	0	0.0	68	0.001	242.1	Original compost column uncompacted
362	16.01%	9.5	163.6	67	0.033	6.08	
355	17.63%	12	206.6	67	0.065	3.04	Bubbling of liquid in compost
342	20.65%	16.5	284.1	67	0.11	1.73	
320	**25.75%**	**28**	**482.2**	**65**	**0.36**	**0.49**	**Test load similar to load at bottom of 8 ft pile**
313	27.38%	36	619.9	58	1.7	0.10	
312	27.61%	38	654.4	60	1.3	0.13	
311.5	27.73%	40.9	704.3	57	1.4	0.11	
309	28.31%	42.3	728.4	60	2	0.09	
309	28.31%	31.5	542.4	57	0.53	0.28	Response after draining

- Excess water was observed in the 3:1 mixture at 35% compaction by the presence of bubbling.

- There was only a slight increase in pressure drop in the 2:1 mixture up to about 25% compaction. The load on the compost at this point was 482 lbs/ sq ft, or almost the same as the 493.8 lb/sq ft weight at the bottom of an 8 ft pile having a density of 61.72 lbs/ cu ft.

- Excess water and bubbling was observed in the 2:1 mixture at about 20% compaction.

- An increase in pressure drop was observed when the 2:1 mixture was compacted more than 25%.

- Hysteresis was observed by the usual falloff in the load measured after each increase in compaction.

- Excess water was the major contributor to significant increase in pressure drop.

The 3:1 compost mixture had very high permeabilities, greater than 1 Darcy until the most extreme compaction. When the water was drained at that point, the permeability increased dramatically. The 2:1 mixture had high permeabilities up to about 25% compaction. At this point it had nearly 0.5 Darcy permeability. This was still considered good permeability for compacted conditions at the bottom of the pile and the permeability at higher elevations was expected to be much higher. The 2:1 mixture, therefore, appeared to be a good choice for the pilot study. Under the most compacted conditions for the 2:1 mixture, the solids maintained 72% of the original volume. Assuming a residual pore volume of 35% under such compacted conditions, the solids were estimated to comprise 47% of the original volume. Therefore, the uncompacted pore volume of the 2:1 mixture was 53%. A conservative value of 65% pore volume was used to determine the airflow turnover rate in the pilot test.

Respirometry Tests

Respirometry tests were conducted to observe the effects of straw to sludge ratios and nutrient addition on oxygen consumption. Four straw:sludge mixing ratios plus a baseline sludge sample were tested to determine the optimum mixing ratio for biological activity. Figure 17 shows the cumulative oxygen uptake profiles for the respiration test. Each profile represents the average of three tests at a single mixing ratio. It is apparent that cumulative oxygen consumption increases as the straw to sludge ratio increases. This can be accounted for by the mixture having a larger bioreactive component as the ratio increases. There is a slightly increased gap between the 1:1 and 2:1 curves compared to the gaps between the other curves. However, it is uncertain whether or not the gap is statistically significant. Using these results and the compaction test results, a 2:1 mixing ratio was selected for the pilot study.

A second respirometry test was conducted to investigate the effects of varying the nutrient augmentation on a 2:1 compost mixture. Figure 17 shows the cumulative oxygen

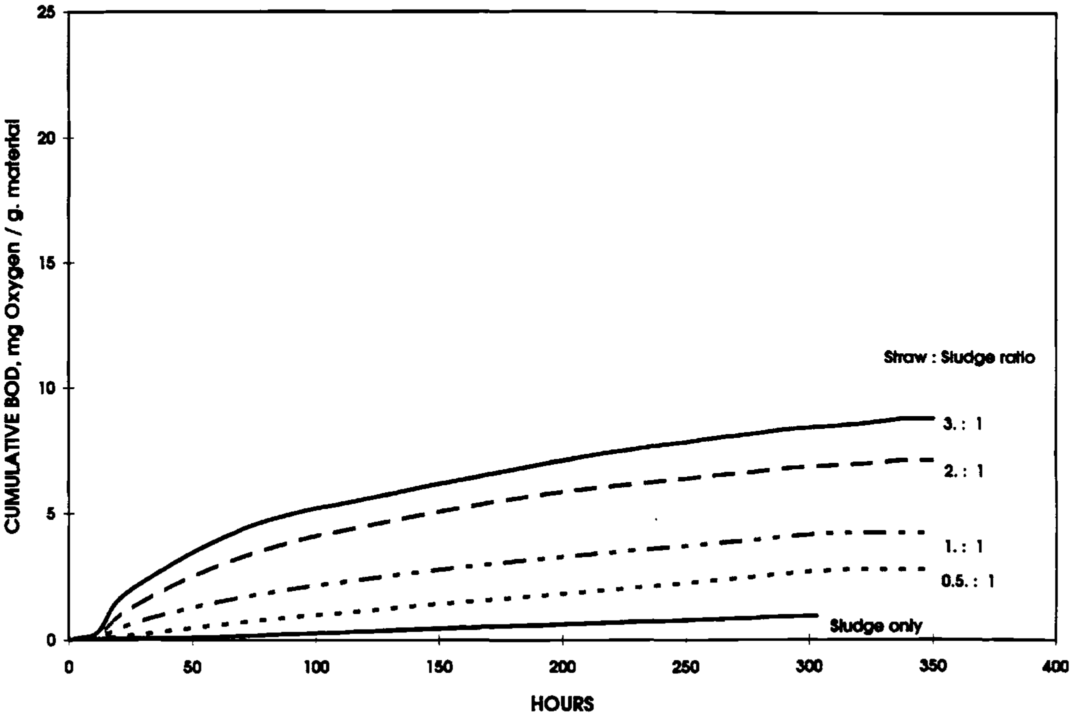


Figure 17. Cumulative Oxygen Uptake of Various Compost Mixtures

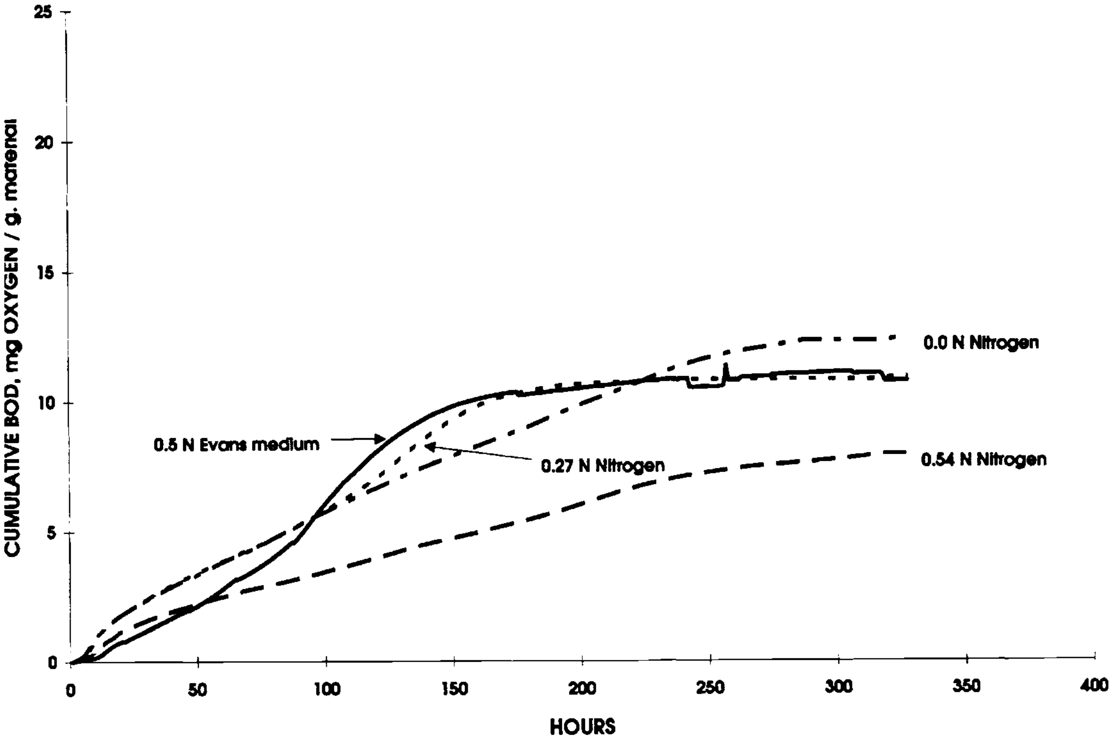


Figure 18. Cumulative Oxygen Uptake for the 2:1 Straw:Sludge Compost Ratio with Different Nutrient Additions.

uptake profiles for the four different nutrient conditions. The unamended base condition had the highest cumulative oxygen consumption of the four and indicates that there was no apparent advantage in adding chemical nutrients or micronutrients. Additionally, the curves suggest that 0.54 N nitrogen as urea may have been somewhat inhibitive to biological respiration. The compost mixture amended with Evans mineral medium did not indicate that there was any advantage in adding micronutrients when compared to either the base condition or to the 0.27 N nitrogen condition. From these data, a decision was made to supplement the pilot compost with urea nitrogen at a concentration of 0.27 N. Although phosphate addition was not included in the respirometry study, a decision was made to include it in the nutrient supplement for the pilot test. The chemical nutrients were considered a small cost and insurance that the pilot would not be nutrient deficient.

Compost Mixing for the Pilot Test

The contaminated soil or sludge feedstock was mixed to homogenize it for use in the compost mixture. The sludge was mixed for four hours with the tractor-tiller. The average O&G concentration of 14 analyzed grab samples was 3.77% dry wt. Using the homogenized sludge with straw as the bulking agent, 5 batches of compost were prepared and then mixed together over the next 17 days.

The weather conditions during the preparation of batches 1 and 2 were below freezing which complicated the process (Figure 19). Low temperatures caused the valves of the nutrient mix tanks and water lines (hoses) to freeze if they were filled with liquid and circulation in them was not maintained. Special effort was required to maintain operation of equipment and avoid equipment breakage from freezing water.

Cold temperatures also lengthened the amount of time it took to prepare the nutrient solutions. The nutrients were added in solution rather than as dry chemicals so that the self-heating process in composting would not be delayed and so that the pH of the mixture

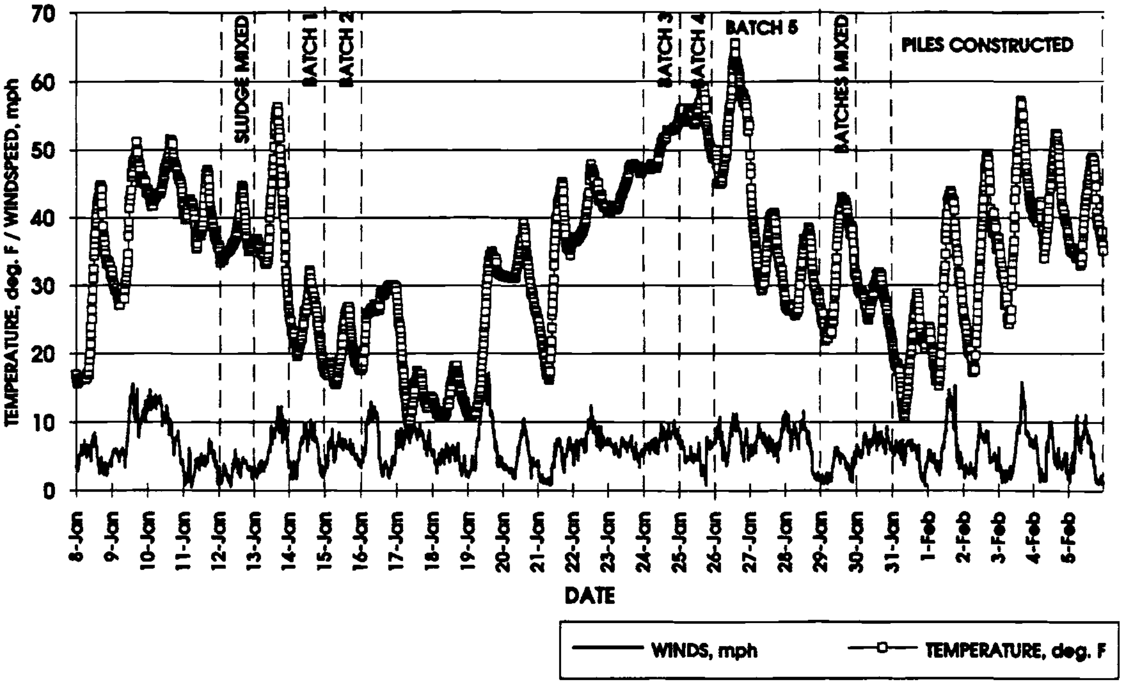


Figure 19. Ambient Weather Conditions During Compost and Pile Preparations.

could be adjusted to normal conditions (pH 6 to 8). Urea appeared to solubilize quite well even though the solution temperatures were in the low 30°F range. However, the superphosphate nutrients did not readily go into solution, so hot water was used to facilitate dissolving. The pH of the solutions prior to adjustment with sodium hydroxide (NaOH) pellets was about 2.0. About 1500 grams of NaOH was added to each batch of solution to increase its pH. Under these conditions, it took about two hours to prepare and neutralize one batch of nutrient solution.

The cold weather also affected the compost batch mixing. When adding liquids to batches 1 and 2, the compost partially froze. This made it difficult to observe whether or not the compost was at a proper moisture level. When too much fresh water was added at a time, the tractor slid on the ice created by the excess water frozen to the concrete. When the compost was too dry, the sludge would not form into a paste well enough to mix with the straw.

After the first two batches were made, ambient temperatures dropped to 9 to 19°F before rising above freezing again. Weather conditions and work schedules halted mixing batch 3 for 8 days until January 24. A temperature probe was inserted into batches 1 and 2 and their internal temperatures were about 28°F. They were uncovered and spread out to thaw so that they could later be mixed together more easily with the last 3 batches. No further delays in mixing and pile construction occurred after that time. Even though the cold weather was unfavorable for such activities under most circumstances, it benefited the study by retarding biological activity during the protracted preparation and pile building time periods.

When the first batch was made, the sludge aliquot was spread out in a long row with the straw bales lined up alongside the row. The bales were moistened with the nutrient solution so that they could absorb the liquid. Interestingly, the straw appeared not to be very absorbent and the liquid quickly soaked through to the concrete. About 60 gallons of the nutrient solution were applied to the 25 bales of straw. The remaining 40 gallons of solution were then applied to the sludge. The sludge was tilled at this point. The sludge had large clods that easily broke down into smaller ones and eventually became somewhat pasty.

It was originally believed that the straw could quickly be added to the mass and mixing could be accomplished in relatively short order. However, the straw had to be added into the mixture by hand by one person, a little at a time, while tilling was conducted by the tractor-tiller operator. Water was added intermittently to maintain a good workable consistency. About 4 to 6 passes with the tiller were required to mix in a freshly applied layer of straw. The process was extremely tedious and each batch took 10 to 12 hours to mix and homogenize even though the tiller itself worked very well in mixing the components. The straw did not bind up around the tiller blades and the clods of sludge were completely broken down.

The remaining compost batches were prepared by first wetting the sludge with enough liquid to form a paste and break down the clods by tilling before straw was added to it. This was decided after observing that the straw did not immediately soak up the nutrient solution when it was in bale form. The treatability tests indicated that the CC would not be highly affected by this change in wetting order. Addition of straw and liquids to the remaining compost batches was then performed on the interval basis previously described. Figure 20 shows one of the batches after mixing was completed. Figure 21 shows a close-up of the consistency of the mixture. The materials were blended together quite well. Some matting of mixed compost did occur from compaction of the wheels of the tractor.

Figure 20. Photo of Finished Compost Batch

The first two compost batches were mixed on January 14 and 15 and the last three were mixed on January 24 through 28. The interruption between the second and third

Figure 21. Close-Up Photo of Compost Mixture (felt-tip pen in foreground)

batches was due to temperatures well below freezing. The five batches were mixed together on January 29, and 14 grab samples were taken from the final mixture.

Static Pile Construction

Because of the short reach of the front end loader, the SP was built from side to side rather than from the ground up while installing the specified equipment. Construction of the first side was quicker than the second side since access to the pile with the front end loader was unobstructed at this point. The first side settled more because it was not as compacted during this period of the SP construction. As shown in Figure 13, the heights of the air flow tubes are lower on the left side of the photograph than on the right side. The SP construction was completed after 1 ft of compost was added to the end of the pile, where the forced-air tubes were capped and the thermocouples and porous filters were embedded. The final SP dimensions were 16 ft x 16 ft x 8 ft. This pile was large enough to observe discrete differences in internal pile temperatures and gas concentrations. It also allowed enough space for 12 sampling events of 8 cored samples each without a major disturbance in the pile. A smaller pile with similar instrumentation might have been more affected. The pile was also large enough to easily self heat, especially during the cold months of the year.

In total, compost mixing and pile construction took two weeks, excluding down time, which was twice what was originally planned. A field compost application could be expected to require much less time for mixing and construction on a per volume compost basis. Extensive mixing to homogenize compost would not be as critical for a field project as it would for a treatability or pilot test. The amount of pile monitoring equipment installed in a field application project would also be greatly reduced.

Windrow Pile Construction

The WR was constructed from the remaining compost. The material was very simply piled in the prescribed dimensions of 20 ft L x 5 ft W x 4 ft H. The total preparation time was about ten minutes, which included some hand shoveling to neaten the pile. The long axis of the WR was oriented from east to west with the east end close to the edge of the concrete pad and the west end near the center of the pad. This orientation allowed the pile to be easily turned with the front end loader by simply approaching it from one side and rolling it over. The east end received sunlight in the morning while the west end remained in the shade throughout the day. This orientation was probably responsible for some of the temperature differences observed during the test.

Static Pile Operation

The SP test was run concurrent with the WR test both of which lasted for 121 days. During this time the internal temperatures of the SP were constantly monitored. Gas measurements were collected 4 times a week and core samples were collected periodically. The air flow was turned on and off during the test and its flow was adjusted.

Temperature Effects and Blower Operation

Initial Self Heating Period. Initial core temperatures in the center cross section at the start of the test were 40 to 45°F and increased 7 to 10° during the first week. During this same period, ambient temperatures, shown in Figure 22, ranged from 9 to 60° and averaged about 35°. Figure 23 shows the core temperature response during the first 20 days with profiles representative of the temperatures at the 4 elevations. On about day 8, the core temperature began to increase exponentially. The temperature increase at the 1 ft elevation showed a significant lag compared to the 3, 5, and 7 ft elevations. This was most likely caused by the concrete pad, which had been previously chilled from exposure to January temperatures, acting as a large heat sink. This effect might have been minimized had the pile been built upon straw or some other insulating material. The 3 and 5 ft elevations, near the center of the pile, were the most insulated and were able to retain their temperatures better than the 1 and 7 ft elevations. The 7 ft elevation appeared to benefit from heat rising from the lower elevations. During the first 10 days while the pile was heating up, the blower was not turned on to circulate air into the pile.

Blower Operation

Turnover Rates. On the tenth day, the blower was turned on to pull air through the pile under vacuum. At this point the pile was still covered with plastic sheeting, so the outside air was brought into the pile through the passive tubes. When the blower was started,

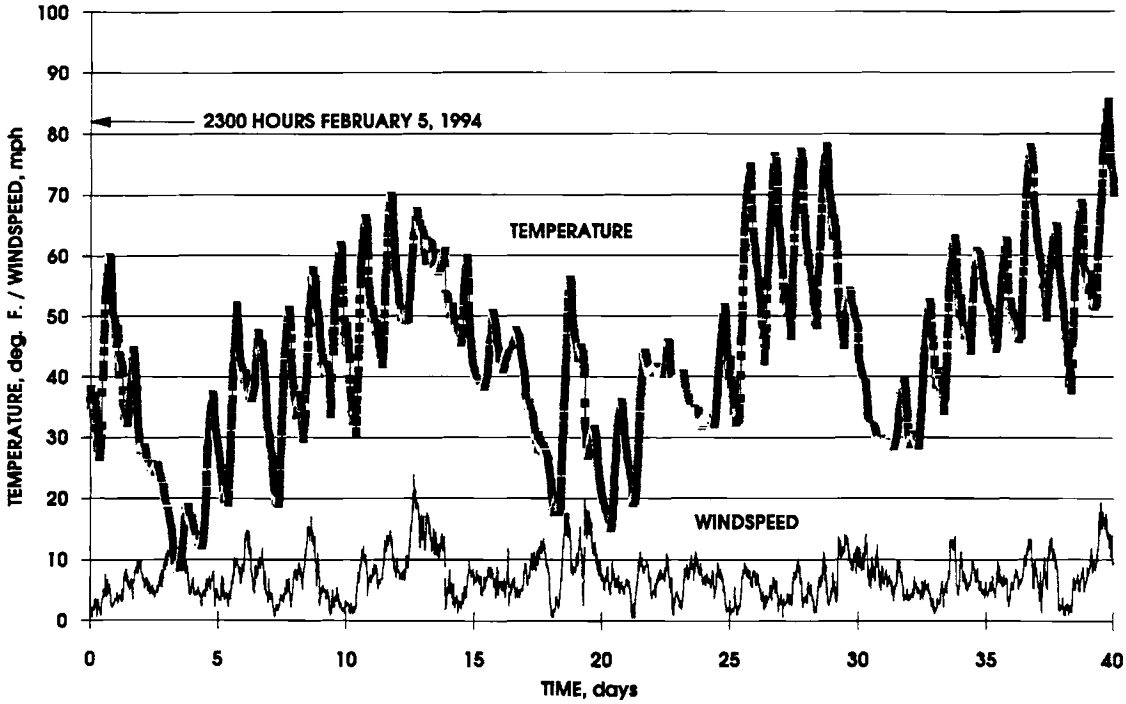


Figure 22. Ambient Weather Conditions During the First 40 Days of Composting

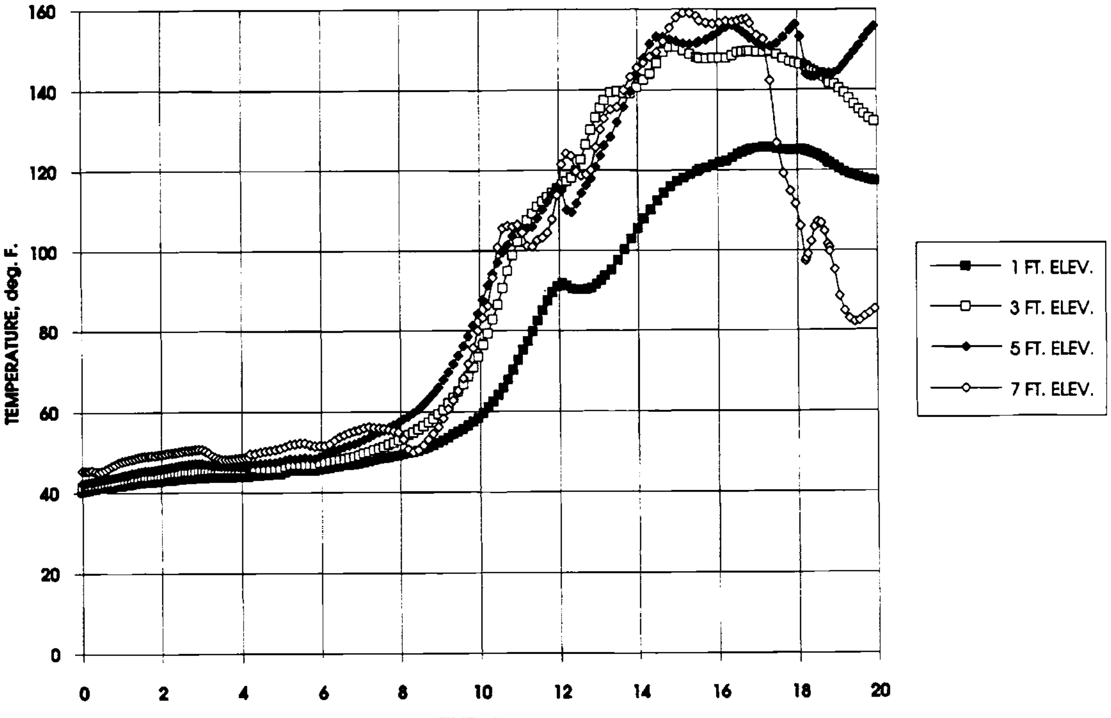


Figure 23. Core Temperature Response in SP During First 20 Days of Composting

the air flow rate was set to allow 10 pore volumes (PV) per day of exchange. The rate was based upon 1134 ft^3 total volume of the pile times an estimated 65% porosity derived from the compaction tests which produced a PV of 800 ft^3. The tubes at the 6 ft elevation were assumed to require one-half the flow as the ones at the 2 ft elevation since the pile was tapered toward the top and the tubes were half as long. Therefore, these 3 tubes each used 5.9% of the flow while the 7 tubes at the 2 ft elevation each used 11.8% of the flow. Since an air flow velocity meter was used to calibrate the airflow in each tube, the required velocity was determined by dividing the desired volume of air by the cross-sectional area of the 3 in. diameter air flow tube. Equation 10 shows the formula used to calculate the air velocity for an individual tube for a desired PV/day turnover rate.

$$V_{fpm} = \frac{PV}{day} \times \frac{day}{1440 \text{ min}} \frac{800 \text{ ft}^3}{PV} \times \frac{1}{0.049 \text{ ft}^2} \times \textbf{\% of total flow} \tag{10}$$

where: V is the air velocity for the individual tube to be calibrated at the desired pile PV/d turnover rate. Table XIV shows some of the calculated velocities in the tubes for flow rates used during the test.

TABLE XIV

CALCULATED VELOCITIES FOR FORCED-AIR TUBES FOR CALIBRATION AT SPECIFIC SP AIR DAILY TURNOVER RATES

PV/d	V_{fpm} for 6 ft Tubes	V_{fpm} for 12 ft Tubes
10.00	6.70	13.40
20.00	13.40	26.80
40.00	26.80	53.60
80.00	53.60	107.20

The Wood's speed controller was used to set the blower speed for the desired PV/d flow rate. The speed controller functioned by controlling the frequency of the electricity provided to the blower. A turnover rate of 10 PV/d required such a low air velocity that the speed controller was initially set for an output frequency of 2.4 Hz. Calibration was difficult because the air velocities required were also low. A considerable amount of interference from ambient winds was observed on the display of the velocity meter as the reading was quite unstable. This interference was finally attributed to south winds of 8 to 13 mph blowing against the pile which was uncovered at the time and into the ends of the passive tubes and permeating through the pile and into the forced-air tubes. When the cover was replaced, the interference was reduced and calibration was possible. Calibration was tedious since adjusting the airflow in one tube affected the flow rates in all of the others. Three or more iterations of individually adjusting flow rates in all of the tubes were required to properly calibrate the airflow.

The blower was initially set at the 10 PV/d turnover rate because it was uncertain how much cooling would occur from the forced aeration. Three days later (day 13), the SP core temperatures appeared to be unaffected by the forced air, so the blower speed was doubled and the cover was removed. By day 16 the core temperatures at the 3 upper elevations had reached 150 degrees, so the blower speed was increased again to 5 times the previous setting, resulting in a turnover rate of 100 PV/d. The cover had been replaced earlier in the day. That evening, the ambient temperature began to decrease from above 40°F into the low 30°s. Since the blower was pulling air from the pile, the moisture in the air condensed over the night in the uninsulated and chilled tubes leading from the pile to the air distribution box. By the next day (day 17) most of the lines were full of water. Air flow in some of the tubes was restricted enough that gurgling could be heard as the blower pulled air through them. The tubes were disconnected and drained. The blower speed was reduced and the tubes were recalibrated. The temperature of the SP was still high, so the cover was removed for the duration of the test. At this point, a decision was made to calibrate all tubes at the same air velocity. It was believed that this would help cool the hot upper elevations and still provide the lower elevations with oxygen for biological activity. The recalibration then provided a calculated 30 PV/d turnover rate at the 6 ft elevation and a 15 PV/d turnover rate at the 2 ft elevation. At the end of the day, the tubes were drained again before leaving the facility. The ambient temperature continued to drop overnight and reached a low of about 18 degrees.

The next day (day 18), the tubes were drained and the blower direction was reversed to blow air into the pile to eliminate the problem caused by the water condensation. The blower was momentarily increased to full speed at 60 Hz with the controller. When this was done, a cloud of steam bellowed from the top of the pile. The blower was set at a speed for 60 and 30 PV/d turnover rates in the upper and lower elevations, respectively. After just a few minutes, the passive tubes at the surface of the pile began to show water vapor and were warm to the touch. When the blower direction was finally reversed, there was not enough time left in the day to calibrate the tubes. On day 19, the tubes were disconnected for the last time to drain the residual moisture left in them. After they were reconnected, the blower speed was set at 19.2 Hz on vacuum and the tubes were recalibrated for an average 105 and 70 PV/d upper and lower turnover rate, respectively. Calibration was again made difficult by 10 mph winds with the cover removed. After recalibration, the blower direction was again set for positive pressure or forced air. The tubes would have been calibrated on forced air instead of on vacuum, but the air velocity meter readings were quite erratic. A final calibration under vacuum operation at 19.2 Hz was conducted on March 1, 1994 (day 31). Winds were about 5 mph and did not cause large fluctuations in the velocity meter during calibration. The calculated turnover rates in the upper and lower tubes were 155 and 70 PV/d, respectively.

The blower operation was varied a few more times after this including an accidental shutdown for 3 days. The blower operation for the entire test is shown in Table XV.

<u>Temperature Effects from Blower Operation</u>. Changes in pile temperature as a result of blower operation are difficult to establish. Figure 24 shows the temperature profile of the core temperatures for the entire test. A comparison of this figure with Figure 22, which shows the ambient temperature profile, suggests that the SP core temperatures were more

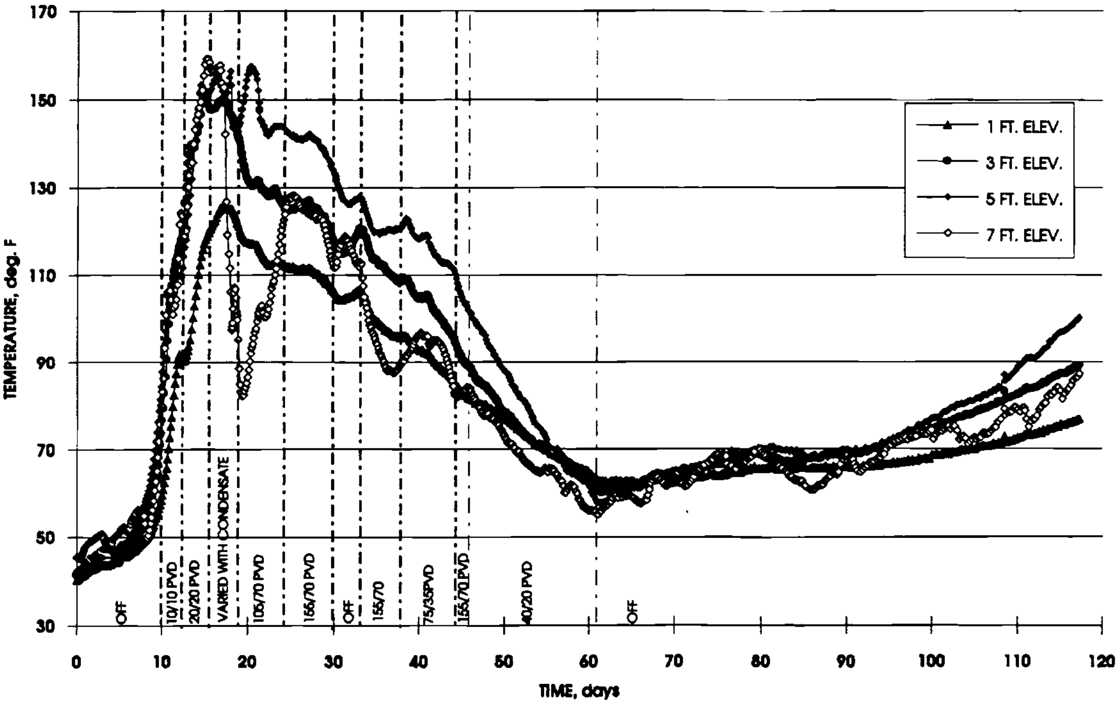


Figure 24. SP Core Temperatures During the 121 Day Test Period

TABLE XV

STATIC PILE BLOWER OPERATION DURING TEST

	Test Day	Direction - Freq., Hz.	PV/d Upper Tubes	PV/d Lower Tubes	Event
February 5	0.00	--	0.00	0.00	Initial self-heating
February 15	10.00	Vacuum--2.4	10.00	10.00	Start blower; calibrate tubes
February 18	13.00	Vacuum-4.8	20.00	20.00	Inc. rate; remove cover
February 21	16.00	Vacuum--24	100.00	100.00	Inc. rate; replace cover
February 22	17.00	Vacuum--4.8	30.00	15.00	Drain tubes, lower rate, calibrate, remove cover
February 23	18.00	Forced--9.6	60.00	30.00	Drain tubes, reverse blower, inc. rate
February 24	19.00	Forced--19.2	105.00	70.00	Drain tubes, calibrate, inc. rate
March 1	24.00	Forced--19.2	155.00	70.00	Calibrate
March 8	31.00	--	0.00	0.00	Blower stopped at 9 A.M. from storm
March 11	34.00	Forced--19.2	155.00	70.00	Blower restarted after gas sampling
March 15	38.00	Forced--9.6	75.00	35.00	Lower rate
March 21	44.00	Forced--19.2	155.00	70.00	Inc. rate after gas sampling
March 22	45.00	Forced--4.8	40.00	20.00	Lower rate after gas sampling
April 8	62.00	--	0.00	0.00	Stop blower permanently
April 14	68.00	--	0.00	0.00	Seal passive air tubes

influenced by ambient conditions than by aeration. This is especially evident on day 17 when ambient temperatures dropped sharply. Figure 23 more easily shows that when the cover was removed on day 17 to help reduce the high temperatures, the 7 ft elevation, which was 1 ft from the surface, began to drop significantly and tracked the ambient temperature. The temperatures at the 1 and 3 ft elevations also decreased, but not as sharply. Temperatures at the 5 ft elevation oscillated around the 150°F level and appear not to have been affected by ambient temperatures. It is possible that most of the flow from the forced-air tube at the 6 ft level was directed upward in the path of least compaction and higher permeability. This could have prevented cooling at the 5 ft level. This trend was not duplicated at the 1 ft level which was also below a forced-air tube. In this case, it would have to be assumed that compaction and permeabilities at the 1 and 3 ft levels were similar.

Once the air control problems were overcome, the air flow rate was increased to 1/3 of the blower capacity. This coincided with the sharp drop in the ambient temperature so it was difficult to determine to whether the pile was being cooled by effective aeration, by the cold ambient temperature, or both. A decision was made not to increase the air flow rate since the temperatures in three of the four elevations were decreasing. By this time, the 1 ft and 7 ft elevations were in the mesophilic temperature range (75-113°F) and the 3 ft elevation appeared as though it would also decrease into that range. The effect of forced aeration is perhaps best seen in the slight increase in temperatures at all elevations from day 31 to day 34 when the power to the blower was interrupted. When aeration was resumed at the previous rate, the temperatures dropped sharply at all levels in the SP. These trends do not appear to be strongly influenced by ambient temperatures during that period. The pile temperatures began to increase on day 31 before the low ambient temperature did. Then when the blower was turned on again, the pile temperatures decreased while the ambient temperatures increased. Subsequent changes in the aeration rate during the test had less dramatic effect as the biological activity had slowed down. By day 35 all core elevation temperatures were in the mesophilic range. On day 62 the blower was shut off. By this time the core elevation temperatures had converged to the ambient temperature which was about 60°F. The gradual increase in the core temperatures after that time closely followed the steady incline of the ambient temperature as the test proceeded into springtime. Figure 25 and Figure 26 show the ambient temperature profiles for the last 80 days of the test.

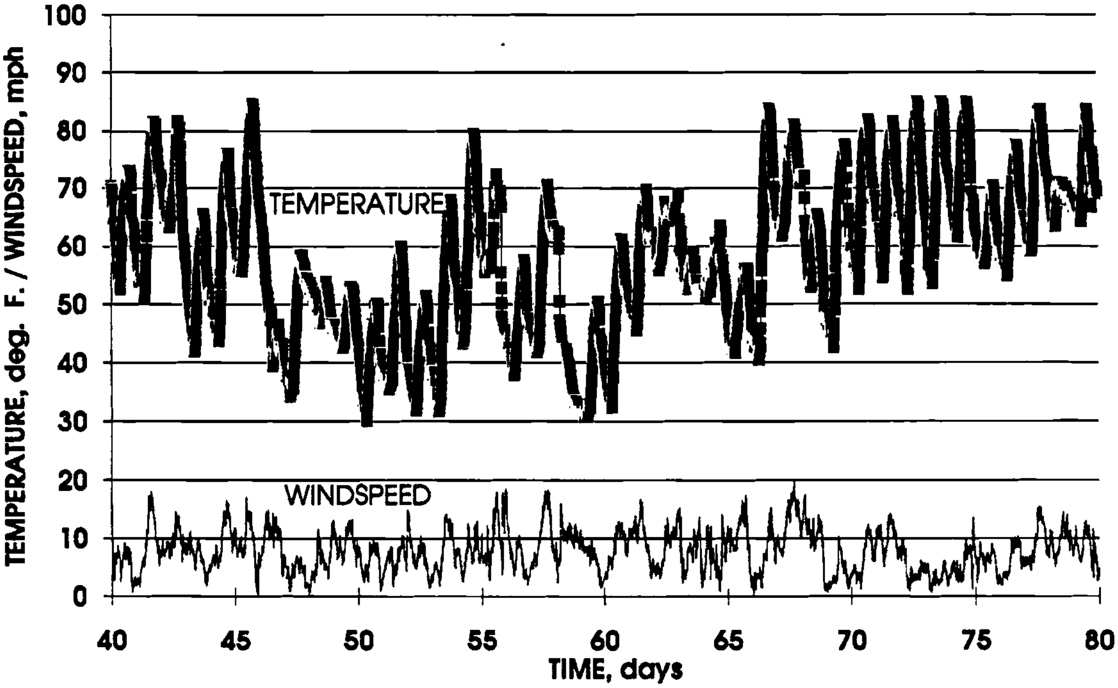


Figure 25. Ambient Weather Conditions from Day 40 to Day 80 During the Compost Pilot Test

<u>Temperature Profiles</u>. Figure 27 shows the specific locations of the numbered thermocouples in the two cross-sections of the SP. Thermocouples T0 through T19 were located in the center cross section and T20 through T39 were located in the end cross section. The temperatures in the two cross-sections can be viewed collectively in Figure 28 and Figure 29. A wide range of temperatures can be seen in these profiles, more markedly in the end cross section of Figure 29. These are a bit difficult to comprehend so the temperatures at specific elevations were plotted separately. Figure 30 shows the temperature profiles for 4 elevations in the center cross section while Figure 31 shows the same for the

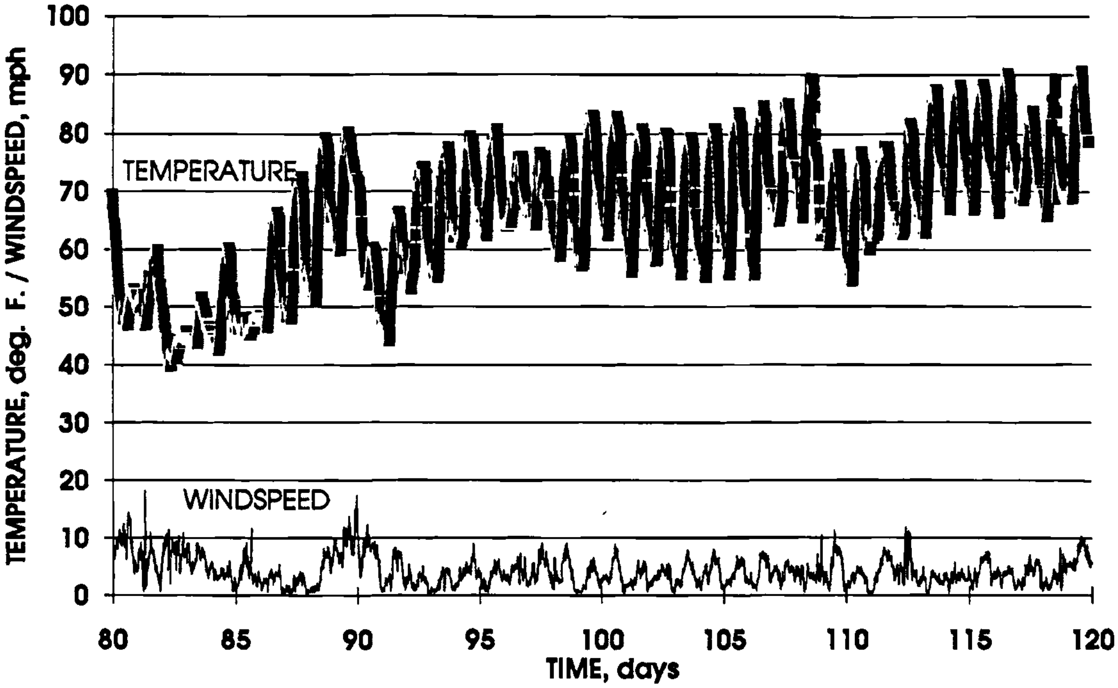


Figure 26. Ambient Weather Conditions from Day 80 to Day 120 During the Compost Pilot Test

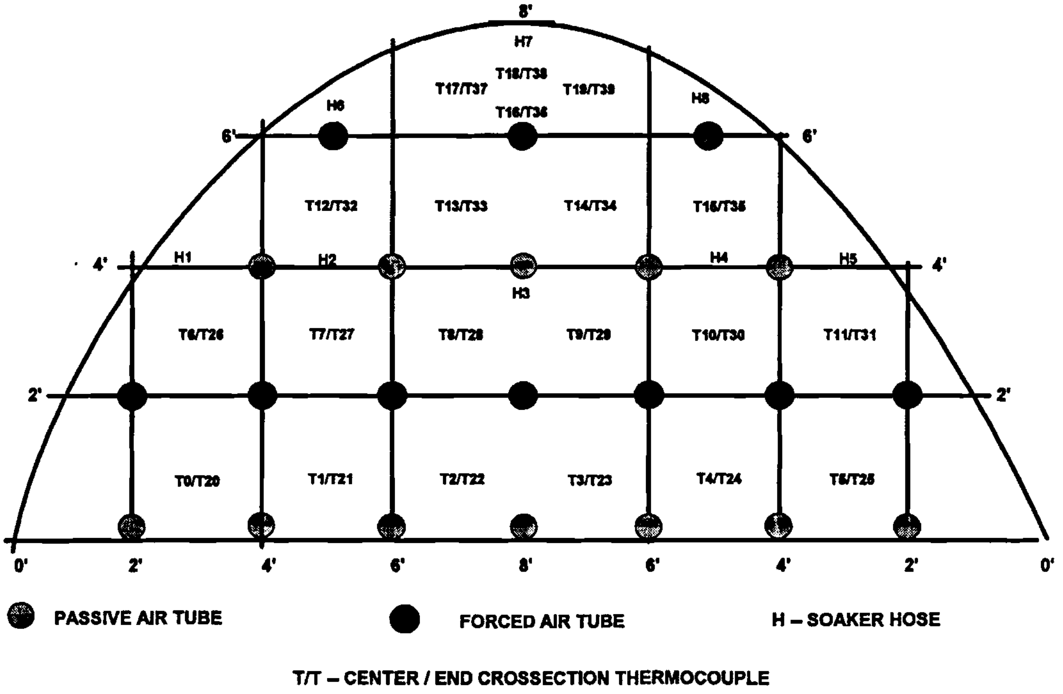


Figure 27. Cross Section Showing Thermocouple Placement

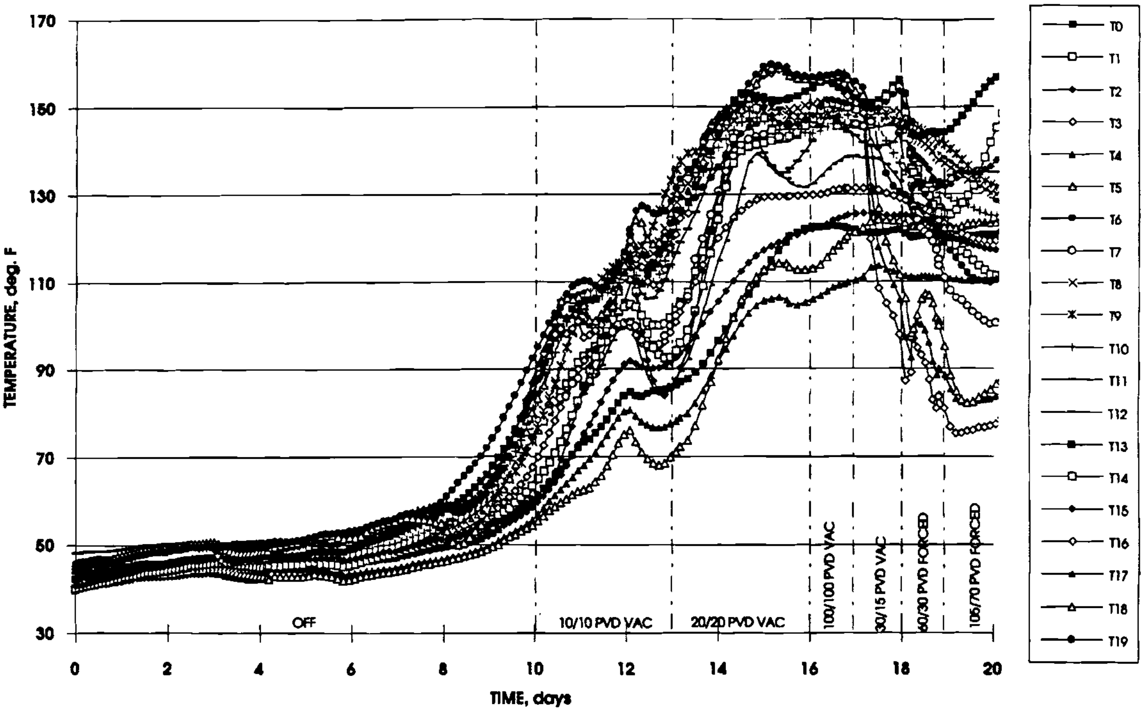


Figure 28. Collective Temperature Profiles of All Thermocouples in Center

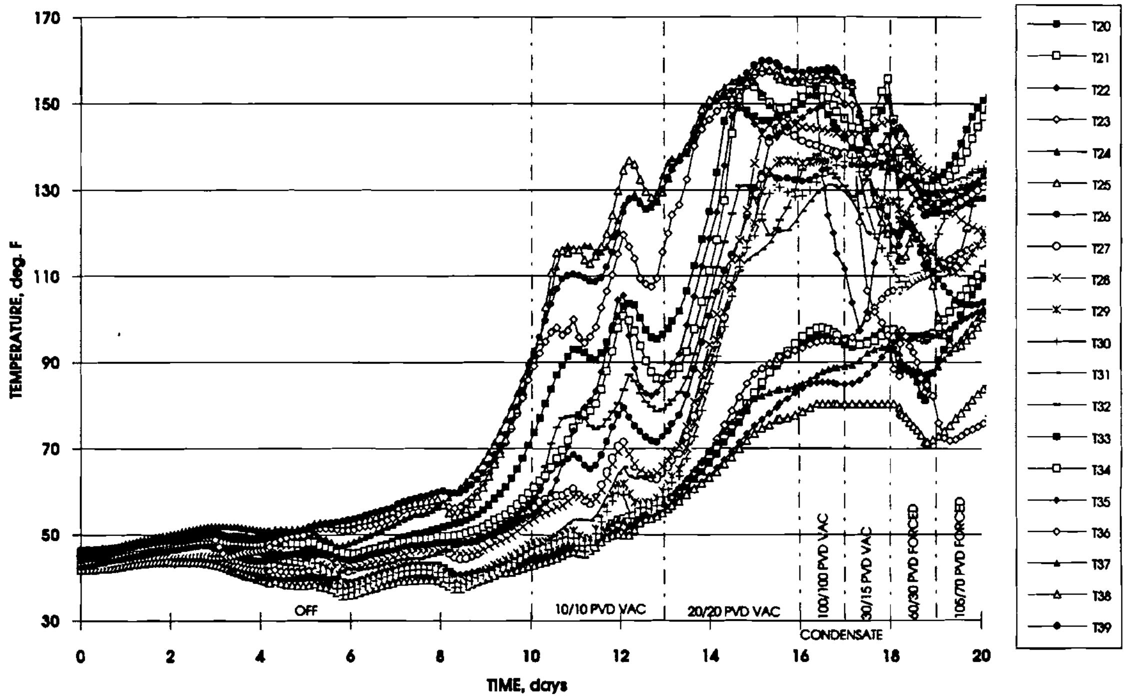


Figure 29. Collective Temperature Profiles in End Cross Section

end cross section. When grouped in this manner, it can be seen that the temperatures at each level in each cross section were generally the same. Temperature profiles at individual locations that did not match their respective groups as well were likely caused by differences in positioning near boundaries. Such differences are seen around day 20 at the 7 ft elevation where the thermocouples T16 and T36 were placed 6 inches below the 7 ft elevation and closest to the center aeration tube. These locations experienced greater cooling during this low outside temperature period than all other positions in the pile. T18 and T38 were 6 inches above the 7 ft elevation and were closest to the top surface and were most affected by ambient temperatures. Thermocouples T17 and T19 in the center cross section and T37 and T39 in the end cross section were placed at the 7 ft elevation. Except for T17, they had higher temperatures during this time than the others in the 7 ft elevation "group." Other standouts were either caused by hot spots of increased biological activity or from inaccurate positioning near boundaries or air tubes. The temperature plots shown in Figure 23 and Figure 24 were chosen as most representative of the group at each elevation. When the center and end cross-sections at each elevation are combined, the boundary effects can be observed. Figure 32 shows that the temperature maximums at the 1 ft elevation are similar but that there was a lag in the temperature increase, and the maximum was not sustained quite as long. This trend is less dramatic as the elevation increases as demonstrated in Figure 33, Figure 34, and Figure 35. Except for the 1 ft and the 7-1/2 ft elevations temperature response appears to be influenced more by elevation than by position near the boundary. Temperature maximums 1 ft inward in the end cross section matched their counterparts in the center cross section reasonably well. An encouraging point is that heat is maintained in the SP near the surface of the pile at most elevations. Therefore, biodegradation should proceed to the same degree throughout the pile except for the very near surface of the pile, the part near the ground surface, and the lower perimeter of the pile. These data also suggest that temperature in a very robust system might be controlled by reducing the pile height.

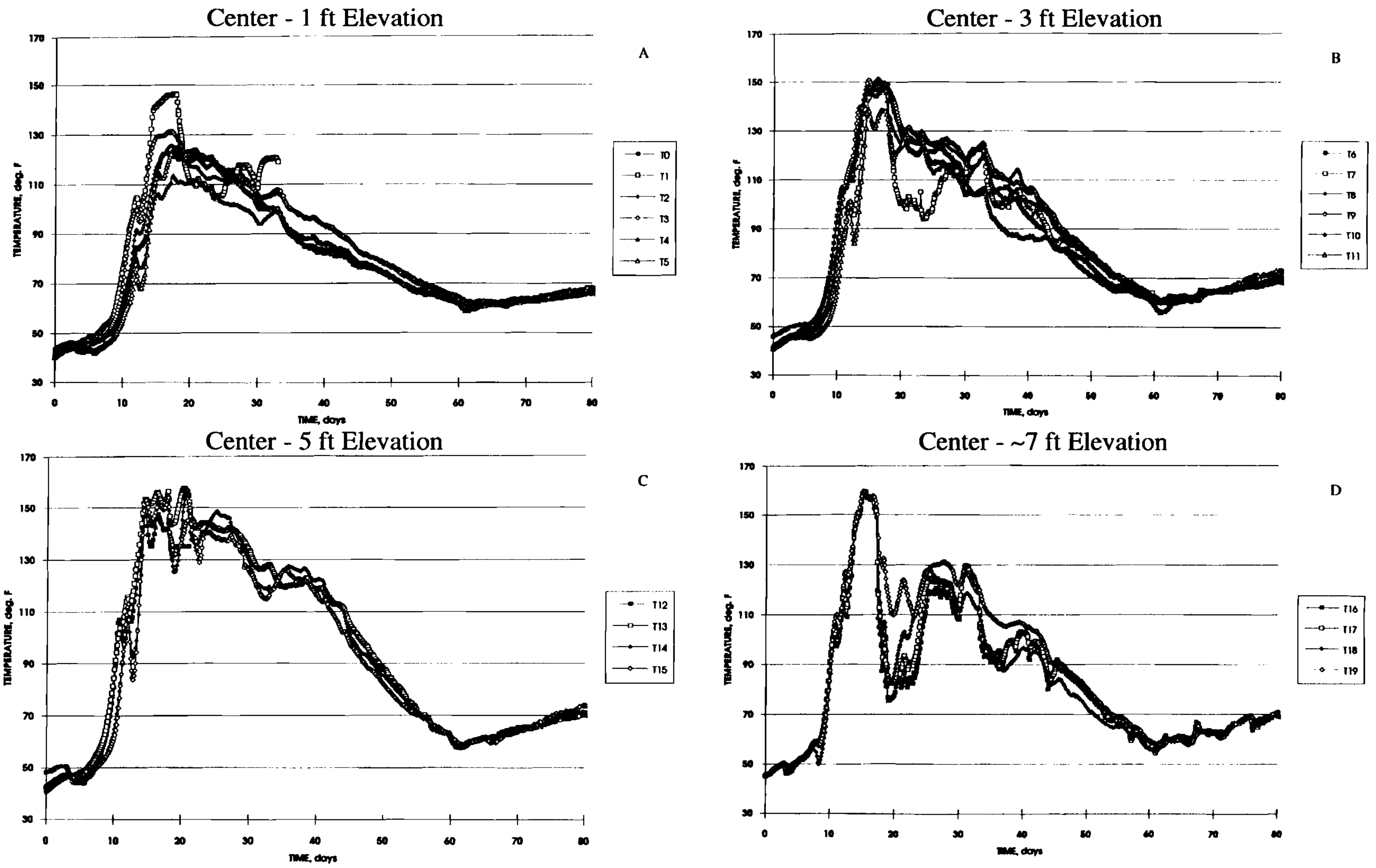


Figure 30. Group of Four Temperature Profiles in Center Cross Section

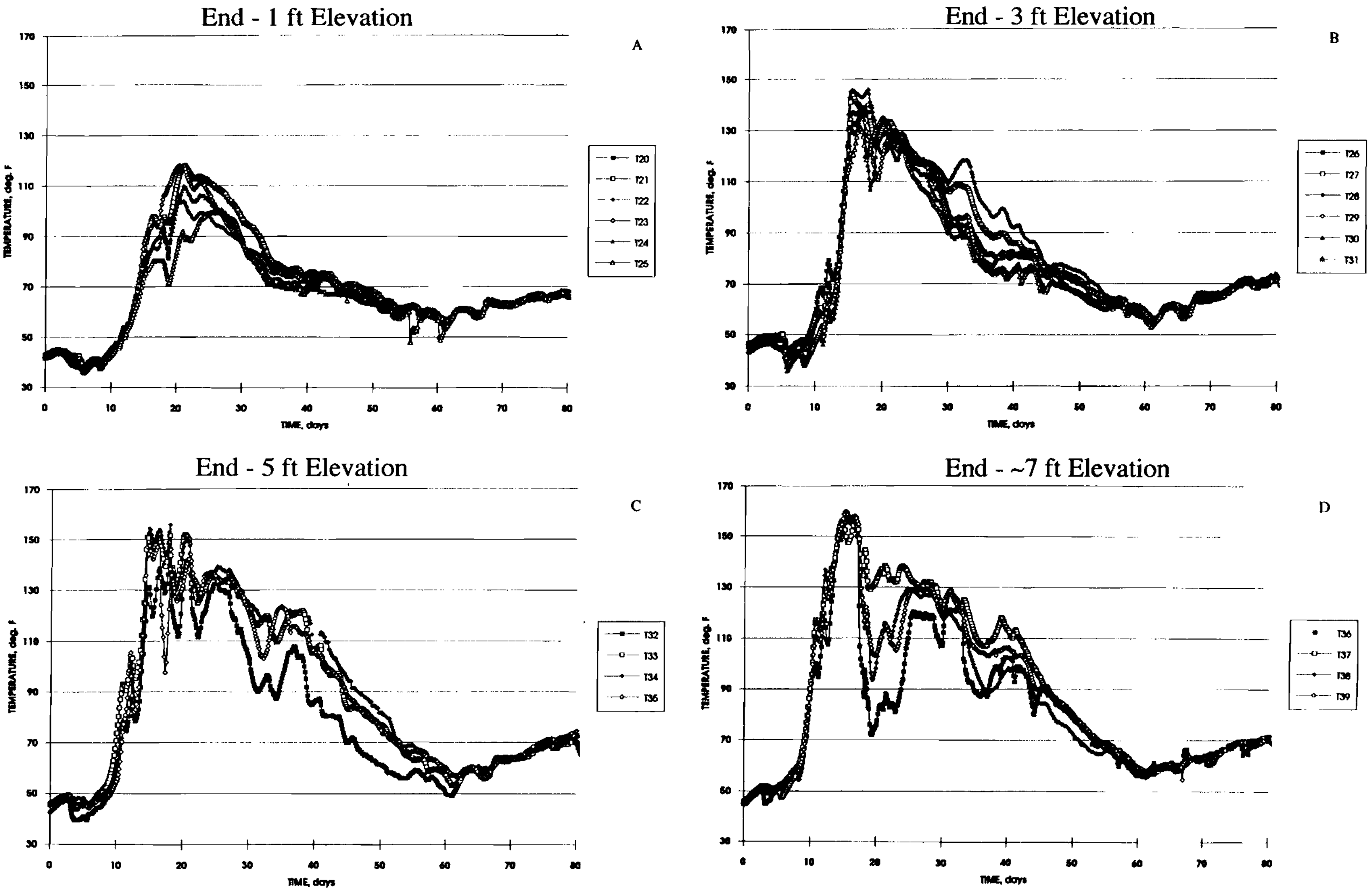


Figure 31. Group of Four Temperature Profiles in End Cross Section

The temperature profiles shown in Figure 23 are consistent with the findings of others for temperature measurements in a forced aeration compost pile. Epstein et al. (1976) measured temperatures at the bottom, center, top, and side of three static piles which consisted of various mixtures of municipal sludge. Temperatures in the centers and tops of their piles were generally higher than in the bottoms and sides. The temperature in the centers of the piles were also more stable than those in the tops of piles.

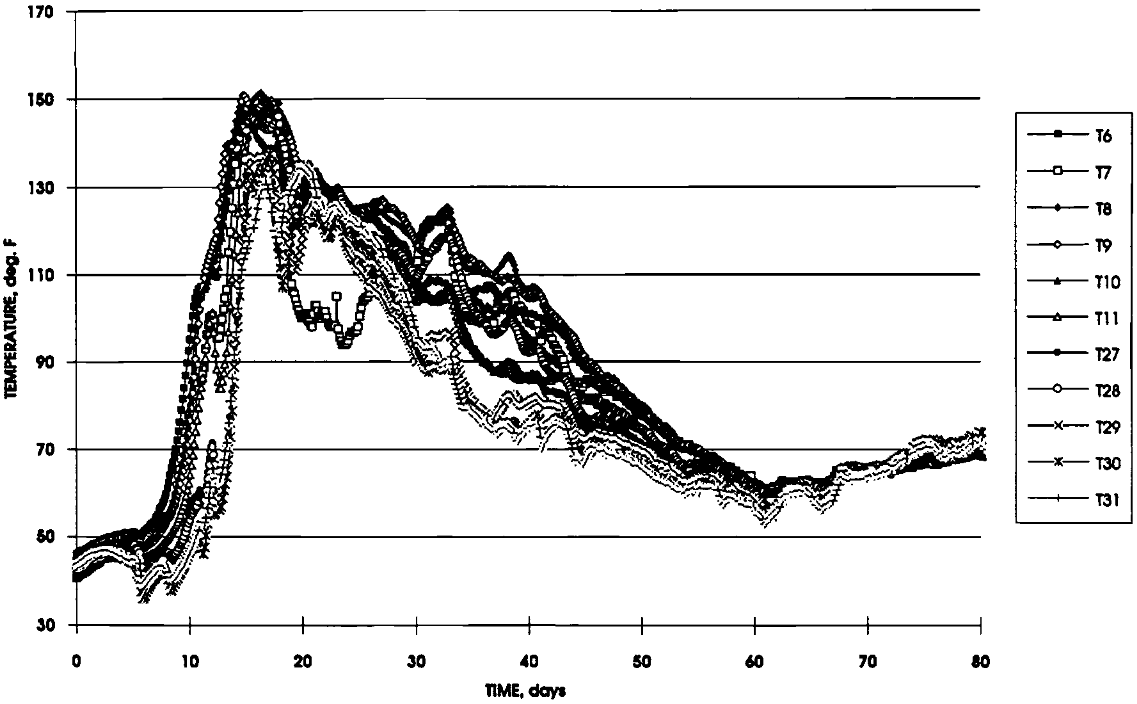


Figure 32. Overlay of All 1 ft Elevation Temperatures Center & End Cross Sections

SP Gas Measurements

Measurements of the concentrations of oxygen (O_2), carbon dioxide (CO_2) and volatile organic hydrocarbons (VOCs) were taken from the tubes attached to the porous filters embedded in the SP (Figure 36 and Figure 37).

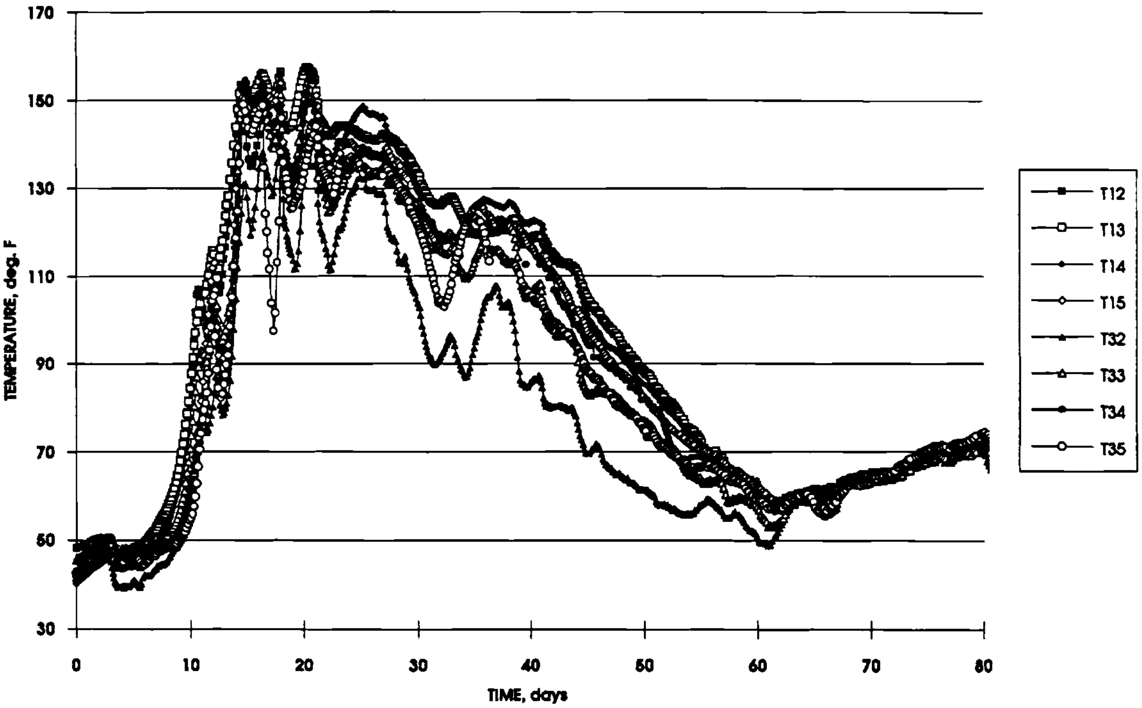


Figure 33. Overlay of All 3 ft Elevation Temperatures

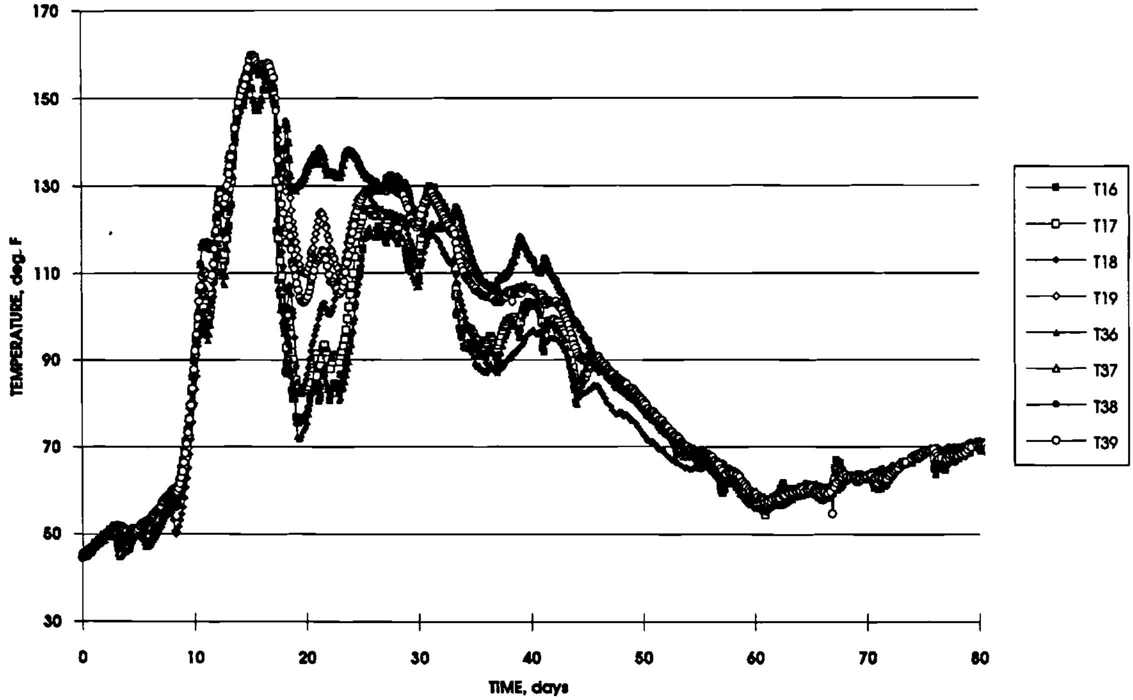


Figure 34. Overlay of All 5 ft Elevation Temperatures

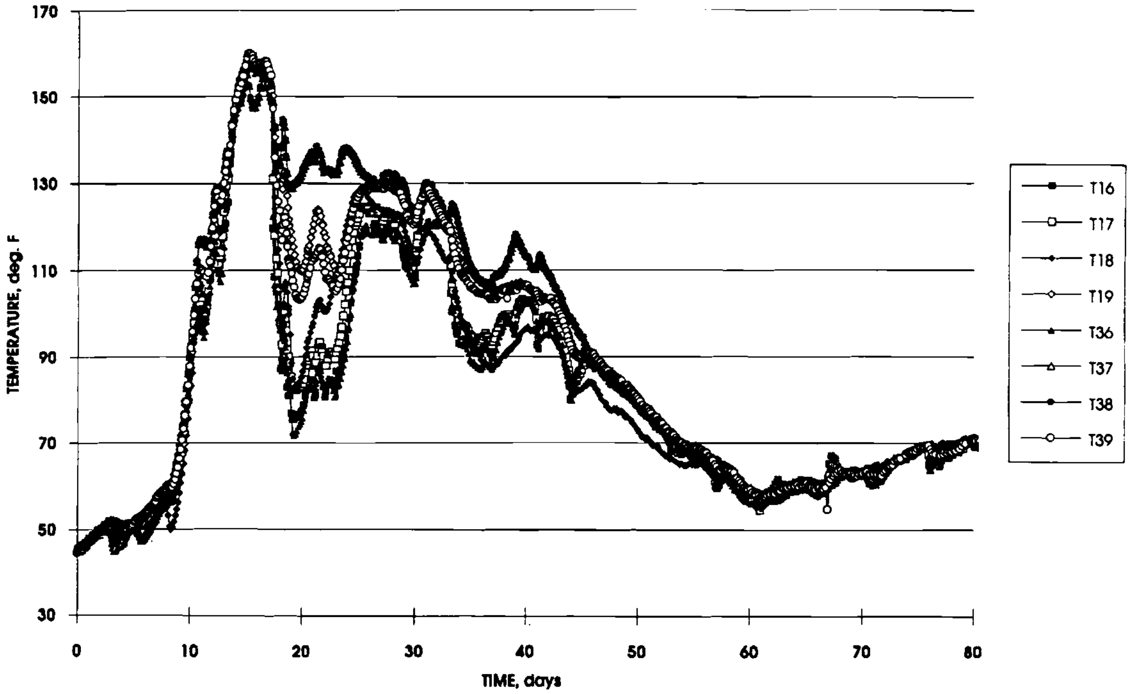


Figure 35. Overlay of All 7 ft Elevation Temperatures

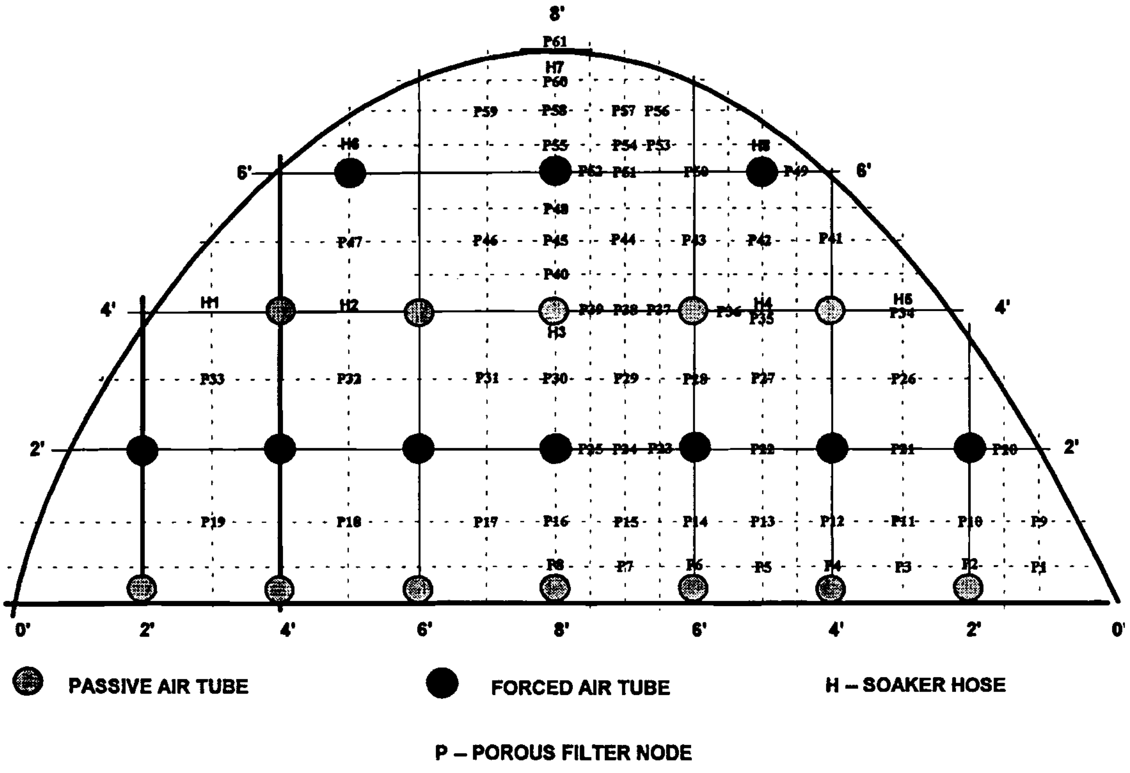


Figure 36. Numbered Locations of Porous Filters in the SP Center Cross Section

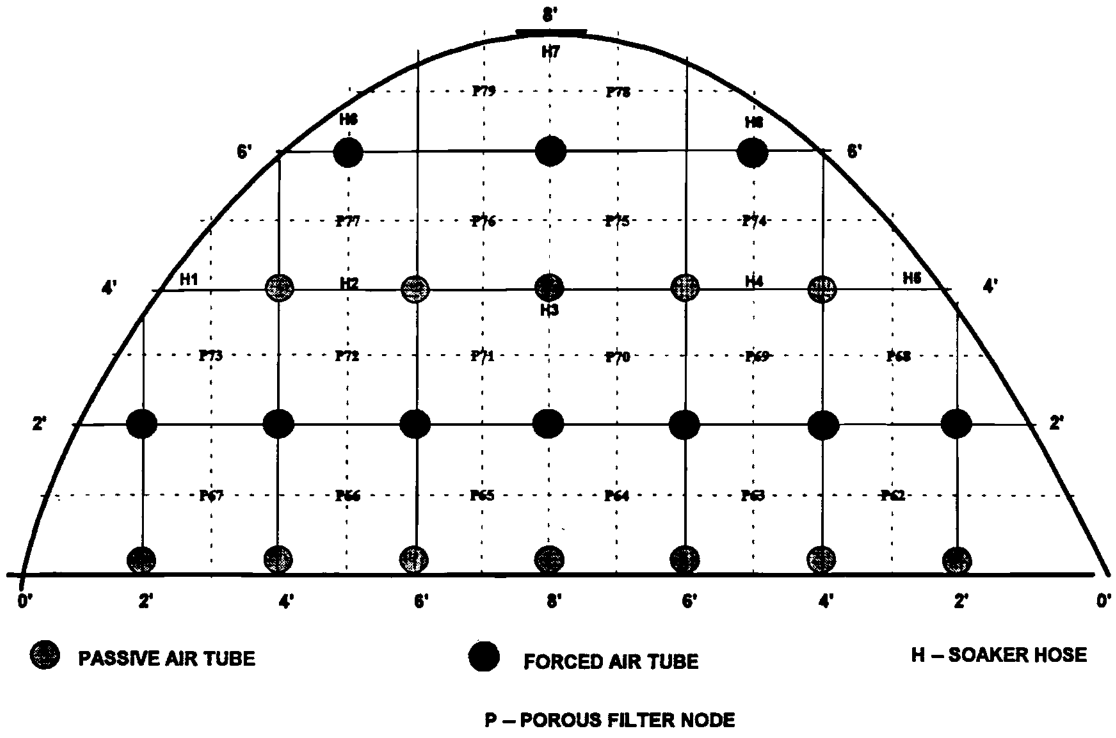


Figure 37. Numbered Locations of Porous Filters in the SP End Cross Section

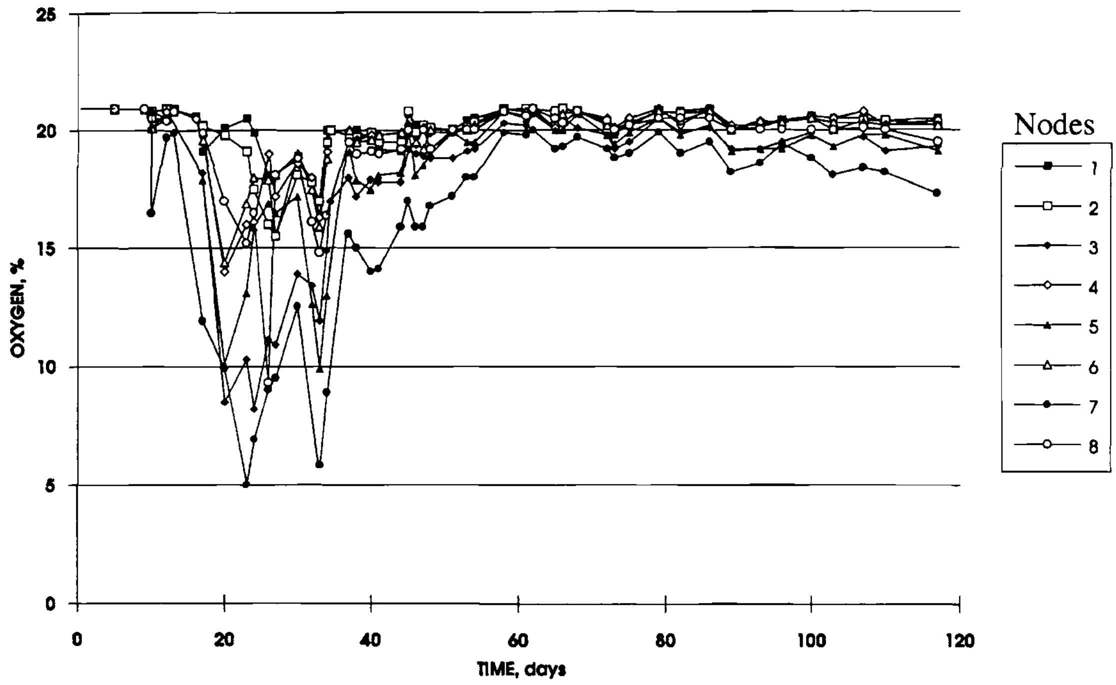


Figure 38. Oxygen Profiles at the 0.5 ft SP Elevation, Center Cross Section

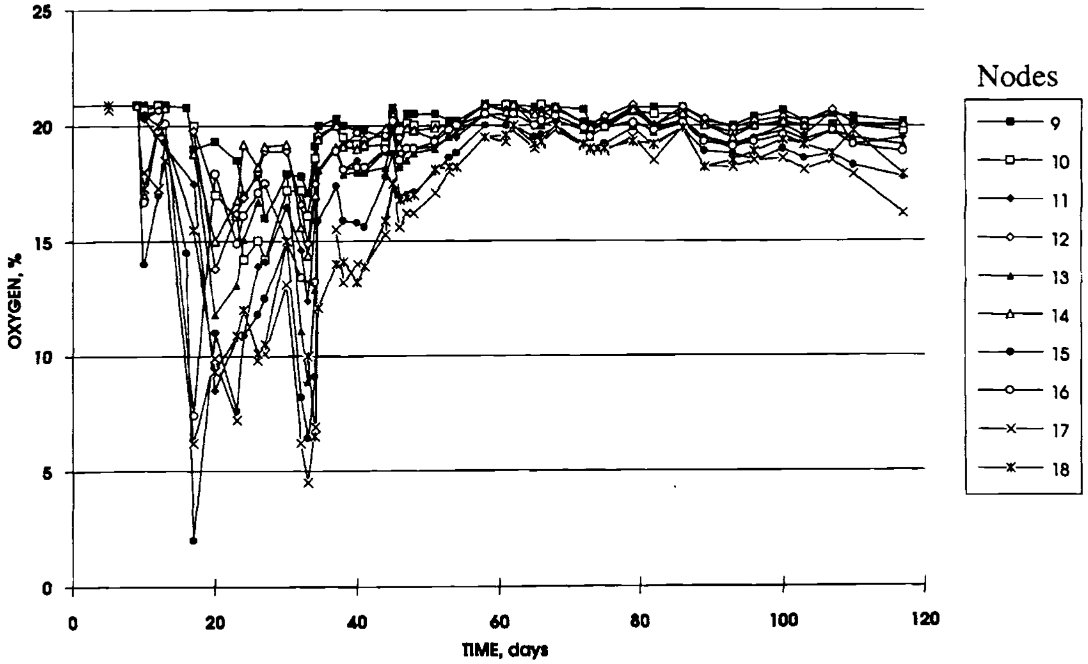


Figure 39. Oxygen Profiles at the 1 ft SP Elevation, Center Cross Section

Oxygen. The oxygen profiles at the gas nodes in the SP during the pilot study are shown in Figure 38 through Figure 46 for the center cross section and Figure 47 through Figure 49 for the end cross section. Two distinct periods of oxygen depression are seen with minimum values on days 18 and 33. The O_2 depression at day 18 occurred during the initial period of high activity as the pile temperatures reached their maximums. This was concurrent with the problems experienced with low air flow from condensation in the air flow tubes. The O_2 depression at day 33 was caused by the sudden halt of forced aeration from the 3 day power outage. Once the forced aeration was halted permanently on day 60, no significant O_2 depression occurred. By this time, core temperatures had converged to about 60 degrees even at a reduced aeration rate. These temperatures and the low O_2 consumption at this time indicate that biological activity was low. On day 68, the passive air tubes were sealed off to prevent passive aeration from ambient air or winds entering the SP. No significant changes were seen in the gas measurements after this event. A number of

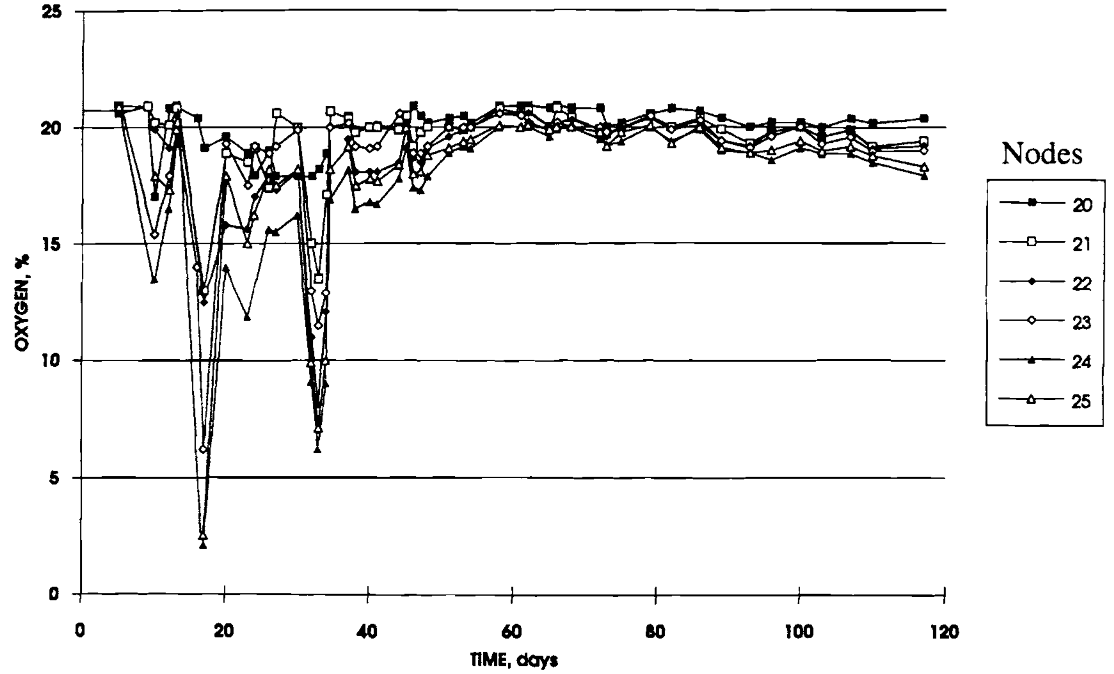


Figure 40. Oxygen Profiles at the 2 ft SP Elevation, Center Cross Section

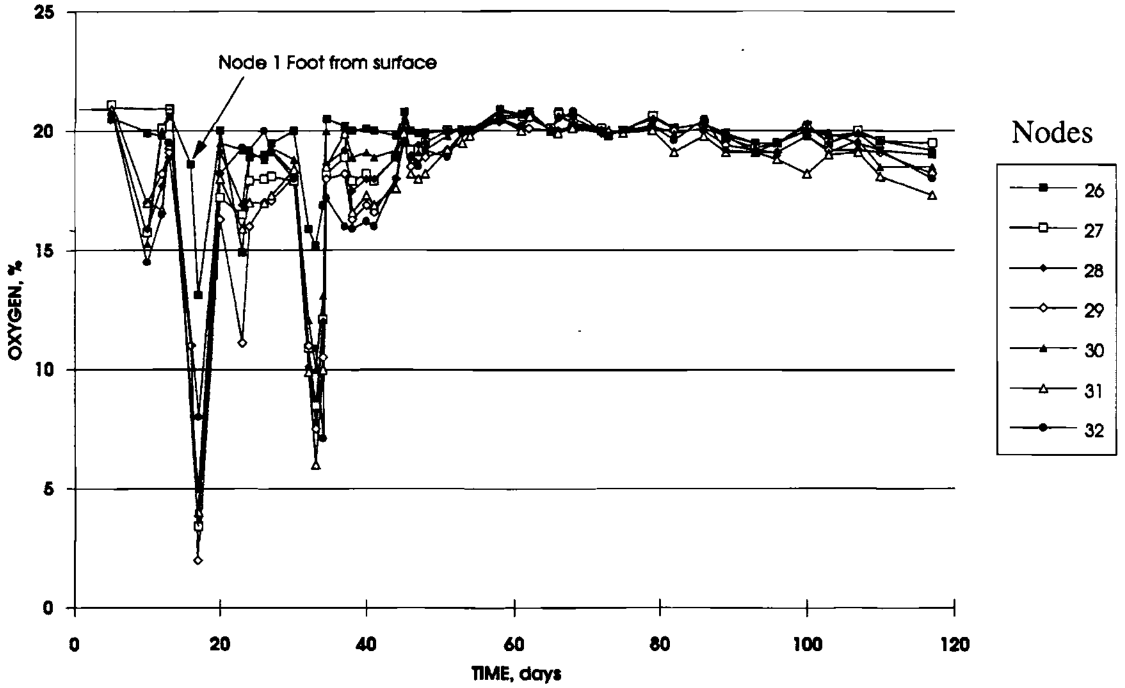


Figure 41. Oxygen Profiles at the 3 ft SP Elevation, Center Cross Section

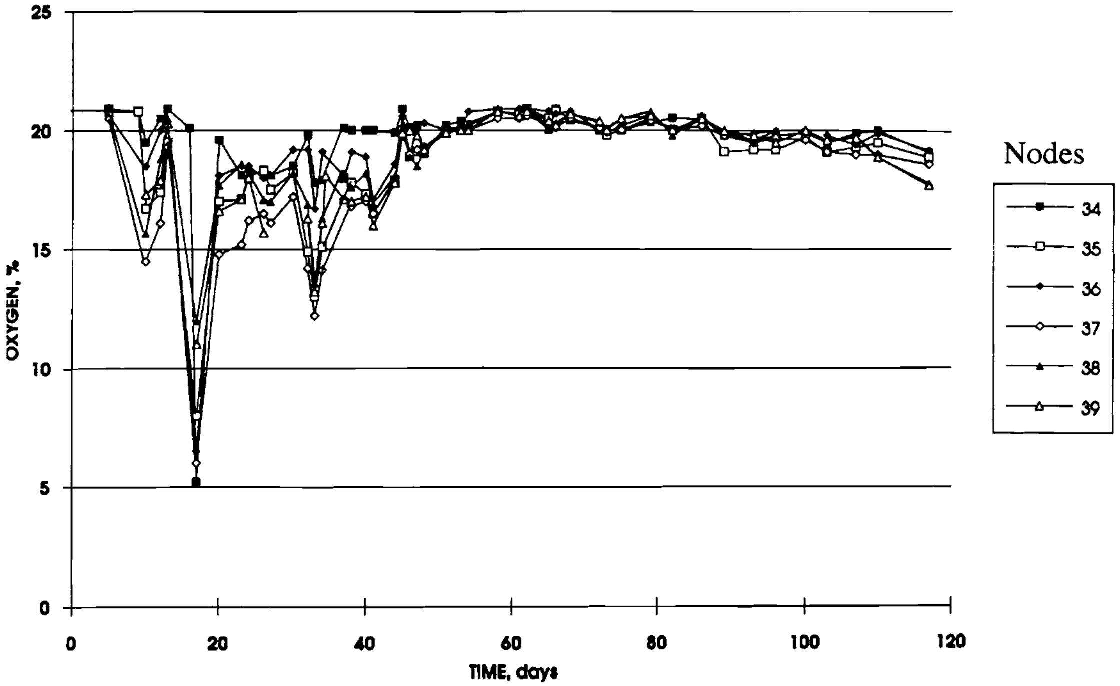


Figure 42. Oxygen Profiles at the 4 ft SP Elevation, Center Cross Section

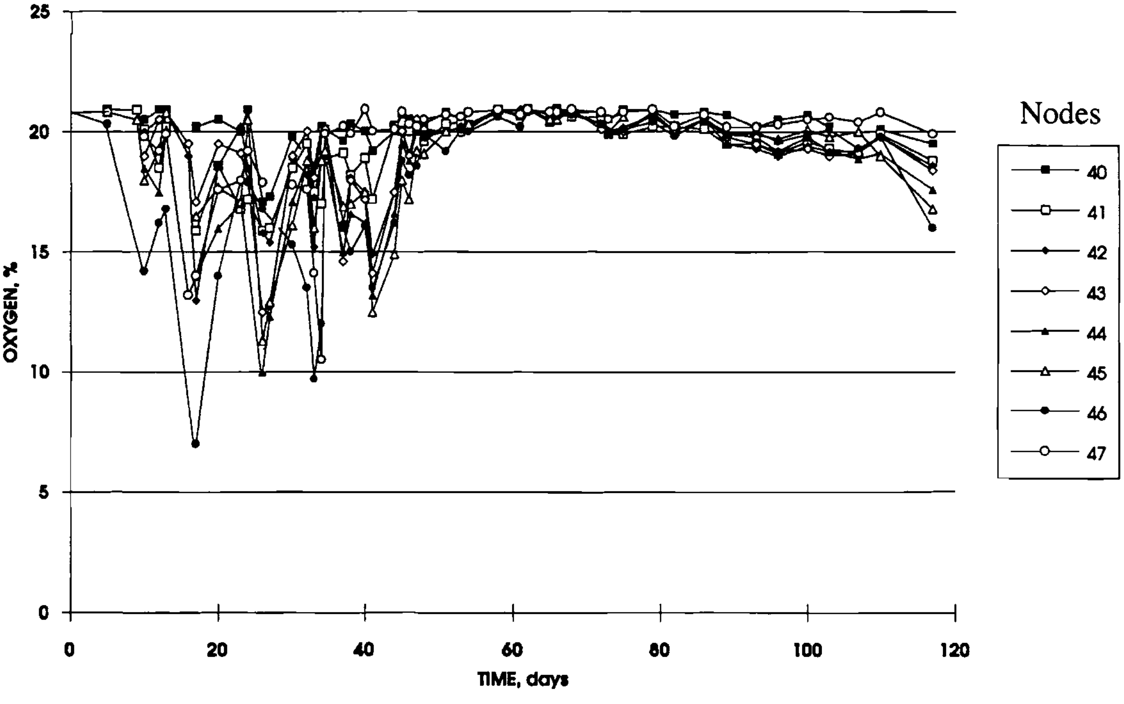


Figure 43. Oxygen Profiles at the 5 ft SP Elevation, Center Cross Section

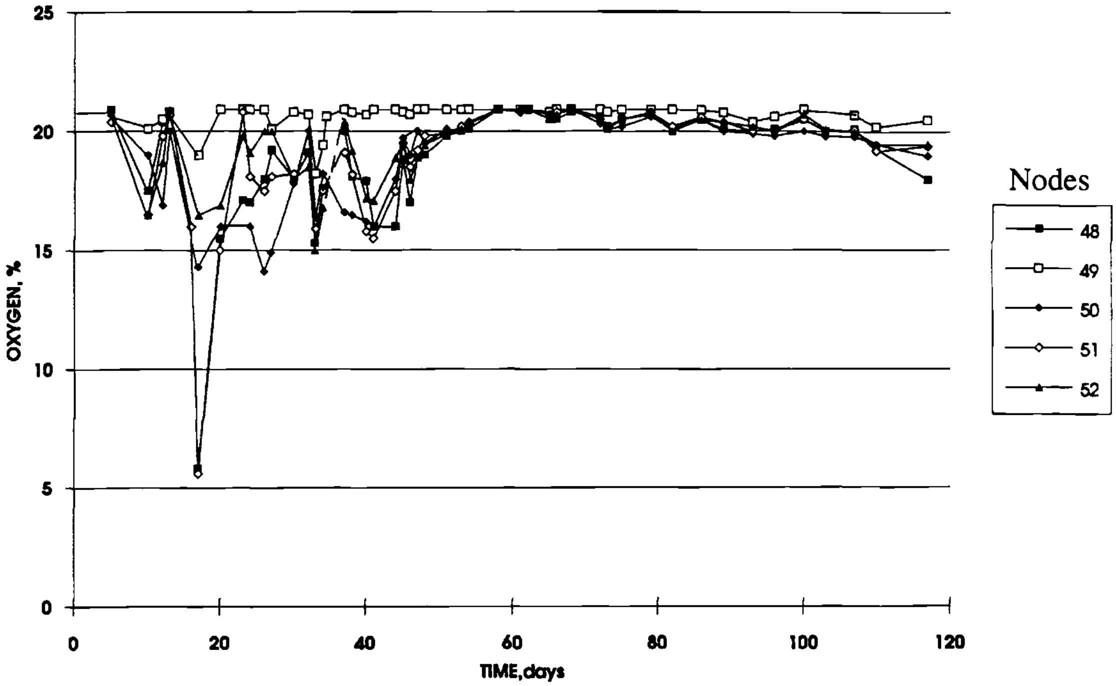


Figure 44. Oxygen Profiles at the 6 ft SP Elevation, Center Cross Section

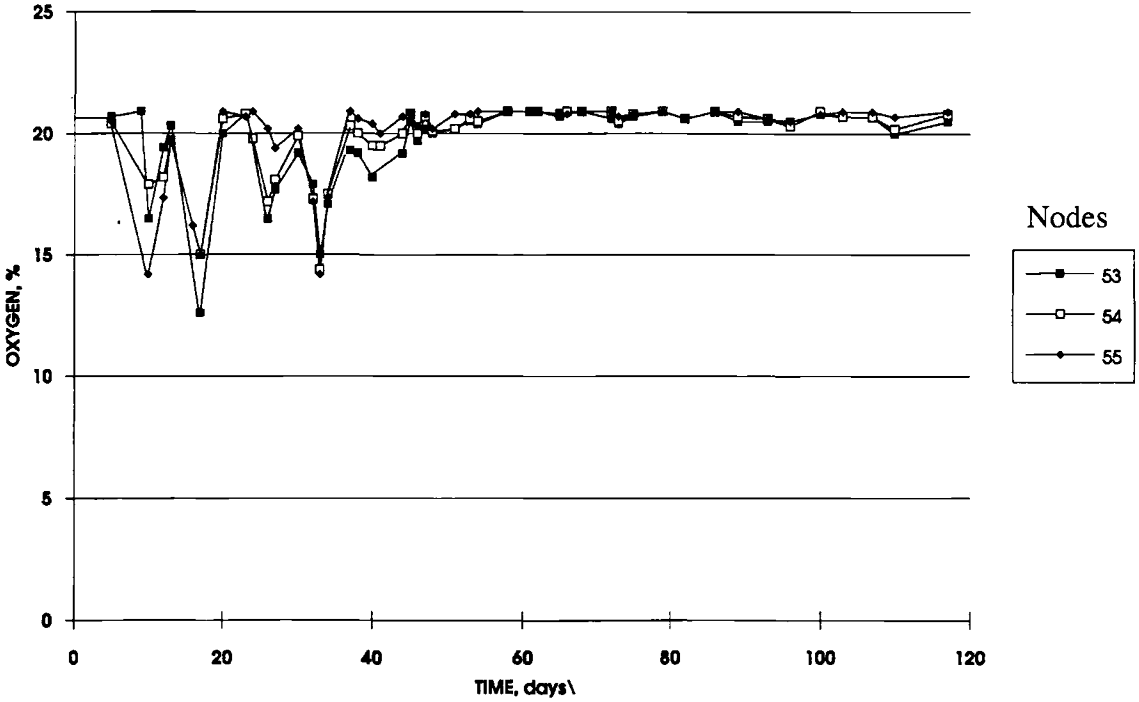


Figure 45. Oxygen Profiles at the 6.5 ft SP Elevation, Center Cross Section

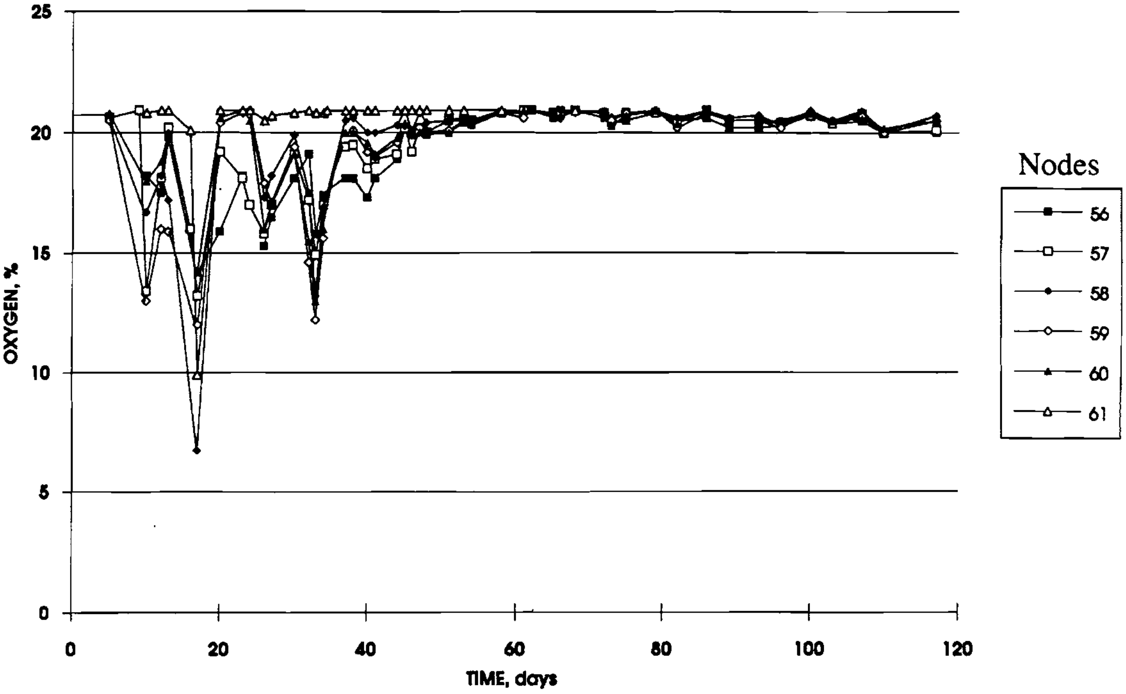


Figure 46. Oxygen Profiles at the 7 ft and Above SP Elevation, Center Cross Section

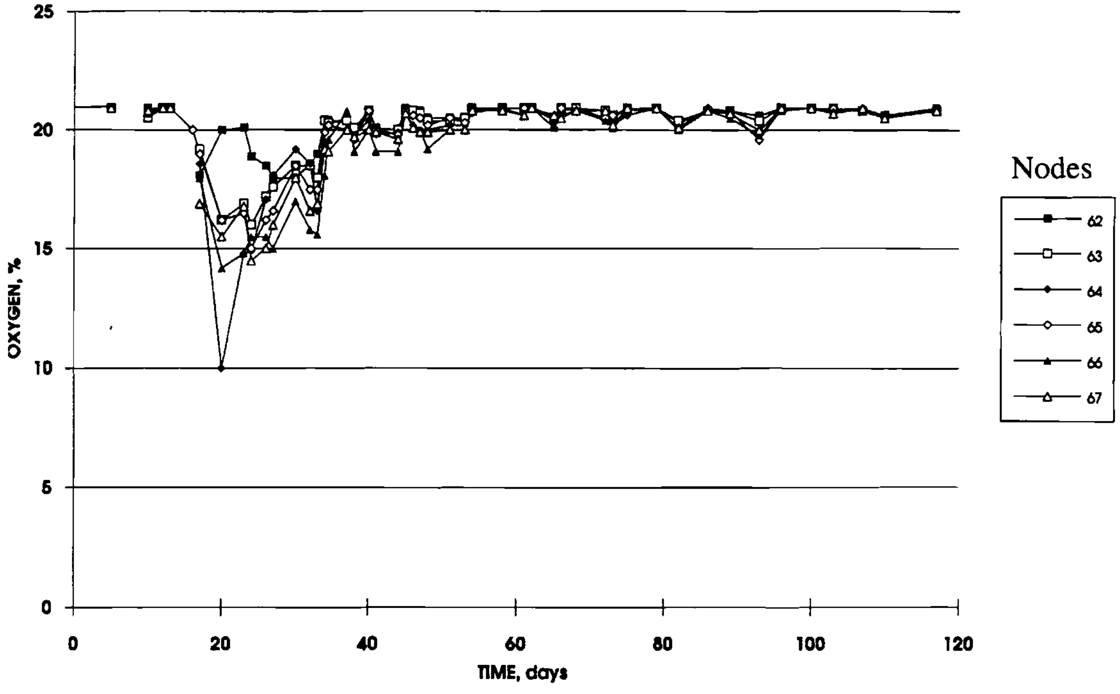


Figure 47. Oxygen Profiles at the 1 ft SP Elevation, End Cross Section

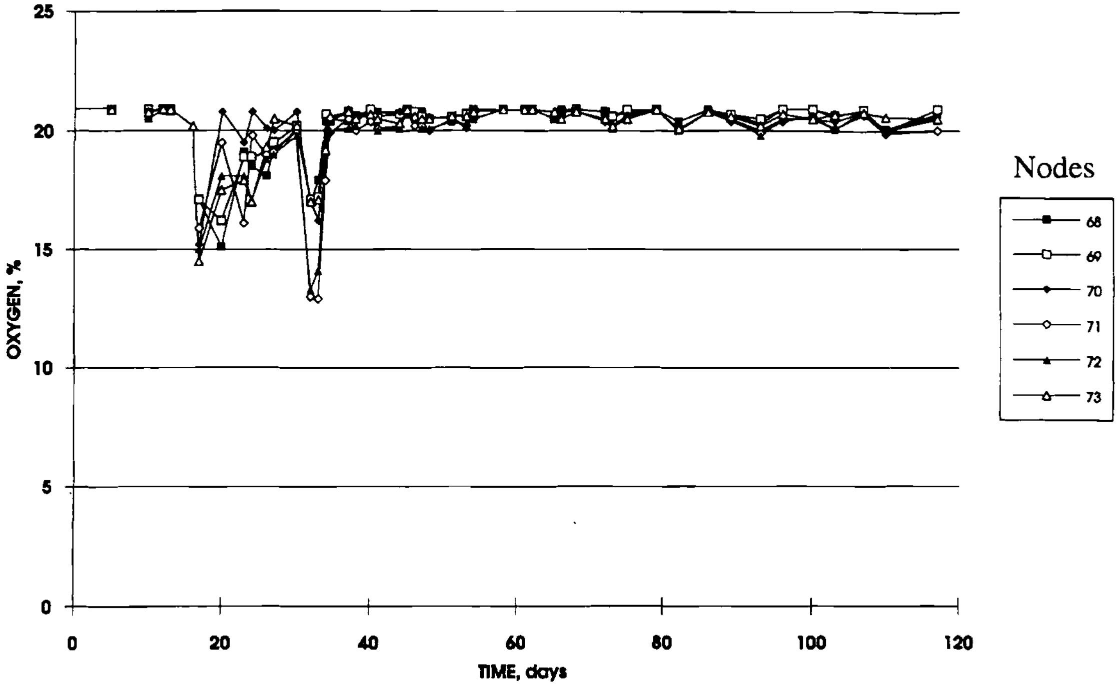


Figure 48. Oxygen Profiles at the 3 ft SP Elevation, End Cross Section

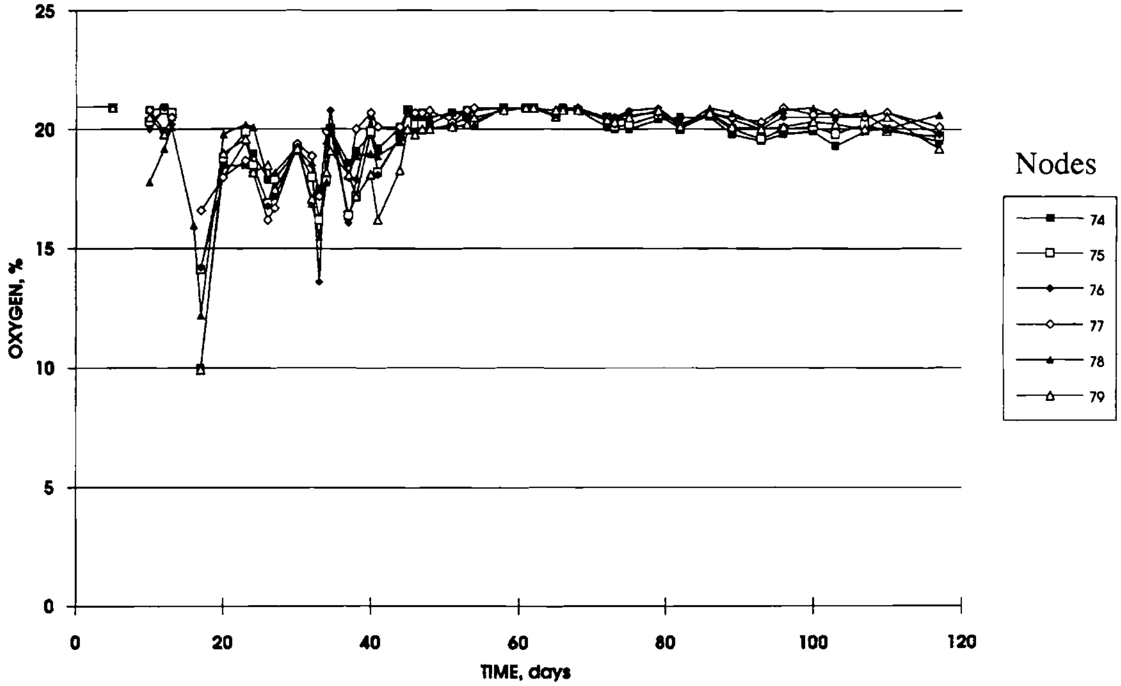


Figure 49. Oxygen Profiles at the 5 and 7 ft SP Elevations, End Cross Section

observations can be made from the O_2 profiles during the period of highest aeration which was from days 19 to 30.

- Most O_2 values were at least 15% throughout the SP during periods of high biological activity and high air flow. This was well above the 5% O_2 threshold (U.S. EPA, 1985) for aerobic respiration.

- There was a general trend for O_2 values to be lower toward the center of the SP with values usually lower at nodes between air tubes.

- At the beginning of the test, O_2 consumption was not as rapid at the 0.5 ft elevation as it was higher in the pile (Figure 38). Similarly, O_2 values remained low at this elevation, less than 10% around day 22, after the O_2 concentrations had recovered at higher elevations. The difference between the O_2 response at the 0.5 ft and higher elevations was likely caused by the combination of 2 factors: (1) a lag in self-heating at the 0.5 ft elevation resulted from the compost being cooled by the concrete surface; (2) compaction was greatest and thus aeration was lowest in this section of the pile.

- Nodes between pairs of air tubes tended to have lower O_2 concentrations than those adjacent to passive air tubes. O_2 concentrations at nodes nearer the centerline of the SP were generally lower than those nearer the side of the SP.

- The O_2 profiles at the 1 ft elevation (Figure 39)were similar to those at the 6 inch level. Some nodes at this level also experienced O_2 depression on or around day 22. The nodes in the midpoints of air tube quadrants, i.e., those at 3 ft., 5 ft., and 7 ft from the edge, had lower O_2 concentrations, 7.5% to 12%, than those positioned directly between passive and forced-air tubes, 14% to 18% O_2. A wider air tube spacing pattern may have resulted in much lower O_2 concentration during this period of high activity at this air turnover rate.

- At the 2 ft level (Figure 40), all nodes maintained O_2 at 15% or above on day 22 except for P24 (7 ft midpoint) which measured 12%. This trend was also evident in P29 at the 3 ft level (Figure 41) which was directly above P24 and in the midpoint of an air tube quadrant. A similar trend occurred directly below P24 in P15 at the 1 ft level. This may have been a local area of higher compaction or higher activity.

- The O_2 concentrations at the 3 ft level (Figure 41) on day 22 were all 15% or greater. A comparison to lower O_2 concentrations and lower activity from lower temperatures at the 1 ft level (Figure 39) suggests that more air was driven up from the forced-air tubes at the 2 ft level (Figure 40). Aeration at the 1 ft level (Figure 39), however, was still effective as seen in the quick recovery in O_2 when the blower was restarted on day 33.

- The O_2 concentrations at the 4 ft level (Figure 42) were all above 15% on day 22 indicating good airflow in the horizontal plane. Less O_2 depression on day 33 than the 3 ft (Figure 41) and 5 ft (Figure 43) elevations suggests that passive aeration occurred during blower shutdown in the vicinity of the passive aeration tubes.

- At the 5 ft elevation (Figure 43), the center region, 6 to 8 ft from the side, had the highest temperatures in the SP and the lowest O_2 values, 10-12.5%, at the 5 ft level. This level was below the level with the forced-air tubes at 6 ft (Figure 44). Although oxygen was sufficient, cooling was not. As in the case of the 1 ft level and a comparison of the temperature and oxygen values at 5 ft and 7 ft (Figure 46), more of the air from the 6 ft aeration tubes appears to have blown upward.

- The O_2 values were high in the nodes at 6 ft and above (Figure 44, Figure 45, and Figure 46). Temperature and oxygen values on day 33 indicated that high biological activity was still occurring at this time. The higher O_2 resulted from less compaction and greater airflow in this region than in the region below.

- Figure 47, Figure 48, and Figure 49 concur with the temperature data that biological activity occurred near the surface of the SP. Although these figures may not seem as profound as the oxygen profiles for the center cross section, the nodes in the end cross section were influenced by air diffusion into the pile and somewhat lower biological activity as previously demonstrated in the temperature profiles. Nevertheless, quick recovery of O_2 upon restarting the blower on day 33 is evident. Such quick response shows that aeration was also effective along the ends of the SP.

- Figure 50 isolates the O_2 response at the 1 ft elevation for the 20 to 40 day time period to show the effects of the blower shutdown on day 30 and its restart on day 34. There was almost complete recovery to stabilized O_2 values within 3 hours after forced aeration was restarted. All elevations above 1 ft had equally rapid O_2 recovery.

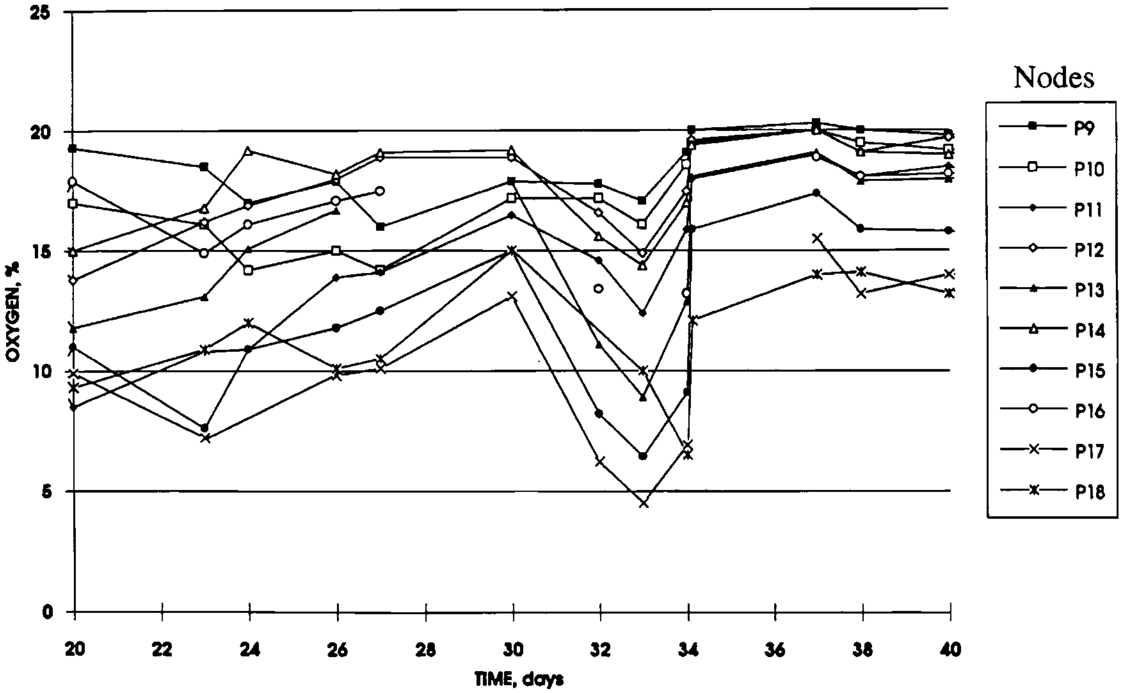


Figure 50. Close-Up of O_2 Response at the 1 ft Elevation from 20 to 40 Day Time

O_2/CO_2 Correlation. CO_2 response during the pilot study was almost a complete inverse of the O_2 response. Figure 51 shows the CO_2 profile for the 3 ft elevation. It can be compared to the O_2 profile at the same elevation in Figure 41. Appendix E contains CO_2 profiles for all of the elevations in the SP. From the O_2 and CO_2 profiles, the minimum O_2 and the maximum CO_2 values at each elevation on day 33 were selected and tabulated. A simple correlation was developed in this study, whereby oxygen was divided by its maximum normal concentration (MNC_{O_2}), carbon dioxide was divided by its maximum aerobic concentration (MAC_{CO_2}), and the two fractions were summed. No similar correlation has been found by the author in the literature. Equation 11 may be used to describe the correlation:

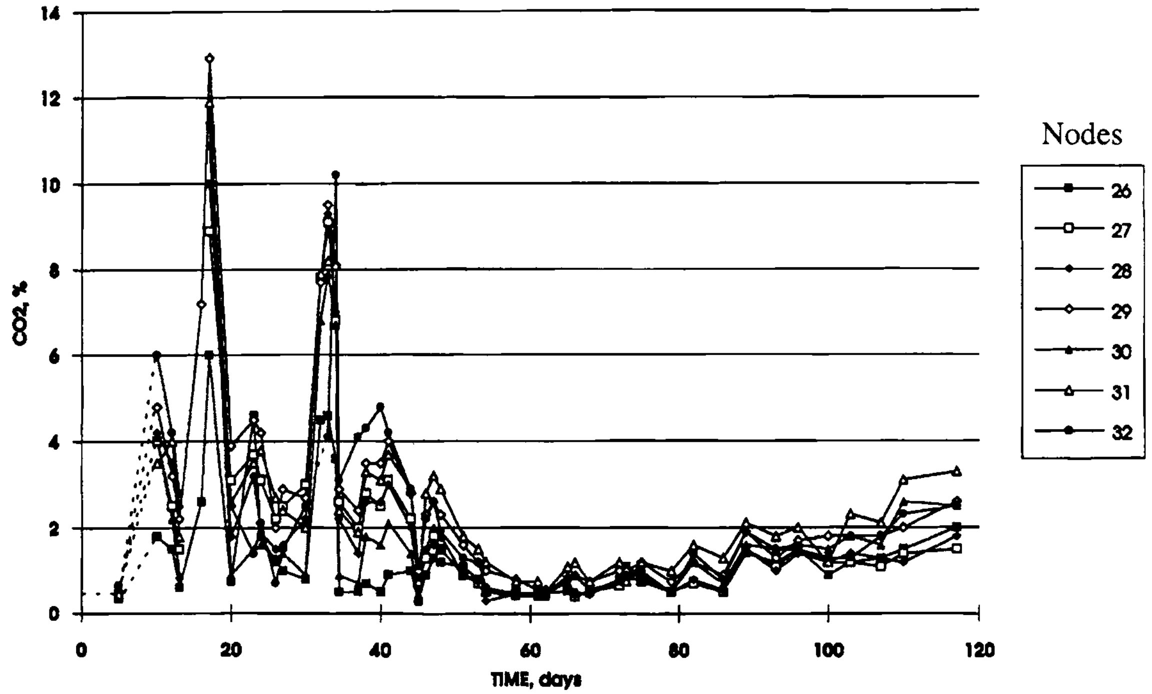


Figure 51. CO_2 Profile at the 3 ft Elevation

$$\frac{[O_2]}{MNC_{O_2}} + \frac{[CO_2]}{MAC_{CO_2}} = 1 \tag{11}$$

where the concentrations of O_2 and CO_2 are expressed as decimal percentages. When using the (MNC_{O_2}) as 20.9%, which is the concentration of O_2 in the atmosphere, and a somewhat arbitrary (MAC_{CO_2}) of 15.0%, the correlation fit the data in Table XVI to within 5% of unity. It appears that under these conditions, the correlation may be used to estimate the concentration of one gas if the other is known.

TABLE XVI

CORRELATION OF O_2 AND CO_2 IN SP GASES

Elevation	O_2,%	CO_2,%	O_2 as % of MNC	CO_2 as % of MNC	Sum
0.5 ft	6.00	10.00	29.00	67.00	0.96
1 ft.	4.50	11.00	21.50	73.00	0.95
2 ft.	6.00	10.00	29.00	67.00	0.96
3 ft.	6.00	10.00	29.00	67.00	0.96
4 ft.	12.50	7.00	60.00	47.00	1.07
5 ft.	10.00	8.00	48.00	53.00	1.01
6 ft.	15.00	5.00	72.00	33.00	1.05
6.5 ft	14.00	5.00	67.00	33.00	1.00
7 ft +	12.00	6.50	57.00	43.00	1.00
1 ft (end)	15.50	4.50	74.00	30.00	1.04
3 ft (end)	13.00	6.50	62.00	43.00	1.05
5 and 7 ft (end)	13.00	6.00	62.00	40.00	1.02

However, the proper value for MAC_{CO_2} has not been statistically determined. The equation can be rearranged and rewritten as shown in Equation 12 to solve for MAC_{CO_2}.

$$MAC_{CO_2} = \frac{[CO_2]}{1 - \frac{[O_2]}{MNC_{O_2}}} \qquad (12)$$

Simplified, the equation that results is,

$$MAC_{CO_2} = \frac{MNC_{O_2} \bullet [CO_2]}{MNC_{O_2} - [O_2]} \qquad (13)$$

Using the measured gas values in Table XVI, the mean value for MAC_{CO_2} equals 15.6 with a standard deviation of 1.4. After using the value of 15.6 instead of the arbitrary value of 15.0 for MAC_{CO_2}, the identity sums ranged from 0.93 to 1.05, with a mean value of 0.986 and a standard deviation of 0.047. A large set of measured oxygen and carbon dioxide values could be used in a rigorous exercise to statistically establish a proper literature value for the MAC_{CO_2}. The MAC_{CO_2} value could then be used in one of two ways: (1) measured oxygen and carbon dioxide values could be substituted into Equation 13 to calculate MAC_{CO_2} as a check against the established literature value. If the calculated MAC_{CO_2} value is significantly below the literature value, then one of the measured values may be incorrect, the system may not be operating under aerobic conditions, or there may be significant consumption of oxygen by soil minerals. Oxygen values less than 5% may indicate a shift from aerobic to anoxic conditions and reduce this coefficient. Incomplete mineralization of substrate or a high rate of conversion to biomass will also affect the coefficient to some degree. Additional research may be necessary to determine the sensitivity of these factors on the MAC_{CO_2} coefficient. (2) Equation 11 can also be used to estimate one of the two gases after measuring the value of the other. Using this method, the investigator would have to be aware of operating conditions and the previously mentioned factors.

VOCs. Profiles of volatile organic hydrocarbon (VOC) emissions measurements are included in Appendix F. All but a few data points remained low, less than 140 ppm as hexane, throughout the study, even during periods of highest activity. Once again, as in the case of the other gases, during periods of low or no aeration, a spike occurred in the gas measurements. However, the VOC concentrations did not rise into the hundreds or thousands of ppm. This implies that either the biodegradation rate of larger HCs was slow or that mineralization was effective and produced few low HC weight alkanes in the process.

Observations During Operation of the SP

Several observations were made during the operation of the SP. During the period of highest microbial activity and temperatures, the SP emitted a strong ammonia odor characteristic of cattle manure. This lasted for about 2 weeks. During this time period, water vapor could be seen quite readily in about the top 1/3 of the pile. When the ambient temperature was well below freezing, this vapor condensed on the surface of the pile and formed ice crystals in some places. When the cover was removed permanently on day 17, a thick white fungus was observed on the surface of the pile in many places. Quite a number of wheat seedlings germinated around day 20 on the surface of the pile. This suggests that the sludge may not have been inhibitive to normal plant growth. The pile settled about 2 ft in height during the first 24 days. Thereafter, it settled approximately 6 more inches.

Windrow Operation

Temperature Effects and Pile Turning

Initial Self-heating Period. The WR began to self-heat from day 10 to about day 20 as shown in Figure 52. The start of this period was about the same as that of the static pile shown in Figure 24. It is encouraging to note that this occurred when ambient temperatures oscillated around freezing and had a low of 9°F on day 4 (Figure 22). The self-heating process was interrupted by the second turning event on day 20 and was greatly impacted by a cold weather front with temperatures below freezing. The WR was also watered during this event. Turning and watering reduced the average pile temperature by 40 degrees between day 20 and the next monitoring event on day 22. The WR quickly recovered as pile temperatures again increased sharply up to day 33.

Temperature Effects from Turning. Out of the 7 turning events during the test, only 3 produced significant temperature reductions in the WR. They were essentially caused by cold weather fronts that occurred at the same time as these 3 events (days 20, 33, and 60). The temperature drop in the pile between days 33 and 34 was about 40 degrees as had previously occurred on days 20 to 22. The temperature drop on days 60 and 61 was more subtle than these two events, but can be substantiated by the cold ambient tempera-

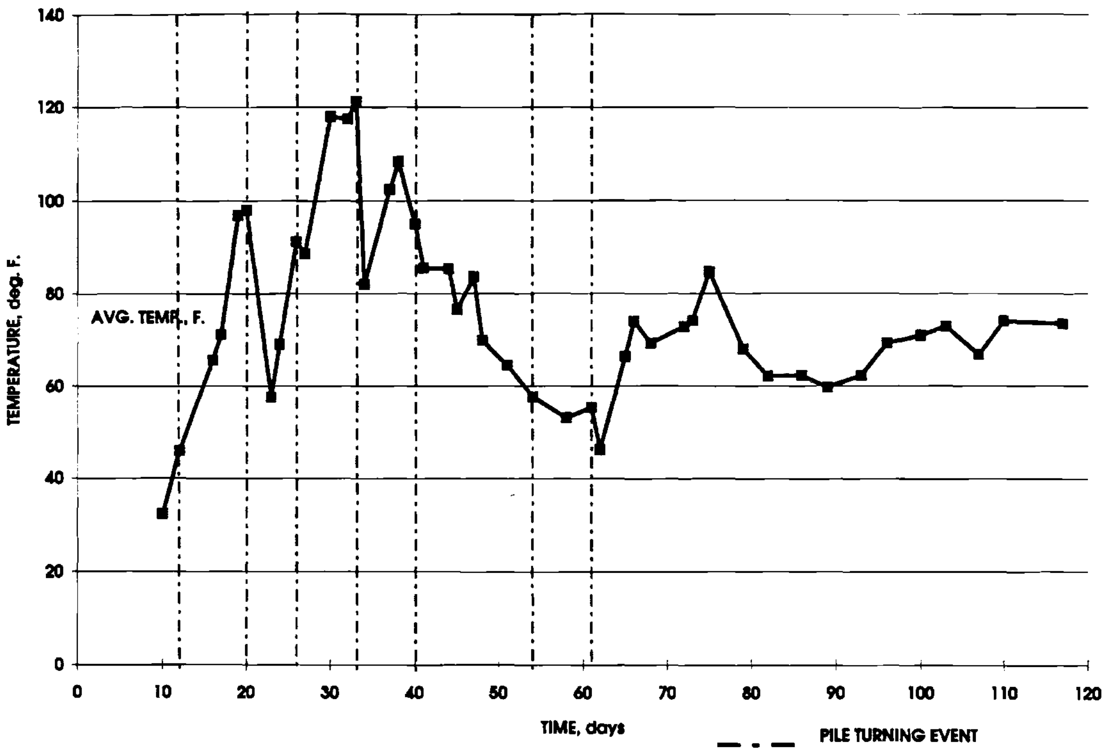


Figure 52. Temperature Profile of the WR Pile

tures shown in Figure 25. The other 4 events appear to have been unaffected by the milder ambient temperatures during pile turning.

Overall Temperature Profile. The average temperature in the WR was 120°F, which slightly exceeded the upper boundary of the mesophilic range. This temperature was sustained for about 2 days before being lowered by the fourth turning event. By comparison with the temperature profile of the SP shown in Figure 24, the duration of the active period of composting in the WR was about the same; self-heating began on about day 10 and the active period concluded about day 61. Thereafter, the WR and the SP temperatures tracked ambient conditions. Comparatively, the temperature rise of the WR during self-heating did lag that of the SP for two reasons. First of all, when the tests were initiated, the WR pile temperature was near freezing while the SP temperature was at least 10 degrees higher. Secondly, since construction of the SP took six days to complete, its starting time was arbitrarily set at the end of construction. This afforded it some degree of temperature maintenance or increase above freezing. The WR took roughly twice as long as the SP to achieve peak temperatures because of these factors and because of the interruptions caused by pile turning. Additionally, the SP had a larger thermal mass than the WR and therefore retained its pile temperature better than the WR did. It may be easily reasoned that the WR would have achieved much higher peak temperatures if the turning events had not occurred. Furthermore, the pile temperature may be easily controlled by turning during cold weather.

Gas Measurement Data

Profiles of the average gas concentrations are shown in Figure 53. As in the static pile, oxygen and carbon dioxide concentrations were inversely related. During the period of highest activity, days 26 to 33, the lowest O_2 and the highest CO_2 and VOC concentrations were measured. The average O_2 concentration remained above 15% for the entire test which indicated that pile manipulation was very effective in supplying adequate oxygen for aerobic respiration. The VOC concentrations remained under 100 ppm during the test. As in the case of pile temperatures for the WR, all of the gas concentration changes (peaks) were less than those in the SP. This was due to the manipulation of the WR directly aerating the pile as well as reducing the effects of compaction. Upon turning, the WR compost tended to form small clods of various sizes, the largest being about the size of a human fist. This pile structure allowed greater passive diffusion of gases through the pile than in the more compacted static pile.

Observations During Operation of the WR

Because of the concurrent operation of the SP, it was difficult to discern the extent of odor produced by the WR. Casual observations were that less odor was noticed in the vicinity of the WR than in the vicinity of the SP, which had a definite odor during peak activity. Much less water vapor was observed from the WR than from the SP. Most of it was produced on the eastern end of the WR pile. This end of the pile may have benefited

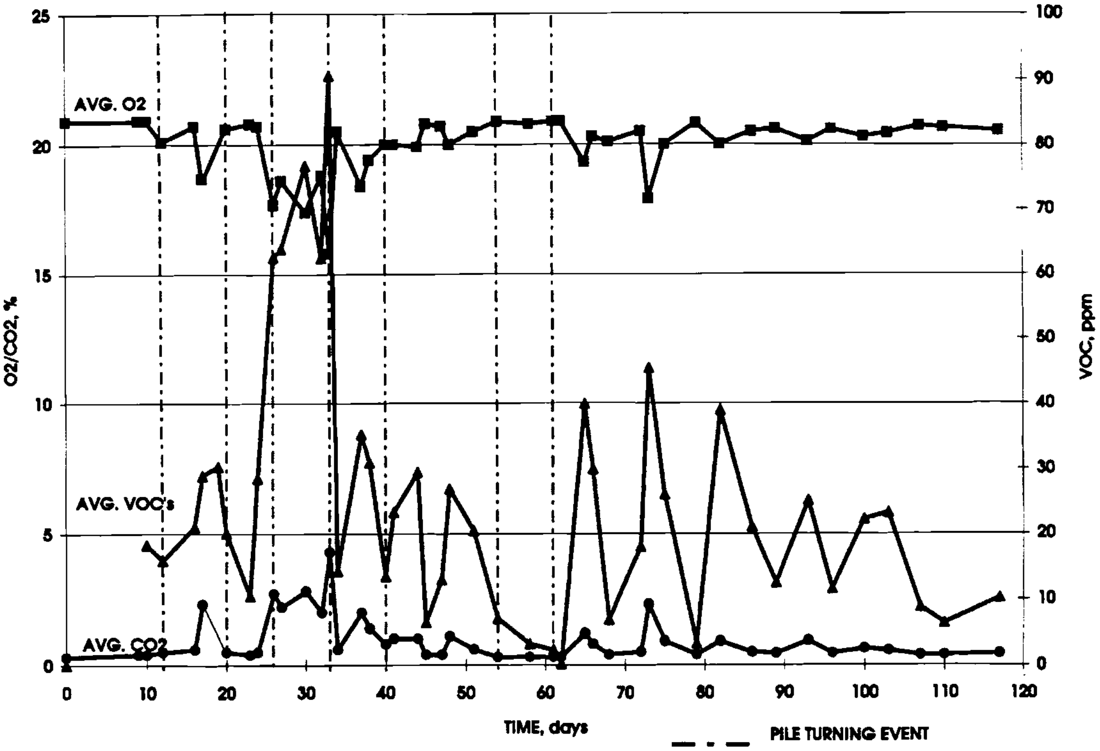


Figure 53. Gas Profiles of the WR Pile

from the morning sun which probably assisted the self-heating process. Water vapor was very noticeable on day 33 when the pile had reached its maximum temperature and ambient temperatures were in the 30°s. From about the middle to the end of the test, the color of the compost of the WR remained darker than that of the SP. These differences are consistent with observations of others (Golueke, 1977 cited in Haug, 1993). Actinomycetes have been discovered to form large colonies consisting of blue-gray or light green powder or filaments in undisturbed compost piles. If the compost is mixed mechanically, as in the case of the windrow, such large colonies do not form. Figure 54 shows a comparison of excavated samples of each compost material. Although color differences were observed in the two materials, they do not appear to be related to O&G levels. The straw in the WR seemed to have decomposed less than the straw in the SP. As mentioned previously, the WR compost had a less compacted structure and contained numerous small clods formed by frequent pile manipulation. These clods likely facilitated gas diffusion through the pile.

Unlike the SP, no plant seedlings were observed in the WR. The frequent turning probably destroyed any that germinated. Finally, although no discrete measurements were made, the WR compost pile size appeared to have diminished compared to its original size. However, its comparative reduction seemed to be less than that of the SP. This was probably due to the manipulation of the WR.

Figure 54. Photo of Excavated Samples Showing the Color Contrast Between the WR Sample (Left) and the SP Sample (Right)

Biodegradation

Numerous analyses were conducted on the compost samples collected from the SP and WR piles to measure biodegradation of the target HCs.

Oil and Grease Reduction

The profiles of the reduction of O&G from the original concentration of 3.47% dry wt for the two compost piles are shown in Figure 55. The curves show a more substantial decrease in the O&G concentration in the WR samples through the end of the 121 day test period than in the SP samples. The dotted lines in the figure represent linear regressions of each series of data. The correlation coefficients were -0.8422 for the WR data and -0.2501 for the SP data. It is uncertain why the O&G analyses for the SP samples had such a large variation in the data which resulted in its poor correlation coefficient. The standard deviations for the group of analyses for individual sampling events were usually less than 10% of the average O&G value. Appendix G lists the extraction data of all samples plus the averages and standard deviations for both the SP and WR piles. Samples marked with asterisks and bold type were considered outliers and were not included in the average O&G calculations or standard deviations. The order of sample preparation and extraction was random. If the sample preparation and extraction had been the reason for the variability in the data, then the same variability would have been present in the WR data.

Using the regressions, the reduction of O&G from the original concentration at the end of 121 days was 20.2% and 8.4% for the WR and SP respectively. This results in an O&G half-life of 352 days for the WR and more than 4 years for the SP.

The O&G contributed by the straw and the soil itself was small enough that the extractions were not corrected for the background O&G concentration. The O&G content of straw measured 0.98% dry wt and the O&G content of background soil measured at 0.009% dry wt. Since the straw comprised only 3% of the dry weight of the compost, the O&G of the straw only accounted for 0.03% of the O&G in the original compost mixture. Together the straw and the background soil only contributed 0.04% O&G in the original compost mixture.

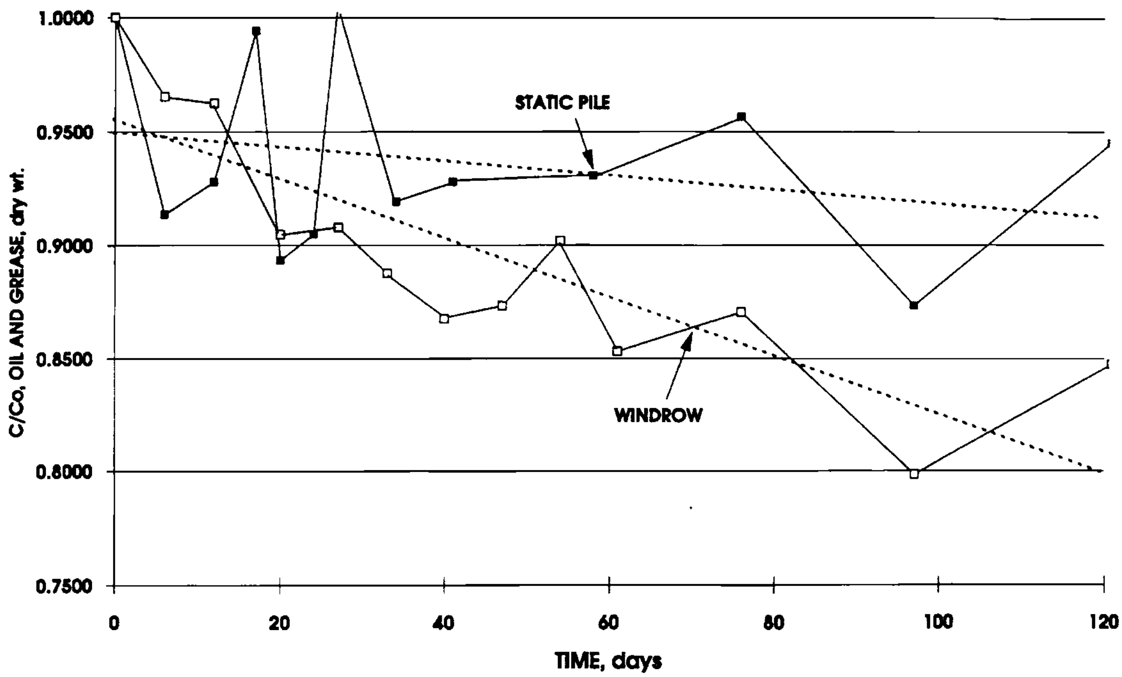


Figure 55. Total Reduction of O&G from Original Concentration

Since the O&G analysis is a conservative analysis, the reduction of petroleum HCs was probably higher than shown by these results. O&G extractions include biodegradation intermediates such as fatty acids and phenols (Bossert and Bartha, 1984). These intermediates also have increased molecular weight because of the heavier oxygen atom attached to their structure. An evaluation of the changes in the GC fingerprints of the compost extracts indicates that the reductions were more than that shown by the O&G data alone.

GC Type-Analyses.

GC type-analyses or "fingerprints" were performed on starting materials, background samples, and the samples collected over the course of the study period. The fingerprints of the sludge feed and the starting compost stock are shown in Figure 56 A and B. The fingerprints were normalized to the y scale in each chromatogram since they are used in a qualitative rather than a quantitative manner. These show a characteristic weathering by the minimal presence of HC molecules in the region from about C19 to C30. These figures are very similar. Upon comparison with Figure 56 C and D, for the background soil and the straw extracts, a contribution to the C31, C33, and C34 peaks in the starting compost material (Figure 56 B) by the straw can be seen. There was little to no apparent contribution to the compost material by the background soil.

When observing the fingerprints over the study period, several peaks may be distinguished as markers or "biomarkers." The C17 and C18 peaks, not present in the background and straw samples, showed little attenuation in the compost samples throughout the test. They may be considered as biomarkers because of their resistance to degradation compared to the other molecules (McMillen et al., 1993b). These biomarkers are useful to gauge the attenuation of the other HC molecules. It is uncertain whether or not these peaks were the branched isoprenoid pristane and phytane biomarkers noted by others. Additionally, the peak at C33 retained its characteristic shape from the contribution of the straw throughout the study period for both the SP and WR samples. Finally, C34 tended to have much less attenuation than the C35+ HC molecules and may be used as a marker to observe the attenuation of the higher-boiling-point fraction of molecules in the fingerprints.

Inspection of the SP samples (Figure 57 through Figure 59) and WR samples (Figure 60 through Figure 62) show similar trends over the test period for each set.

- The C15 peak gradually attenuated in each pile system over the test period and almost completely disappeared in the WR sample by the end of the test.

- There was some attenuation of the C16 peak in each system, with slightly more for the WR system.

- A moderate amount of attenuation of the mid-range molecules C19 to C33 occurred although this range was initially depleted in the sludge feedstock.

- There was a slight reduction in the C35-C37 range.

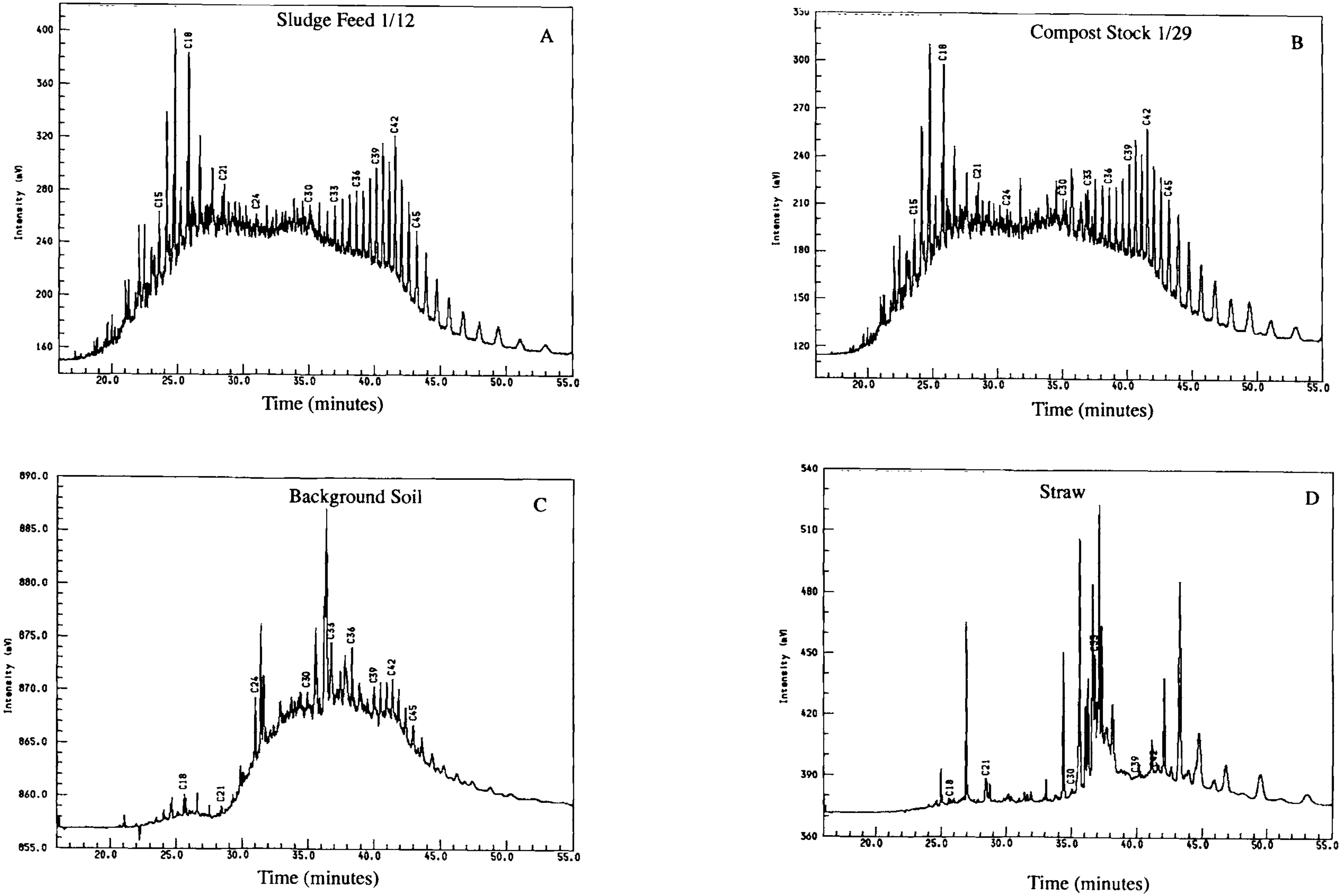
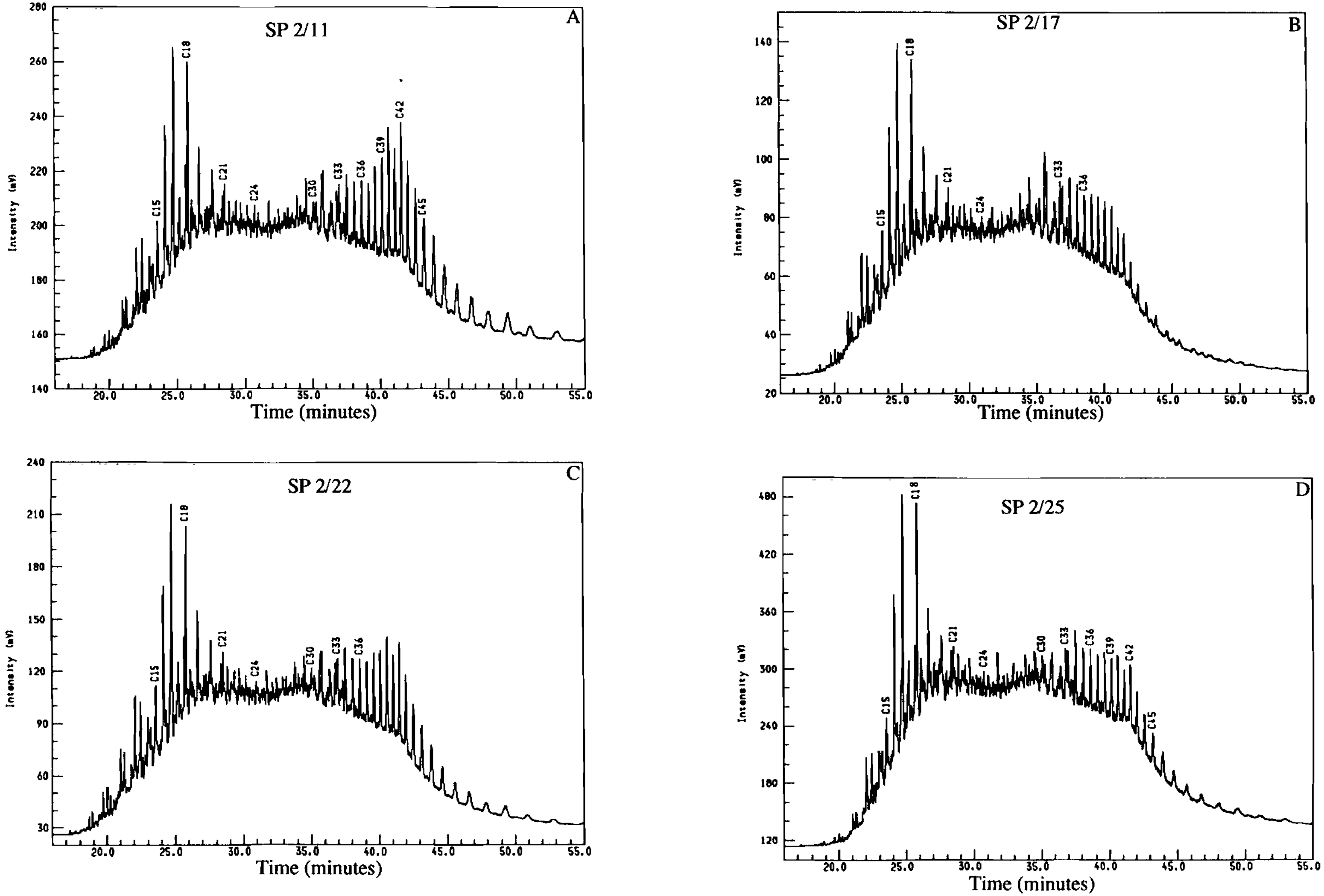


Figure 56. GC Fingerprints of Feedstock Materials


SP 2/11

SP 2/17

SP 2/22

SP 2/25

Figure 57. GC Fingerprints of SP Compost Extracts 2/11, 2/17, 2/22, and 2/25

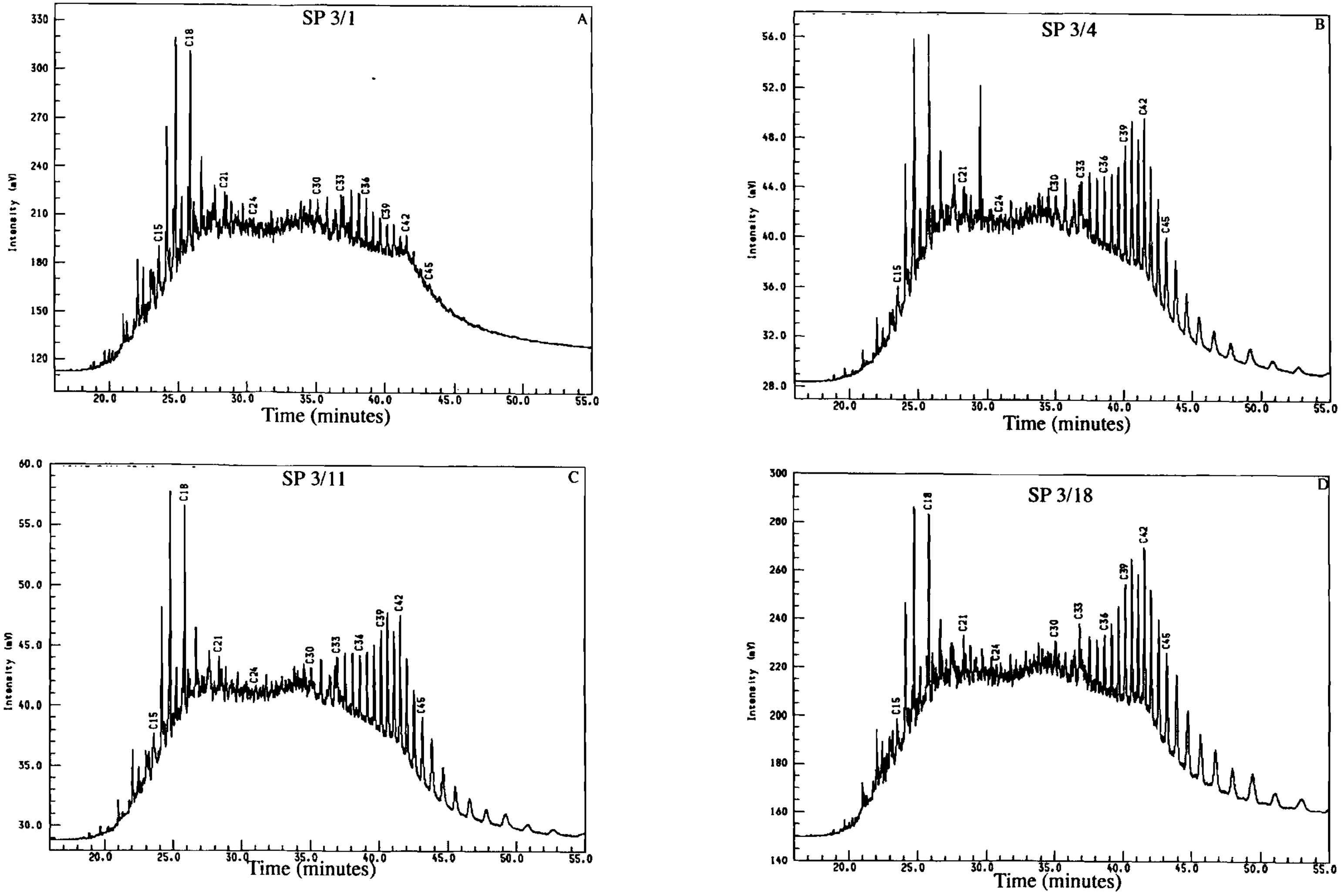


Figure 58. GC Fingerprints of SP Compost Extracts 3/1, 3/4, 3/11, and 3/18

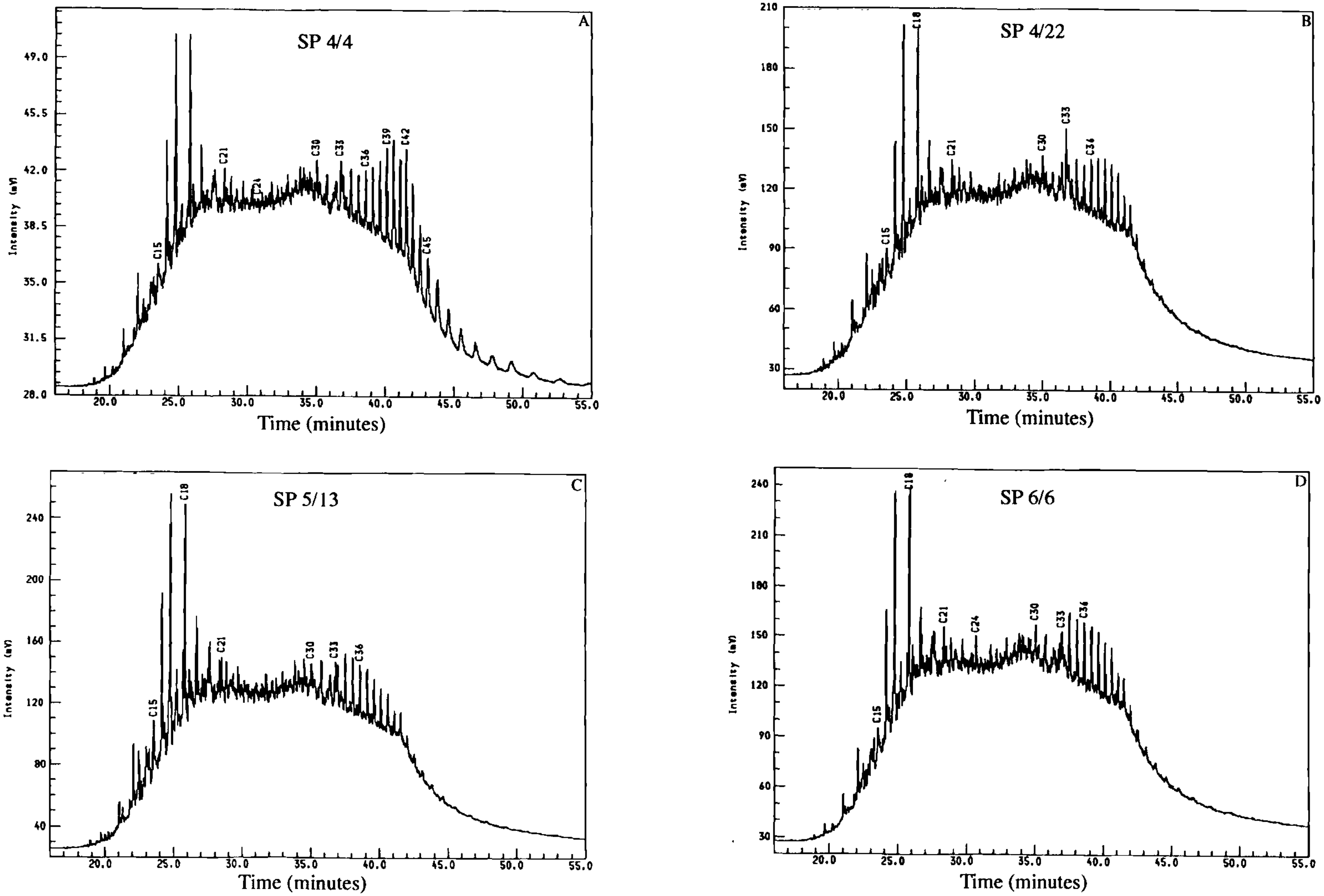


Figure 59. GC Fingerprints of SP Compost Extracts 4/4, 4/22, 5/13, and 6/6

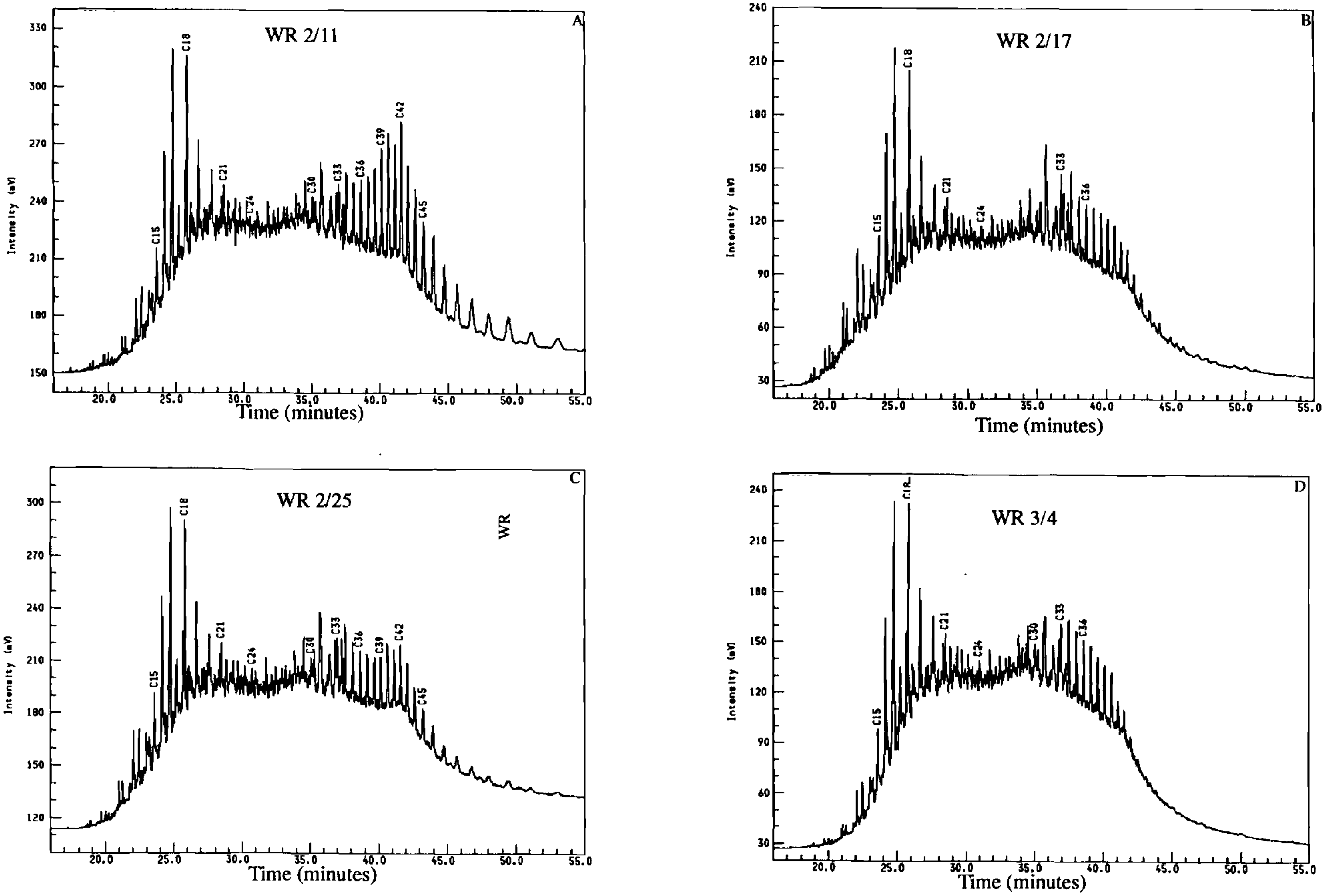


Figure 60. GC Fingerprints of WR Compost Extracts 2/11, 2/17, 2/25, and 3/4

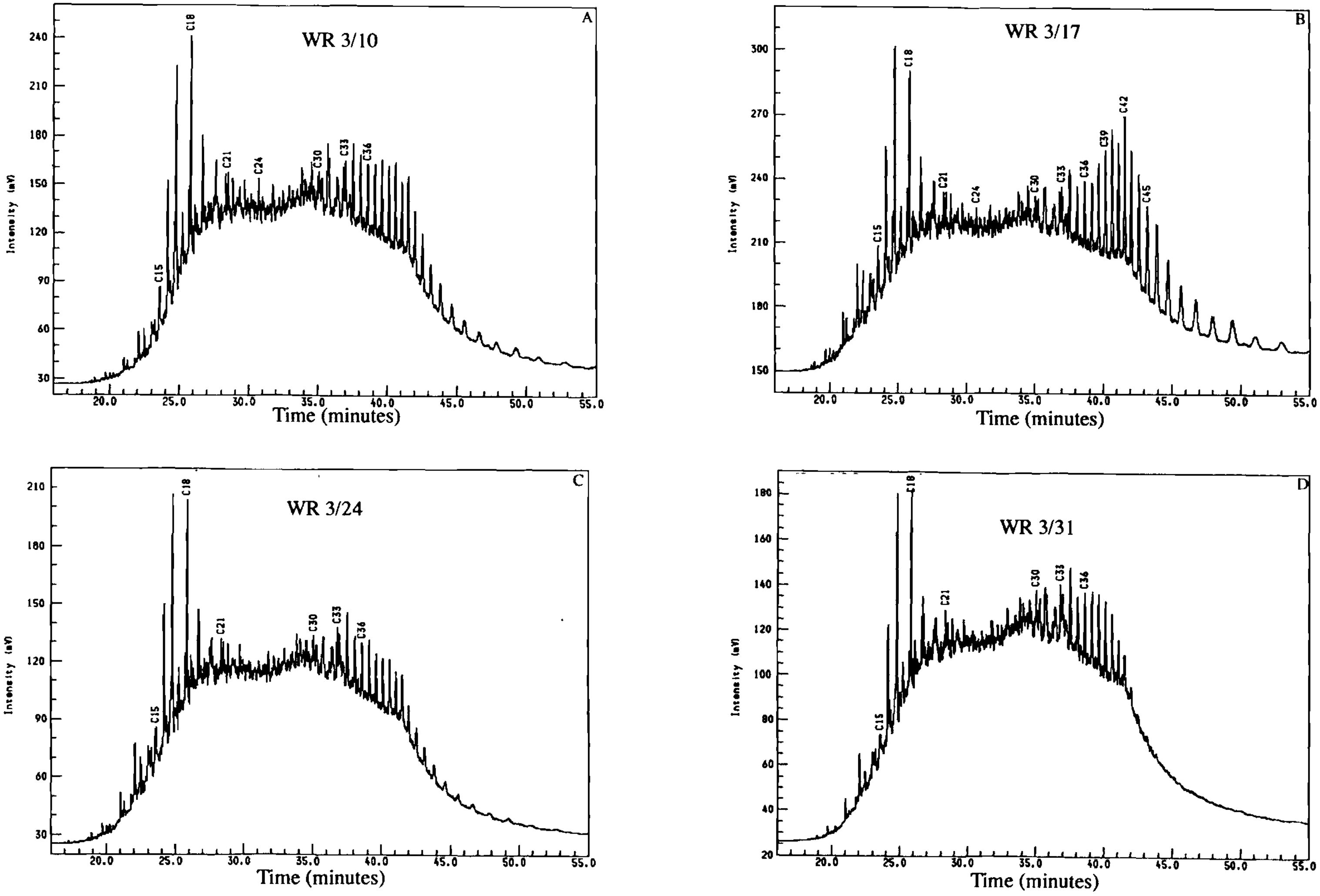


Figure 61. GC Fingerprints of WR Compost Extracts 3/10, 3/17, 3/24, and 3/31

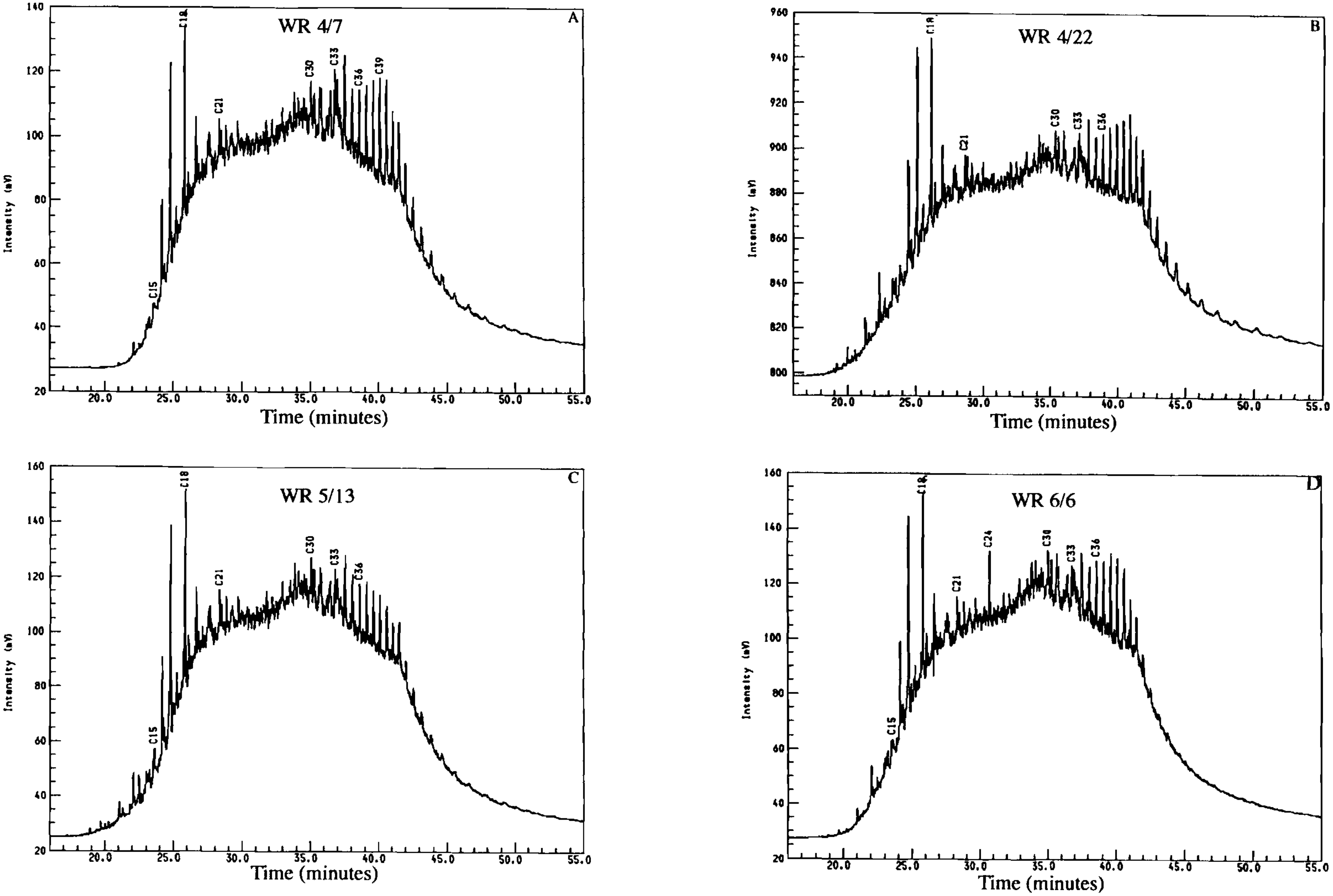


Figure 62. GC Fingerprints of WR Compost Extracts 4/7, 4/22, 5/13, and 6/6

- A significant amount of reduction is evident in the C38-C40 range molecules.
- Attenuation of the high-boiling-point molecules C41 to C54 was high for both pile systems. This was quite evident for SP samples after April 4 (day 58) and for WR samples after March 17 (day 40). Peaks from 41 to 55 minutes are significantly reduced or even totally lacking in the chromatograms after these dates.

Although peak attenuations can be seen over the test period, they are not as large as some investigators have shown in their bioremediation studies using petroleum hydrocarbons. This can be partially explained by the weathered nature of the starting material. The type-analysis is consistent with the O&G analyses of the SP and WR systems which indicated partial reduction of O&G. It is difficult to establish from the type analyses which system had better performance since differences in the chromatograms were subtle. Finally, the greatest changes in the fingerprints occurred by the end of the active compost period, about day 60 of the tests. Additional changes in the fingerprints would not be expected to occur to any large extent if the tests were continued.

BTEX

The results listed in Table XVII show that all concentrations of the initial and subsequent BTEX concentrations were below detection limits except for the initial ethylbenzene concentration in the January 12 sludge material. The low values may have resulted from the vigorous mixing and volatization of these compounds during the sludge homogenization and the compost preparation.

Moisture Content

Real-time measurement of moisture in the SP was unsuccessful due to the failure of the time domain reflectometer. It is suspected that the connection between the extension cable and the waveguide cable was compromised by the moisture in the SP. Therefore, the

TABLE XVII

BTEX CONCENTRATIONS IN SLUDGE AND COMPOST PILES

Sample	Benzene, ppm	Toluene, ppm	Ethyl Benzene, ppm	Xylenes, ppm
January 12 Sludge	< 0.50	< 0.50	1.90	< 0.50
January 29 Compost Feed-stock	< 0.50	< 0.50	< 0.50	< 0.50
February 11--SP Compost	< 0.50	< 0.50	< 0.50	< 0.50
February 11--WR Compost	< 0.50	< 0.50	< 0.50	< 0.50
February 25--SP Compost	< 0.50	< 0.50	< 0.50	< 0.50
February 25--WR Compost	< 0.50	< 0.50	< 0.50	< 0.50
June 6--SP Compost	< 0.50	< 0.50	< 0.50	< 0.50
June 6--WR Compost	< 0.50	< 0.50	< 0.50	< 0.50

moisture content of the collected samples of both piles were measured on the Denver moisture balance. Figure 63 shows that the decreases in MC for both piles were quite similar during the test period. Of peculiar interest is that the WR was repeatedly moistened during the test, but exhibited the same trend as the SP which was only lightly moistened surficially on two occasions. Thus, the SP conserved its moisture to a much greater extent than the WR. The decrease in MC down to 23% by the end of the test did not appear to inhibit the reduction of O&G in the WR. As shown in Figure 54, O&G reduction was continuing in the WR even at 120 days. This was not the case for the SP although its MC was essentially the same as in the WR. These data suggest that moisture content was not the determining factor in the better performance of the WR over the SP. The increased surface area exposure of the WR which resulted from the pile geometry and frequent pile manipulation was possibly a more critical factor.

pH.

The pH profiles of the two systems are shown in Figure 64 and Figure 65 for both 1:1 and 2:1 mixtures. Both profiles show that the pH's of the respective systems remained in the neutral range during the test and that these conditions should not have been inhibitive

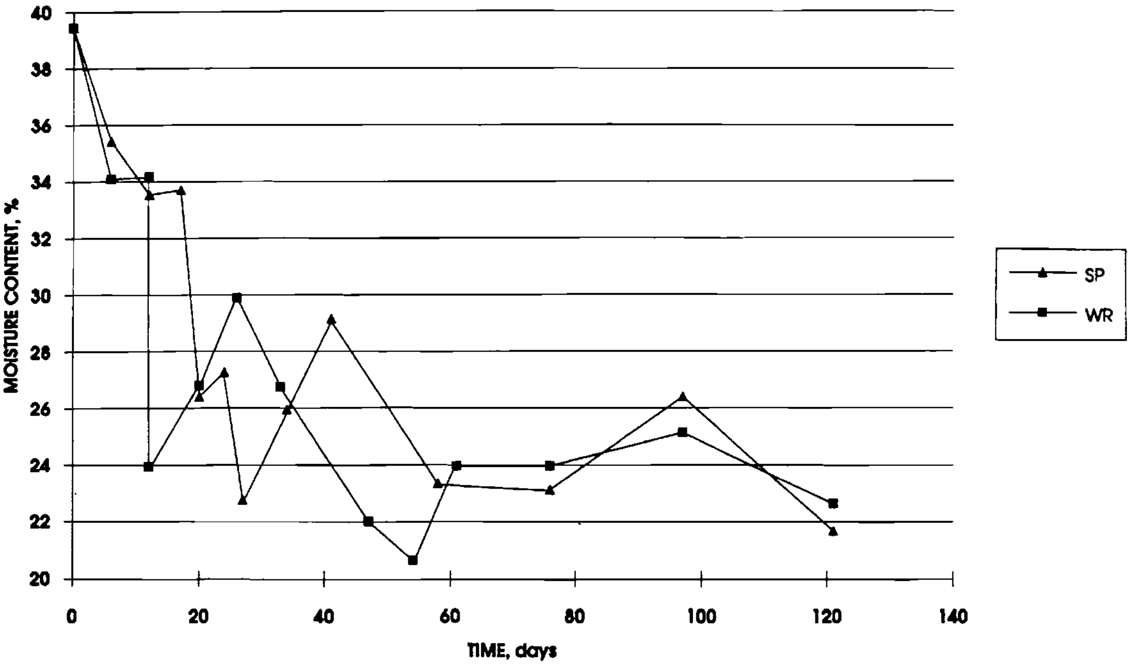


Figure 63. Moisture Content Profiles of the SP and WR Compost Piles

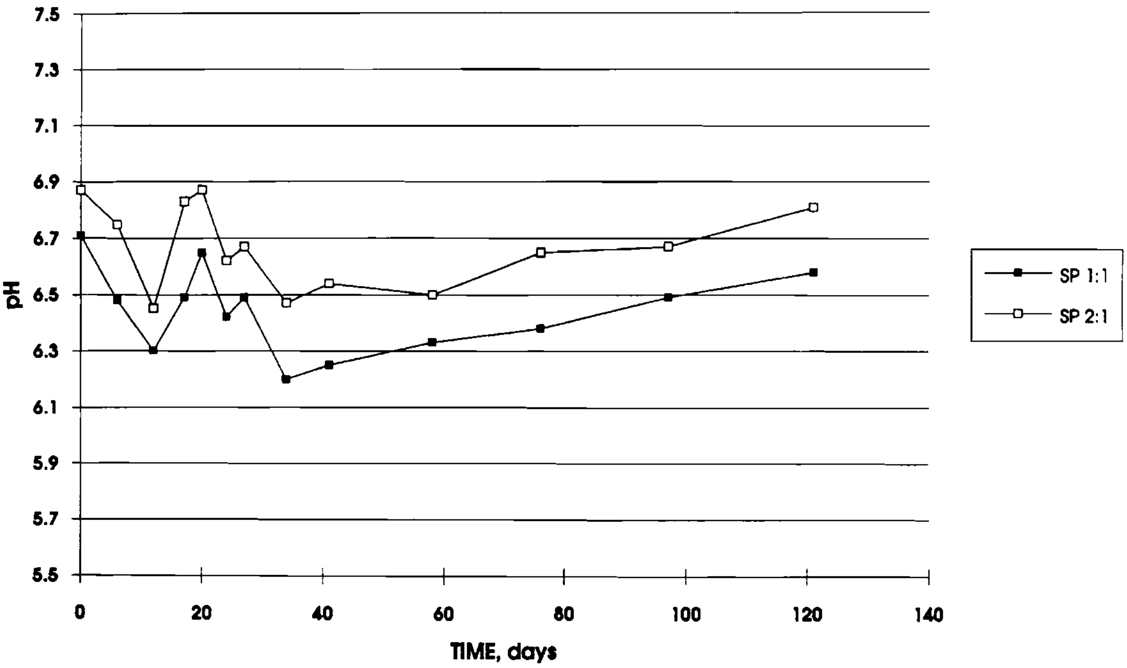


Figure 64. pH Profile of SP Compost

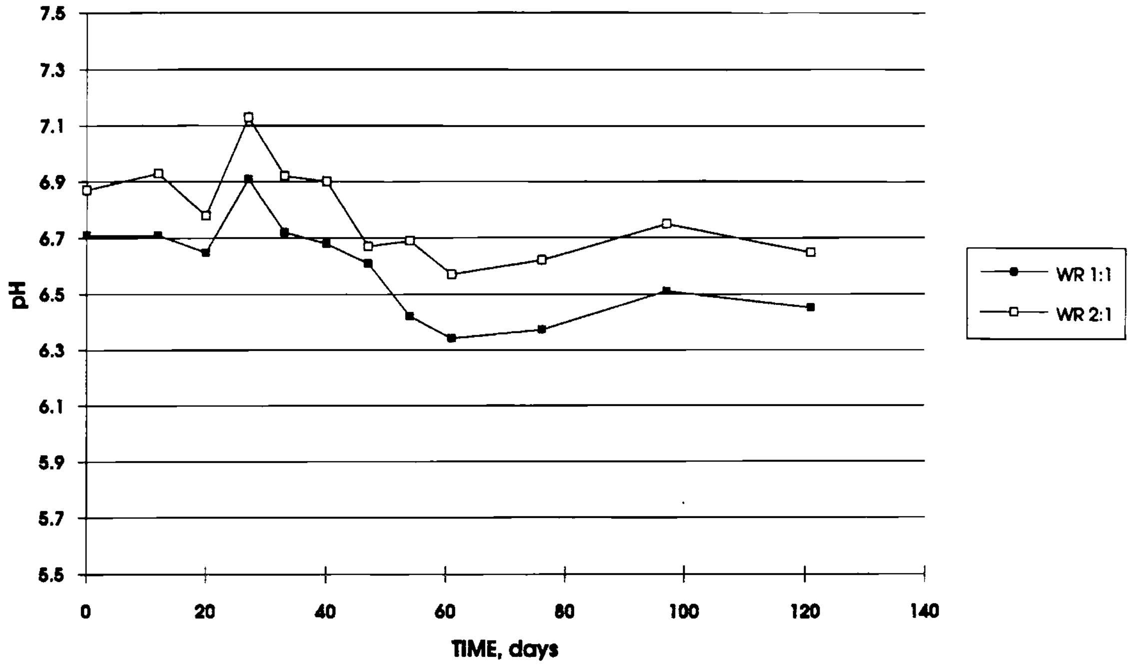


Figure 65. pH Profile of WR Compost

to biological activity. The soil matrix provided an effective buffering system to offset any tendency of the pH to change drastically.

Nitrogen, Phosphorus, and Total Organic Carbon

Nitrogen and phosphorus levels for the each of the piles are given in Table XVIII. No additional nutrients were added to the piles during the test. Total nitrogen from total Kjeldahl nitrogen analyses shows a reduction of 22% in the SP composite sample compared to 41% reduction in the WR composite sample at the end of the test. A comparison between the NH_4^+-N analyses of the SP and WR composites, shown in Figure 66, is consistent with the limited TKN data. The WR data show a reduction of the nitrogen level from 446.6 to 165.2 mg/kg NH_4^+-N in the first 12 days of the test when there was little biological activity in this pile. There was a less dramatic overall reduction in the nitrogen level in the WR during the remainder of the test. Although the distribution of nutrients in the original compost material might be questioned, each nutrient mixture for the compost batches was

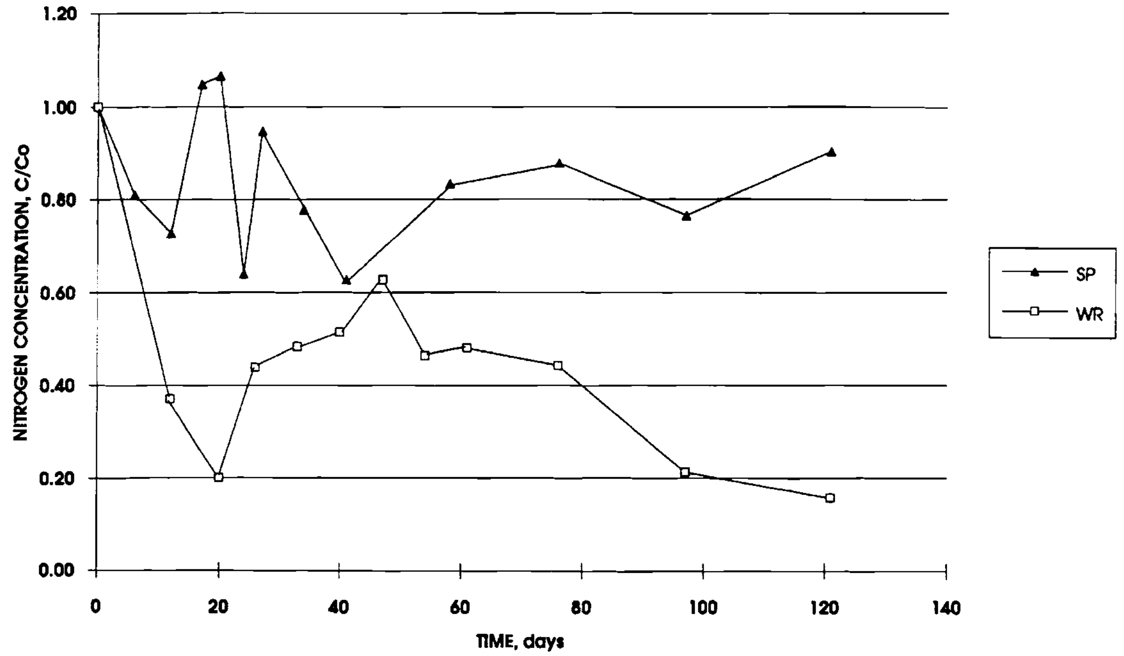


Figure 66. Loss of NH_4^+ -N in SP and WR Compost Piles

carefully made. Therefore, the compost in both piles should have had the same starting composition. One possible reason for the difference in the NH_4^+-N between the two piles is that the periodic mixing of the WR exposed more surfaces of the compost material to sorption of NH_4^+ ions. Other reasons for the difference might be errors in sampling, preparation, and analyses. In any case, the nitrogen lost from the WR pile was at least 50% by the end of the test while the loss in the SP was 20% to 30%.

Inspection of the total organic carbon (TOC) in the two piles during the test, listed in Table XIX, shows that there was essentially no change in the carbon content in either pile during the test. This strongly suggests that biodegradation of petroleum HCs and straw resulted in a substantial formation of cellular biomass with attendant production of polar compounds. This might also support the notion of substantial inclusion of polar compounds in the O&G extractions and that the actual reduction of O&G was greater than that revealed in the extractions alone.

TABLE XVIII

INORGANIC NUTRIENTS IN MATERIALS AND COMPOST

Sample	TKN, mg/kg	NH_4^+-N, mg/kg	NO_3^--N, mg/kg	Total P, mg/kg	EDTA PO_4^{-2}-P. mg/kg	Soluble PO_4^{-2}-P. mg/kg
January 19 Sludge	< 25	22.40	< 1.0	76.00	1.60	< 1.0
Straw	4592	--	--	--	--	--
January 29 Compost Feed	2,240	446.60	< 1.0	542.00	356.00	95.40
February 11 SP Compost	--	361.20	--	--	--	--
February 17 SP Compost	--	324.80	--	--	--	--
February 22 SP Compost	--	467.60	--	--	--	--
February 25 SP Compost	--	476.00	< 1.0	--	--	--
March 1 SP Compost	--	285.60	--	--	--	--
March 4 SP Compost	--	422.80	--	--	--	--
March 11 SP Compost	--	347.20	--	--	--	--
March 18 SP Compost	--	280.00	< 1.0	--	--	--
April 4 SP Compost	--	372.40	--	--	--	--
April 22 SP Compost	--	392.00	--	--	--	--
May 13 SP Compost	--	341.60	--	--	--	--
June 6 SP Compost	1,750	403.20	--	455.00	300.00	71.10
February 17 WR Compost	--	165.20	--	--	--	--
February 25 WR Compost	--	89.60	< 1.0	--	--	--
March 4 WR Compost	--	196.00	--	--	--	--
March10 WR Compost	--	215.60	--	--	--	--
March17 WR Compost	--	229.60	2.60	--	--	--
March24 WR Compost	--	280.00	--	--	--	--
March 31 WR Compost	--	207.20	--	--	--	--
April 7 WR Compost	--	214.20	--	--	--	--
April 22 WR Compost	--	168.00	--	--	--	--
May 13 WR Compost	--	95.20	--	--	--	--
June 6 WR Compost	1,330	70.00	--	477.00	259.00	60.50

The carbon to nitrogen ratios in the two piles increased by the end of the test period. Using the values for TKN, the initial carbon to nitrogen ratio was 18.3 to 1. Using the initial phosphorus level shown as Total P, the N to P ratio was 4.13 to 1 which was essentially the same as provided in the nutrient mixture. Since the amount of nitrogen that was actually bioavailable was less than the TKN value, the true ratio might have been somewhat higher than this number. However, the strong ammonia odor during the highly active period still

TABLE XIX

TOTAL ORGANIC CARBON

Sample	TOC, %
January 12 Sludge	3.2
Straw	19.9
January 29 Compost Stock	4.1
June 6 SP Compost	4.2
June 6 WR Compost	4

suggests that nitrogen was certainly not a limiting factor. This would explain the quick start of the compost process and its high activity. It is uncertain whether or not the activity of this particular compost mixture could have been controlled by changing the nitrogen supplement since the respirometry tests showed no increase in oxygen consumption of nutrient augmented conditions over the unaugmented condition. The WR showed a substantially higher loss of nitrogen during the test than the SP both in terms of TKN and in NH_4^+-N. The C:N ratio for the SP at the end of the test increased to 24:1. Its N:P ratio decreased only slightly to 3.9:1. The changes in the WR were more substantial. The final C:N and N:P ratios were 30:1 and 2.8:1 respectively. Although both piles had a reduction in phosphorus during the test, the changes in the N:P ratio should be considered a function of the change in nitrogen rather than in the change of phosphorus. The N:P ratios were influenced more by the reduction of nitrogen than by the reduction of phosphorus. Furthermore, the value of Total P would not normally be expected to drop since phosphorus should be conserved in the solid and liquid phases of the medium.

Microbial Populations

Microbial populations (Table XX) show that they were not inhibited by any condition within the pile during the test. Since there was a problem with temperature control of the incubator during storage of the compost samples prior to these analyses, the numbers shown here should not be considered absolute values, but rather an indication that there were no biologically inhibitive conditions during the test. The January 12 sludge sample was stored in a separate incubator at 4°C and did not experience temperature fluctuations. Population numbers indicate high initial levels of microbes.

TABLE XX

MICROBIAL POPULATION DURING PILOT STUDY

Sample	Standard Plate Count, colonies/ml	Specific Degraders, CFU/ml
January 12 Sludge	7.0×10^8	5.0×10^8
January 29 Compost Feedstock	1.5×10^9	NA
February 11--SP Compost	1.3×10^9	NA
February 11--WR Compost	4.2×10^8	NA
February 25--SP Compost	1.3×10^9	5.5×10^8
February 25--WR Compost	1.4×10^9	1.0×10^9
June 6--SP Compost	5.5×10^8	NA
June 6--WR Compost	2.5×10^8	NA

Mineralogy and Bioavailability.

Respirometry tests did not indicate that the compost mixture was deficient in macronutrients or micronutrients. Microcosms amended with Evan's mineral medium as the nutrient source for the compost mixture performed no better than microcosms not amended with nutrients. The mineralogy analysis of the sludge feedstock shown in Table III indicates that it consisted of a base amount of quartz minerals with a mixture of clays and potassium, calcium, magnesium, and iron minerals. Although they were not analyzed, it is presumed that micronutrients were also present in trace quantities in this soil matrix. Since the soil composition was nearly 1/3 clay, it is possible that some of the O&G may have been trapped in the clay structure of the soil and rendered less bioavailable to degradation. Although this notion was not investigated in this study, it is currently a topic of interest for investigation by environmental specialists in the petroleum industry. The clayey soil matrix in this study is certainly a more viable candidate for this premise than a sandy soil matrix.

CHAPTER V

CONCLUSIONS

Biodegradation of O&G in the Compost of the Two Pile Systems

This study suggests that biodegradation of some highly weathered crude oil sludges may be impractical with composting due to low biodegradation rates. The time required to reach the biodegradation endpoint of such a sludge may be too long to make the project economically feasible. The O&G content was reduced by approximately 8% wt and 20% wt in the SP and WR, respectively over the 121 day pilot test period. Half-lives of the O&G for these systems were 4 years and 1 year, respectively. However, the actual reduction of sludge HCs in the two composts was probably greater than that indicated by the O&G measurements alone. Inspection of GC type-analysis "fingerprints" of compost extracts indicated that there was significant reduction of the higher boiling point C31+ HC fraction in both the SP and the WR.

Since the O&G endpoint was lower in the WR compost than in the SP compost, biodegradation of the weathered sludge material appeared to be more effective in the WR. The lower O&G endpoint was achieved in the WR even though it took two times longer to reach peak temperatures than the SP and had frequent temperature upsets from mechanical turning events.

The effect that the clays from the sludge in the compost had on the biodegradation rate is uncertain. Soils and minerals vary considerably in their abilities to adsorb and trap other particles. Clays, being highly adsorptive and possessing complex mineral structures, may have inhibited microbial access to HCs by trapping macromolecules within their structures. Such effects on bioremediation of petroleum HCs are not yet fully understood.

Both compost systems were able to self-heat in cold weather conditions about a week after the piles were constructed. The 2:1 straw to sludge compost augmented with nitrogen and phosphorus attained high temperatures near 160°F in the SP and greater than 120°F in the WR. Temperature control was difficult in the SP at the air throughput rate of 155 pore volumes per day. A higher air throughput rate or a reduction in the pile height may have achieved better temperature control in the SP. Temperatures in the WR were only marginally excessive and exceeded the upper limit of the mesophilic range (113°F) by reaching 120°F for about 5 days. Other investigators have found that biodegradation of petroleum HCs occurs more effectively at mesophilic temperatures than at thermophilic conditions. Although the greater reduction of O&G in the WR than in the SP may have been due to maintaining WR temperatures near the mesophilic range, other factors may have also contributed to this improved performance. Periodic mechanical mixing should have exposed more HCs for microbial consumption by increasing contact along surfaces. The frequent mixing also nullified the effects of compaction in the lower elevations of the WR and evened the distribution of the compost materials throughout the test period.

Volatile organic compounds (VOCs) were highest in both piles during the high activity period. Maximum values were generally less than 140 and 100 ppm (as hexane) for the SP and WR, respectively. At other times, VOCs averaged less than 50 ppm in each pile. These low values suggest that either the biodegradation rates of the HCs were low or that mineralization of the HCs was nearly complete. These values were well below the lower explosive limit (LEL) for combustible gases. Composting other highly weathered sludges may be expected to produce the same low levels of VOCs.

Odor from the composting process was significant. Because both piles were in the same vicinity, it was difficult to determine if both piles or just the SP emitted the strong "barnyard" odor. Nevertheless, it is uncertain if the odor could have been controlled by reducing the amount of chemical nutrients added to the compost. Thus, odor and its control, when using straw as the bulking agent, should be considered when planning any large-scale compost project.

Composting using straw and chemical nutrients added to petroleum sludge induces vigorous microbial activity. Self-heating of compost piles can occur in 1 to 2 weeks even under cold weather conditions. Chemical nutrients are inexpensive insurance that self-heating will occur, but some additional cost is associated with applying them to the compost mixture.

Obtaining good homogeneity in compost requires a substantial amount of labor or mixing equipment. Although it is not imperative that compost homogeneity be obtained, hot spots will be reduced or eliminated in compost piles by providing even mixing and distribution of the hydrocarbons. There will be less deviation among samples gathered during any one sampling event. As a result, interpretation of analyses and the understanding of biodegradation will be improved.

Treatability Testing

Straw and sludge were successfully mixed without straw binding up on the blades of small and large rototillers. There was no need to precondition the straw by chopping it before mixing was conducted. This suggests that straw may also be blended successfully to make compost using other types of mixers such as pug mills or ribbon blenders.

The order of wetting the straw and sludge had only a small impact on the water holding capacity of the final compost mixture. The container capacity (CC) of the 2:1 straw to sludge mixture ranged from 90 to 101% using a sludge with approximately 10% wt O&G content. The CCs of other compost systems could be expected to deviate substantially from these values, especially if the bulking agent is highly absorbent. In many cases, it may be most practical to wet the bulking agent and the sludge together when mixing the compost. However, initial CC tests should be conducted to determine the effects of the order of wetting.

Permeability reduction from compaction was more profound in the 2:1 than in the 3:1 straw to sludge compost mixture. The 3:1 and the 2:1 mixtures required approximately 35% and 16% compaction, respectively, before each experienced a significant loss of permeability. When the mixtures reached these levels of compaction, they became water saturated which caused the sudden reduction in permeability. Permeabilities in the 3:1 and 2:1 mixtures dropped from infinite to 11 Darcys and from 240 to 3 Darcys, respectively, from the uncompacted to the compacted-saturated condition. When compaction of the 2:1 mixture was increased to 26% to simulate the degree of compaction at the base of an 8 ft column of compost, the permeability decreased to 0.5 Darcy. The 3:1 mixture was already water saturated at 35% compaction. Although air flow through porous media can still be readily achieved at 0.5 Darcy permeability, the effect of compaction on air flow is quite dramatic. If the compost mixing ratio is less than the above ratios, the permeability will be reduced even more when the compost is compacted.

The air flowing from forced-air tubes in the SP predominantly traveled upward since compaction decreased toward the top of the pile. A shorter static pile height than used in this study would have reduced the effects of compaction on air flow, especially near the bottom of the pile. The internal heat in the pile might have also been more easily controlled since the permeability would have been higher and the thermal mass would have been lower.

Since the WR was turned on a weekly basis, the compost material in the WR remained loose or dissociated throughout the test period. This loose material structure facilitated gas diffusion throughout the pile. Since the WR was frequently manipulated, the effects of any compaction that may have occurred at the bottom of the pile were temporarily sustained.

If the bulking agent to sludge ratio is lowered, the compost material is more easily compacted. In order to provide adequate oxygen to a windrow with a lower mixing-ratio compost than the 2:1 ratio used in this study, the pile would require more frequent turning since diffusion of gases would be reduced.

Respirometry studies showed that oxygen consumption increased as the ratio of straw to sludge in the compost mixtures increased. This suggests that the biological activity was largely centered on utilization of straw rather than the petroleum HCs. This was also observed in the pilot test during the period of high activity. However, there was little correlation between the period of high activity in the pilot test, as indicated by elevated oxygen consumption and temperatures, and O&G reduction. O&G reduction continued at about the same rate over the entire 121 day test period even though the period of high activity in both piles lasted about 60 days.

Respirometry studies were also conducted at the 2:1 ratio at different nutrient loading rates. The mixture with 0.54 N nitrogen had significantly less cumulative oxygen consumption than the unamended mixture and the 0.27 N nitrogen amended mixtures. This suggests that the higher nutrient condition was inhibitive to biological activity. The 0.27 N nitrogen amended mixtures had less oxygen consumption than the unamended mixtures, but reached the endpoint sooner. Since the respirometry studies demonstrated that the 0.54 N nitrogen caused some degree of biological inhibition and the 0.27 N nitrogen did not, the latter was chosen for the pilot test. However, O&G was not analyzed in the respirometer compost mixtures and a correlation cannot be established between oxygen consumption and O&G degradation. Since biodegradation of HCs continued in the SP and the WR long after the highly active period ended and oxygen levels returned to near ambient conditions, HC biodegradation using respirometry should not be evaluated on the basis of oxygen consumption alone.

Conclusions from the Static Pile Test

When composting using the static pile system with a highly active compost, it is more difficult to control heat in the pile than to provide adequate oxygen with forced aeration. When aeration was interrupted in the SP during the highly active period, oxygen declined gradually in the pile over the next 3 days. When aeration was resumed, oxygen concentrations in the most depressed locations increased by 7% in 3 hours (6-13% and 9-16%). Temperatures gradually rose 3° to 8°F during the 3 day interruption and required about 24 hours to return to their previous levels.

Depression of oxygen levels was observed in locations not in the direct air flow paths between air tubes. For the SP compost system in this study, a forced-air tube spacing with greater than 3 ft of lateral spacing might result in oxygen levels less that 5% in deadspots. Such oxygen levels might be even lower for less permeable compost mixtures.

Forced aeration was terminated in the SP after 60 days. At this point, pile temperature and oxygen levels were near ambient conditions. Subsequently, there was not a rise in temperatures or a large decline in the oxygen concentrations in the pile even though biodegradation of HCs continued. Therefore, temperature and oxygen levels in the pile near ambient conditions may not indicate that biodegradation of HCs has ceased or slowed down.

Temperatures were significantly higher in the top half than in the bottom half of the SP. Even though the flow rate of the air was adjusted to provide twice the throughput in the top half over the bottom half, the temperatures in the top half were not adequately controlled. Had the upper set of the forced-air tubes been placed lower and nearer to the center, better control might have been achieved. Air flowing predominantly in the region above them could have ventilated the hottest part of the pile in this manner. The other set of forced-air tubes might have been better located at the base of the pile to even the distribution of the air flow and to more effectively aerate the least permeable area of the pile.

Nearly all of the compost material in the SP was subject to robust biological activity. Elevated temperatures were recorded in all locations of the SP including the very near surface. Oxygen levels decreased to 15% near the surface during the highly active period. and temperatures ranged from 120° to 160°F depending upon location.

The SP tended to conserve moisture much better than the WR. This may have been caused by greater formation of water in the SP due to its higher overall microbial activity. Cored samples near the bottom of the pile also generally appeared to contain more moisture than samples higher in the pile. This phenomenon is minimized in windrow piles since periodic turning redistributes the moisture that accumulates from gravity drainage.

When the SP was operated under vacuum during the highly active period and ambient temperatures were near freezing, water condensed in the forced-air tubes and severely restricted the air flow in the system. This condition was alleviated by operating the system under positive pressure. Therefore, operation of forced-air systems under cold ambient temperatures requires either positive air pressure or elimination of restrictions to air flow from the accumulation of water condensate when operated under negative pressure or vacuum.

Conclusions from the Windrow Test

In this study, reduction of petroleum HCs by windrow composting was more effective than with static pile composting. The WR was simpler and thus faster to construct than the SP. It did not require electrical power or aeration equipment. Although periodic mixing of the WR requires equipment and manpower, an SP also requires some periodic inspection and maintenance of equipment. Once the composting process passes the highly active period, subsequent management of the windrow can be performed on a very low level. Degradation of HCs from a highly weathered sludge may continue in the windrow pile for some time afterward.

The internal conditions in the WR were quickly and easily checked by using portable gas indicators and a thermometer. Thus, manipulation of the WR may be performed based upon oxygen and temperature requirements rather than at scheduled intervals. Periodic manipulation of the WR allows redistribution of moisture within the pile, adequate temperature and oxygen control, improves homogenization of HCs, and facilitates exposure of HCs to microbial activity.

The windrow used in this study had a small pile geometry and was more highly influenced by ambient weather conditions than the SP which was much larger and had better insulating properties. Manipulation of the WR during cold ambient temperatures quickly reduced elevated internal temperatures. However, the pile readily regained the elevated temperatures during the highly active composting period.

Recommendations

Selection and design of a bioremediation project should be conducted with proper planning. The design engineer must consider the appropriate remediation technology based upon facility assets, target contaminant action levels, resistance of the contaminant to biodegradation, and the time required for remedial action. To assist in choosing a remediation technology, an analysis of the sludge is recommended in order to know the distribution of HC molecules and the degree of weathering. Laboratory treatability tests are highly valuable as a next step to determine the capability of the remediation technology chosen. If bioremediation is chosen to mitigate HC contamination, such tests should include respirometry, wetting, mixing, and compaction tests. Laboratory treatability tests can be extended to microcosm or reactor tests which provide information on degradation rates and contaminant level endpoints. Finally, pilot scale tests are recommended so that the operator gains an insight into degradation of contaminants under field conditions and a practical understanding of full scale operations.

For robust compost systems using forced aeration, an SP height of 6 ft of less is recommended to help maintain temperatures in the mesophilic range. Forced-air tubing should then be placed at the 0 ft and 4 ft elevations. Passive tubing would be installed at the 2 ft elevation only.

For both windrow and static pile systems, gas and temperature monitoring should be performed in compost systems. This study has shown that such monitoring can be performed quickly and easily. Both systems can be monitored by inserting probes, which are attached to hand-held instruments, into the pile.

Considerable attention should be given to the composition of any petroleum sludge to be remediated and to the analyses used to accomplish this task. Two or more methods for analyzing the HCs in a complex petroleum sludge are recommended in order to establish an accurate picture of degradation. Since considerable spatial variability in the HC content in soil or compost is likely to occur, especially in applied remediation operations, the operator must balance the number of analyses performed with the sophistication and cost of the analytical methods chosen for this purpose. Too few samples that provide comprehensive information will not likely be representative of the system as a whole. However, a large number of samples analyzed by a single method, such as the oil and grease method, may be conservative in assessing the reduction of target compounds.

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APPENDICES

APPENDIX A

PROCEDURE FOR MEASURING CONTAINER CAPACITY (CC) OF COMPOST MIXTURES

The following procedure provides the overall procedure for measuring container capacity (CC) used in this study. It is followed by the specific method used to measure the CCs of the final 2:1 compost mixtures. The routine used to calculate the CCs is also included.

1. Measure bulk densities of bulking agent and sludge so that laboratory mixtures can be prepared on a mass basis after being converted from the desired volume:volume (v/v) basis. In this example, straw is used as the bulking agent.

2. Prepare large plastic beakers to fill with the material to be tested.
   - Obtain 800 ml plastic beakers.
   - Fill with a known amount of water and mark the volume on the beakers at 500 ml and 800 ml levels.
   - Empty the beakers and drill 1/8 inch drain holes in the bottoms of the beakers (CC beakers).
   - Weigh the empty, CC beakers on a balance and write these weights on the beakers with permanent ink.

3. Prepare a compost mixture where **straw is wetted first.**
   - Using the bulk density of straw previously measured, weigh out in a large vessel, sufficient straw for a desired v/v bulking agent to sludge mixture.
   - Add water until the straw is saturated and excess water is visible. Let this stand for 1 hour.
   - Using the bulk density of sludge previously measured, weigh out and add the appropriate volume of sludge and stir until the mixture is homogeneous.
   - Transfer all material to one of the CC beakers.
   - Partially fill a larger vessel with DI water.
   - Immerse the CC beaker with the compost mixture into the larger vessel with water.
   - Stir the mixture gently to remove trapped air.

- Ensure that the larger vessel has enough water to reach the top of the compost mixture. Add water and stir again if necessary.
- Let the mixture stand for several hours in water.
- Remove the CC beaker from the larger vessel and cover with a watch glass or plastic wrap.
- Suspend the CC beaker to allow excess water to drain for 24 hours.

4. Prepare the same compost mixture where **sludge is wetted first.**
   - Prepare as in No. 3, but wet the sludge first, then add straw.

5. Prepare the same compost mixture where the **straw and sludge are wetted and mixed together**.
   - Weigh the appropriate amounts of straw and sludge for the mixture into a CC beaker.
   - Mix the straw and sludge until the mixture is homogeneous.
   - Lower the mixture into a larger water-containing vessel.
   - Gently stir the mixture.
   - Let it stand for several hours.
   - Remove the CC beaker cover, and let it stand for 24 hours to drain excess water.

6. Weigh the CC beakers and their contents at the end of the 24 hour draining period.

7. Measure the moisture contents of the original unwetted straw and sludge materials.

8. Subtract the water contained in the original unwetted straw and sludge materials to calculate the total dry weight of these components.

9. Calculate container capacity, CC:

$$\mathrm{CC}^{\%} = \frac{\text{Saturated wt} - \text{Dry wt}}{\text{Dry wt}} \times 100$$

Container Capacity for Final 2:1 Compost Mixtures

Prepare 500 ml of 2:1 Compost Mixture

1. Weigh an empty plastic beaker prepared with 1/8 inch drain holes in the bottom (CC beaker).
2. Weigh a dry paper filter cut to fit the bottom of the beaker.
3. Calculate the volume of materials needed on a weight basis.
    - 500 ml / 3 parts=167 ml per part.
    - 2 parts straw x 167 ml per part x 0.123 g/ml straw=41.1 g straw needed.
    - 1 part sludge x 167 ml per part x 1.7174 g/ml sludge=286.8 g sludge needed.

Make a Mixture with Straw Wetted First

1. Place 41.1 g of straw in a separate beaker of water and let it stand for at least 1 hour.
2. Moisten 286.8 g of sludge with 70 ml (approx. 25% of sludge wt) of water and mix thoroughly to make a smooth paste with no lumps. The paste should not have excess water at these rates.
3. Drain the excess water from the straw.
4. Mix the straw and sludge together very thoroughly.
5. Place the weighed filter paper in the bottom of a weighed CC beaker and wet the filter paper.
6. Place all of the compost mixture in the CC beaker.

Make a Mixture with Sludge Wetted First

1. Weigh 286.8 g of sludge in a separate 1 liter beaker.
2. Moisten the sludge in this beaker with 70 ml of water and mix until a smooth paste is formed.
3. Add 41.1 g of straw into the beaker and mix thoroughly. Add water a little at a time if necessary, but not so much as to create excess water.
4. Place the weighed filter paper in the bottom of the weighed CC beaker and wet the filter paper.
5. Place all of the compost mixture in the CC beaker.

Make a Mixture with Straw and Sludge Wetted Simultaneously

1. Weigh 286.8 g of sludge in a separate 1 liter beaker.
2. Add 41.1 g of straw into the beaker.
3. Add 200 ml of water a little at a time to the beaker and mix thoroughly.

4. Place the weighed filter paper in the bottom of a weighed CC beaker and wet the filter paper.
5. Place all of the compost mixture in the CC beaker.

Immersion and Drainage of the Compost Mixture

1. Slowly submerge the mixture in the CC beaker in a larger container of water until the water level reaches the top of the mixture.
2. Cover with plastic wrap and let stand overnight.
3. After the mixture has soaked overnight, remove the beaker from the standing water and allow it to drain into an empty container by resting it on an object which will not block the drain holes.
4. Check the receiving container periodically to ensure that the bottom of the beaker is above the surface of the water.
5. Allow the mixture to drain for 24 hours.
6. Weigh the CC beaker with the mixture.

Sample wetted first	CC Beaker, g.	Filter Paper, g.	Sludge, g.	Straw, g.	Drained Beaker + Compost, g.	Drained Compost, g.	Unadjusted Container Capacity, %
Straw	44.08	0.67	286.8	41.1	577.38	532.63	62.44
Sludge	44.1	0.68	286.8	41.1	540.76	495.98	51.26
Both	43.68	0.67	286.8	41.1	580.81	536.46	63.6

Final Container Capacity Measurement

1. Measure MCs of compost starting materials.
   - Moisture content of Straw = 15.06%.
   - Moisture content of sludge = 13.22%.

2. Calculate dry weight of starting materials.
   - Dry weight of Straw = 41.1 g x (1.00 - 0.1506) = 35.67 g.
   - Dry weight of sludge = 286.8 g x (1.00 - 0.1322) = 243.61 g.
   - Dry weight of straw + sludge = 35.67 g + 243.61 g = 279.28 g.

3. Calculate CC of mixtures on a dry weight basis.
   - CC% = (Drained Compost - Dry Components) / Dry Components.

Sample Wetted First	Container Capacity, %
Straw	90.72
Sludge	77.59
Both	92.09

APPENDIX B

CALCULATIONS FOR WATER AND NUTRIENT ADDITIONS TO COMPOST

The following method was used to determine the amount of water and nutrients to add to the pilot compost mixture. Water was added to raise the MC of the compost to 60% of the CC, which was determined by the CC treatability test conducted at the 2:1 straw:sludge ratio. Nutrients were calculated on the basis normal (equivalent) concentrations using the amount of water in the compost at 60% of the CC. Since the compost was prepared in 5 batches, totals for materials were divided by 5 to show the amounts used in each batch.

1. Determine total volume of sludge.

   The sludge was spread out in a rectangle and tilled to an even height. Multiple measurements were obtained in all 3 dimensions. The values for each dimension were averaged to derive the following dimensions:

   49 ft L x 24.6 ft W x 0.31 ft D

   Thus, the total volume of sludge used to prepare the compost was: 374 ft^3 or 14 yds^3.

2. Determine the volume of straw to add.

   Straw was added to the sludge on a 2 to 1 volume basis. The total volume of straw used was 748 ft^3 or 28 yds^3. It was assumed that the straw volume in the compost piles would resemble that in the compacted bale form. Therefore, the dimensions of 10 bales of straw were measured, and the average volume per bale of straw was determined to be 6.13 ft^3. Thus, the total amount of straw required for the sludge was 123 bales. Since 5 batches of compost were made from the sludge, this number was rounded to 125 total bales (25 bales per batch).

3. Determine compost water requirement.

The following values were obtained by direct measurement:

MC of straw	15.1% wt
Dry bulk density of straw	0.1045 kg/l (baled form)
Straw density @ 15.1% MC	0.123 kg/l (baled form)
MC of sludge	14.56% wt
Dry bulk density of sludge	1.357 kg/l
Sludge density @ 14.56% MC	1.7 kg/l
CC at 2:1 ratio with whole straw	90% (rounded)

a. Determine the dry weights of the sludge and straw so that the amount of water at the CC can be calculated.

Weight of dry straw = (28 yds^3 straw * 764.6 l/yd^3 * 0.1045 kg/l) = 2,237 k
Weight of dry sludge = (14 yds^3 sludge * 764.6 l/yd^3 * 1.357 kg/l) = 14,526 kg
Total weight of dry compost = 16,763 kg

Water content at the CC = 90% * 16,763 = 15,087 kg = 15,087 liters

b. Determine the total amount of water at 60% of CC

60% * 15,087 liters = 9052 liters = 2391 gallons

c. Determine the residual MC in the stock compost materials.

Water in straw = (28 yds^3 straw * 764.6 l/yd^3 * 0.123 kg/l * 15.1% MC * l/kg * 0.264 gal/l) = 105 gallons
Water in sludge = (14 yds^3 sludge * 764.6 l/yd^3 * 1.7 kg/l * 14.56% MC * l/kg * 0.264 gal/l) = 700 gallons
Total water in stock materials = 805 gallons

d. Calculate the amount of water to add to raise the MC in the total compost to 60% of CC.

2391 gallons total - 805 gallons residual = 1587 gallons makeup water to add
Total rounded to 1600 gallons makeup water (320 gallons per batch).

4. Determine nutrient addition.

At the conclusion of the respirometry tests, nitrogen augmentation was established at 0.25N (eq/l) nitrogen. At a 4:1 ratio of nitrogen to phosphorus, the phosphorus augmentation was calculated as 0.0625N phosphorus. Urea, $(NH_2)_2CO$, and superphosphate, P_2O_5, were used as the sources for these nutrients. The amounts of each were calculated as shown below:

Total wt of Urea = 0.25 eq/l * mol/2 eq * 60 g urea/mol * lb/454 g * 9052 l = 149.5 lbs (30 lbs per batch)

Total wt of P_2O_5 = 0.0625 eq/l * mol/2 eq * 142 g P_2O_5/mol * lb/454 g * 9052 l = 88.5 lbs (17.7 lbs per batch)

APPENDIX C

USE OF THE COMPOST CORING ASSEMBLY

The use of the compost coring assembly is shown in Figures 67 through 73. The figures show the complete assembly, use of the plunger-driver, insertion and use of the coring tool, and extrication of the cored sample. Using two people to perform coring, samples may be obtained at a rate of about 15 minutes per sample. This includes the time required to prepare the equipment for sampling, measuring sampling locations, coring, packaging, and labeling samples, and decontaminating the coring tool between samples.

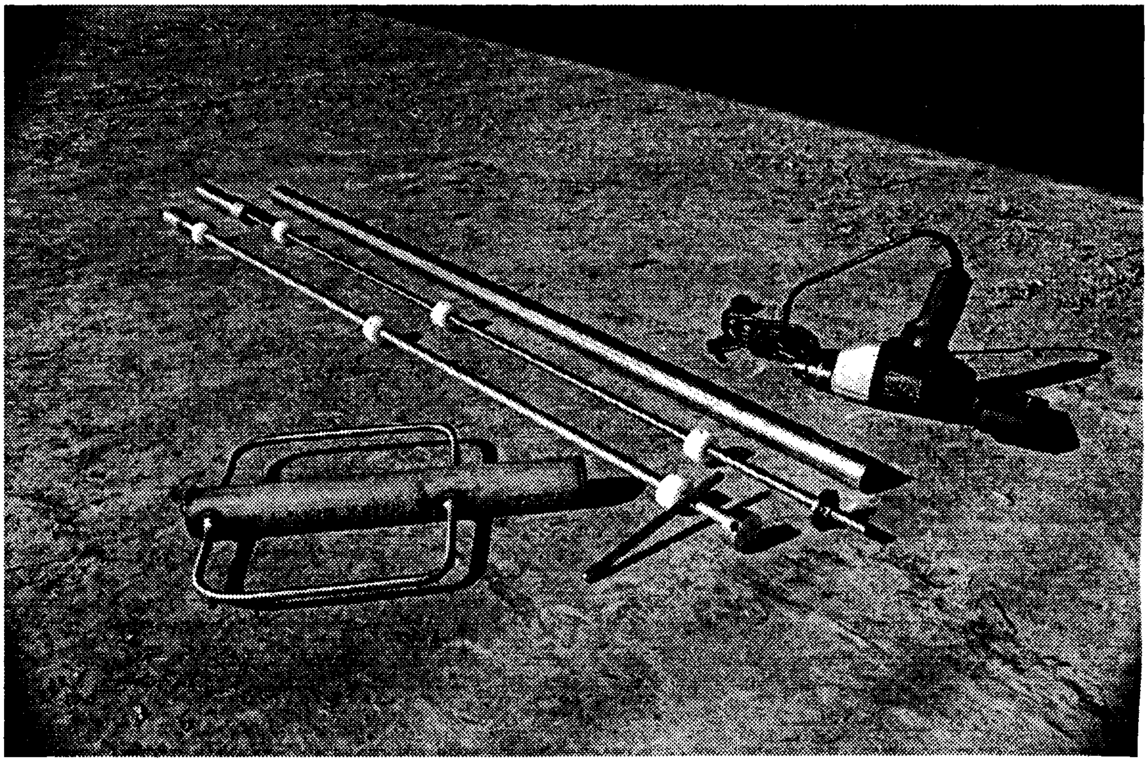

Figure 67. Coring Assembly with Drill Motor

Figure 68. Plunger-Driver Inserted into SS Tubing

Figure 69. Tubing Driver to Sampling Location with Tube-Plunger Driver

Figure 70. Insertion of Coring Tool

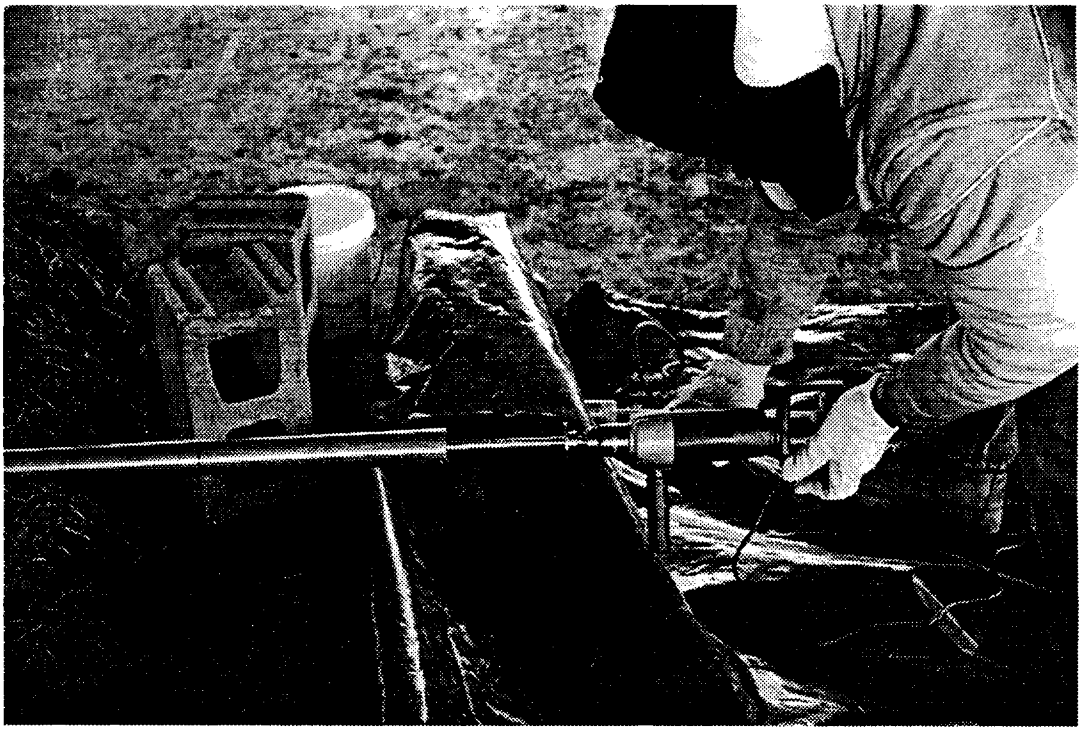

Figure 71. Coring Performed with Drill Motor Attached to Coring Tool

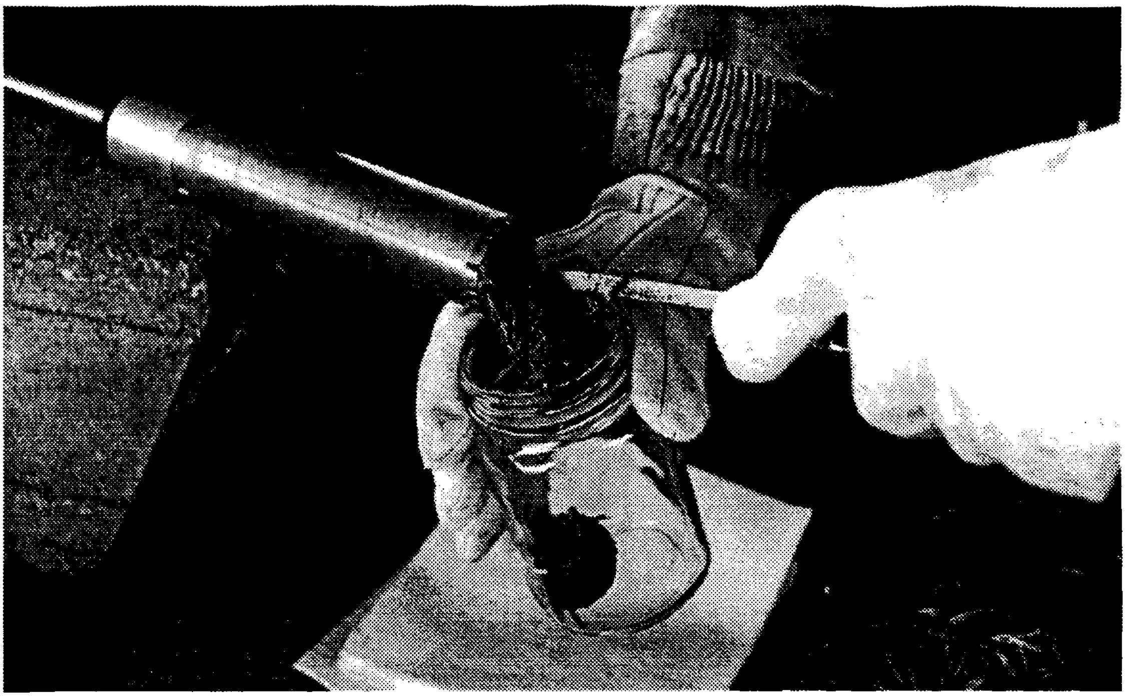

Figure 72. Transfer of Compost Sample from Coring Tool to Sample Container

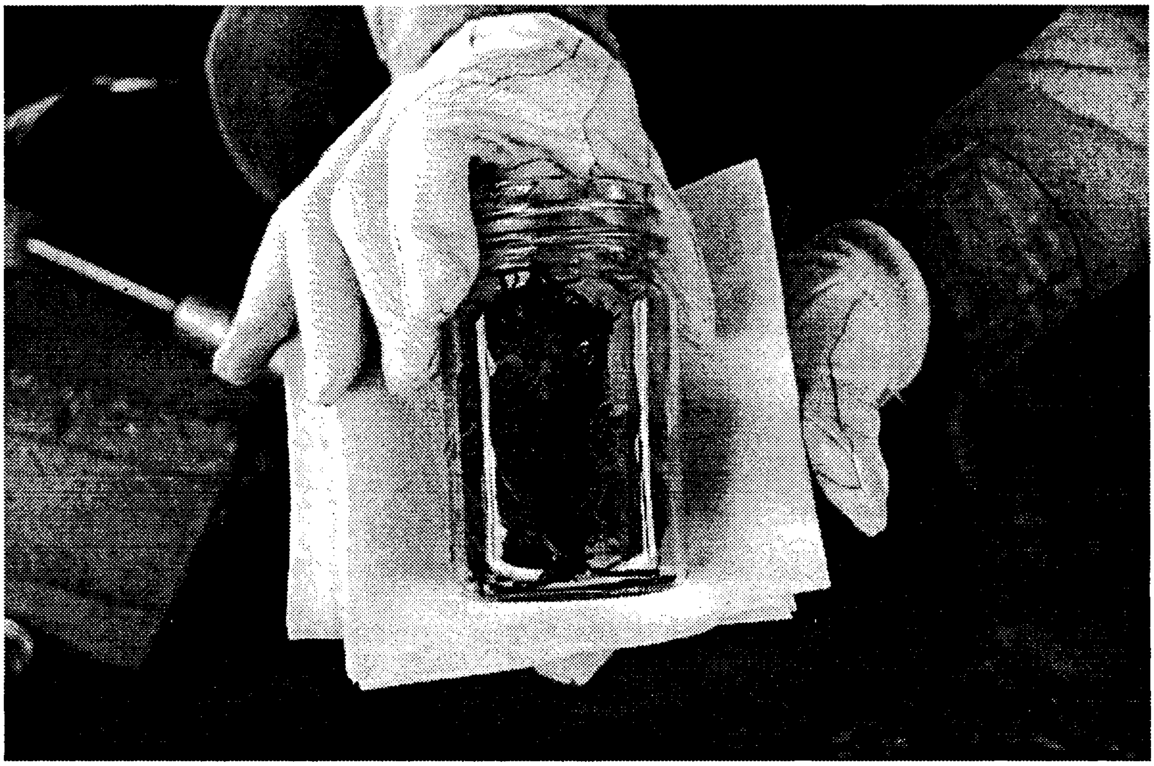

Figure 73. Cored Sample in Half-Pint

APPENDIX D

GAS CHROMATOGRAPH TYPE ANALYSIS METHOD

The compost extracts were run on a Hewlett-Packard 5890 GC with a Flame Ionization Detector (FID). A 1 microliter sample was injected into the GC column by a Hewlett-Packard 7673 Auto Injector. A 15 meter DB-1 column with an internal diameter of 0.32 mm (widebore) with a film thickness of 1 micron was used for chromatographic separation of HC compounds. The carrier gas was zero grade helium. The FID makeup gases were air and hydrogen. The oven temperature ramp is described below:

-25°C initial temperature
5 minute initial time
10°/minute rate
340°C final temperature
30 minute final time

The pressure ramp was:

10 psi initial pressure
5 minute initial time
0.79 psi/minute rate
40 psi final pressure
200 min final time

The injector temperature was oven-tracked (always kept 3°C warmer than the oven temperature before, during, and after temperature ramping). The oven and injectors were cryogenically cooled with liquid nitrogen to achieve the cold temperatures required for analysis. Detector temperature was 345°C. Samples were retention time integrated to identify peaks. The Compost feedstock sample extract was routinely inserted into the sequence of compost samples to monitor the condition of the GC column and ensure the quality of the analysis.

APPENDIX E

CO_2 GAS PROFILES IN THE STATIC PILE

The following Figures (74-85) show the CO_2 response at the sampling node locations shown in Figures 36 and 37. These profiles corresond to the O_2 profiles in Figures 38-49 and the VOC profiles in Figures 86-97.

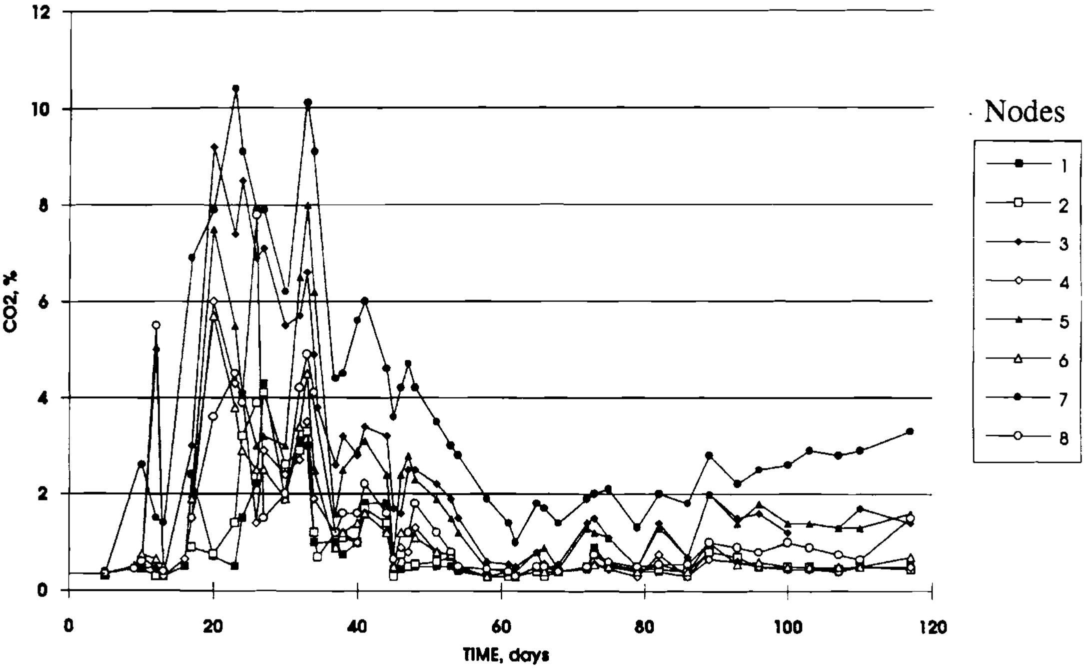

Figure 74. CO_2 Profiles at the 0.5 ft SP Elevation, Center Cross Section

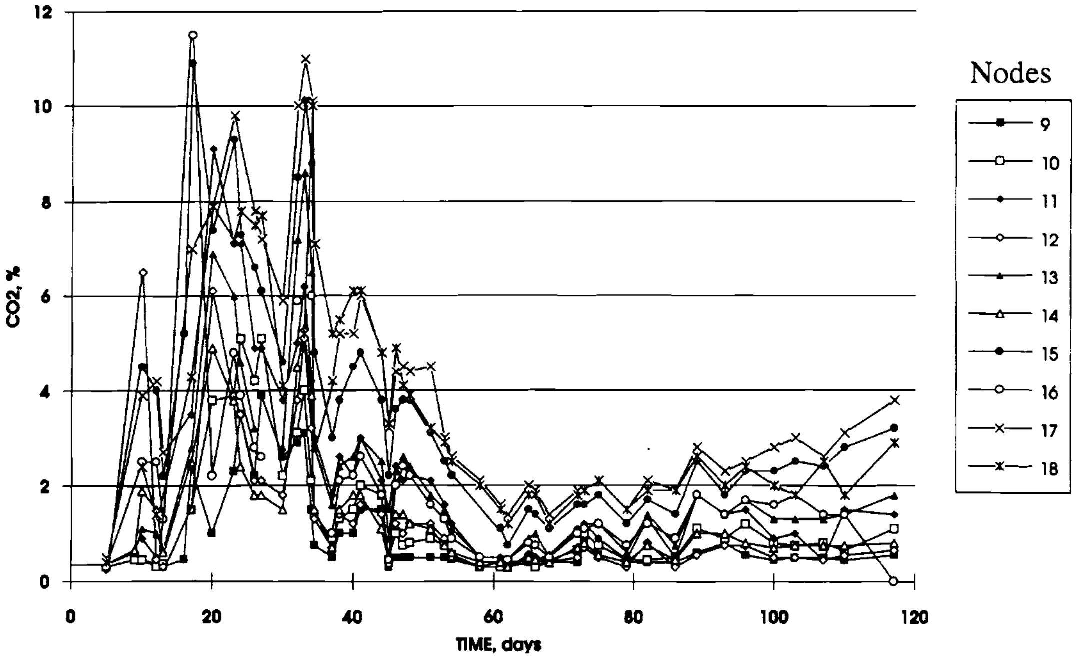

Figure 75. CO_2 Profiles at the 1 ft SP Elevation, Center Cross Section

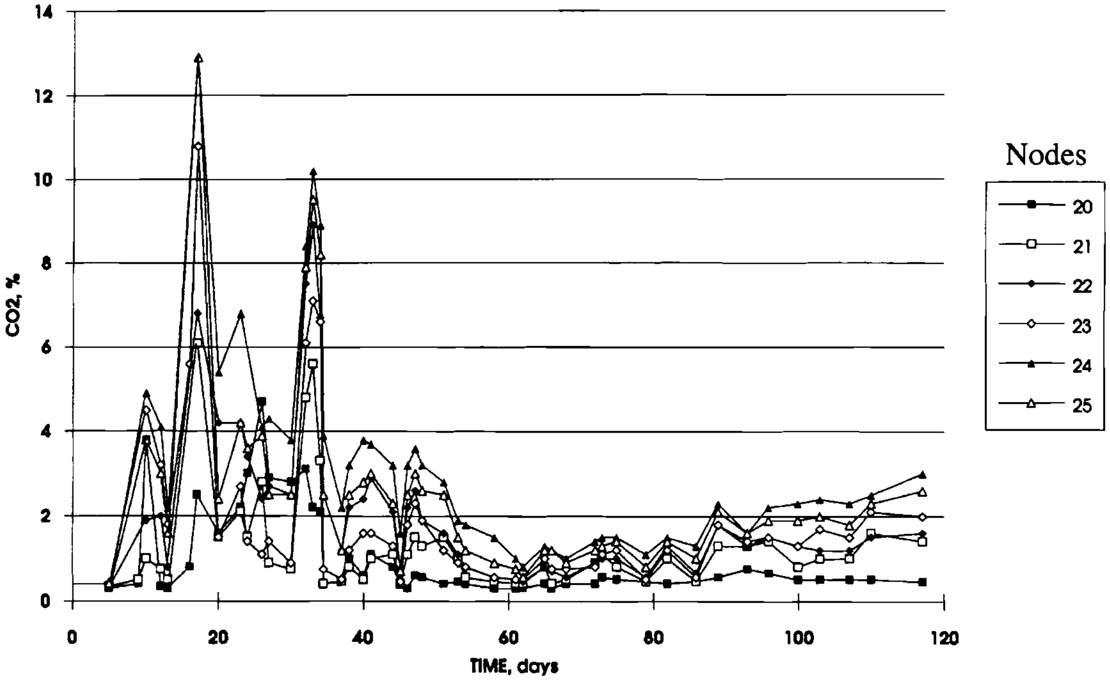

Figure 76. CO_2 Profiles at the 2 ft SP Elevation, Center Cross Section

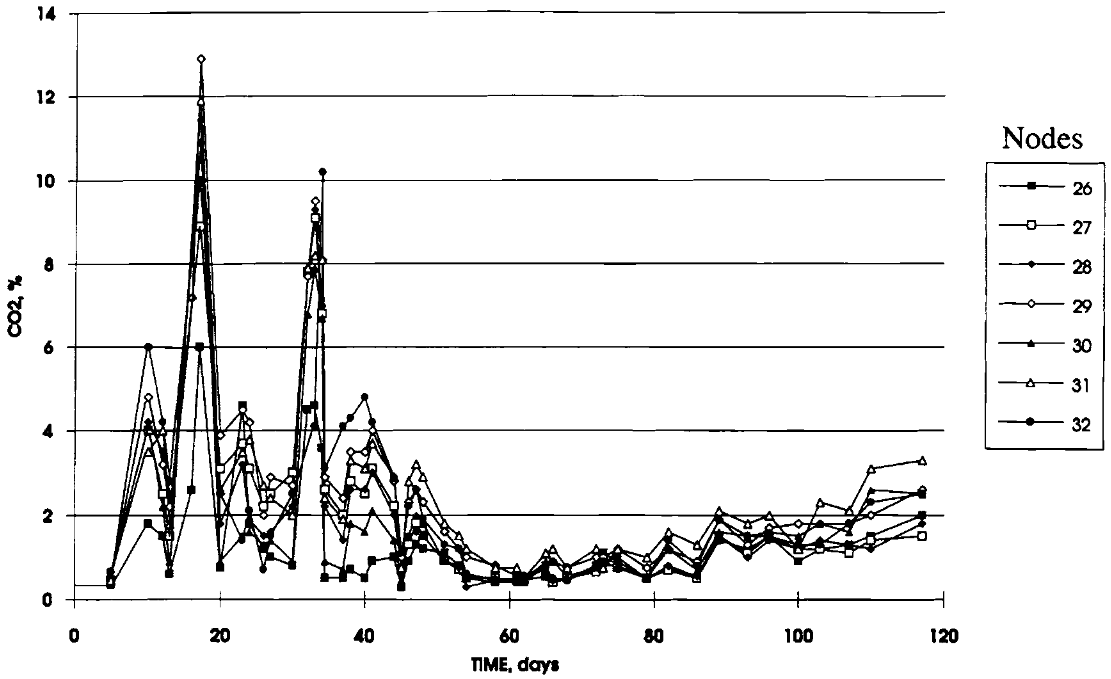

Figure 77. CO_2 Profiles at the 3 ft SP Elevation, Center Cross Section

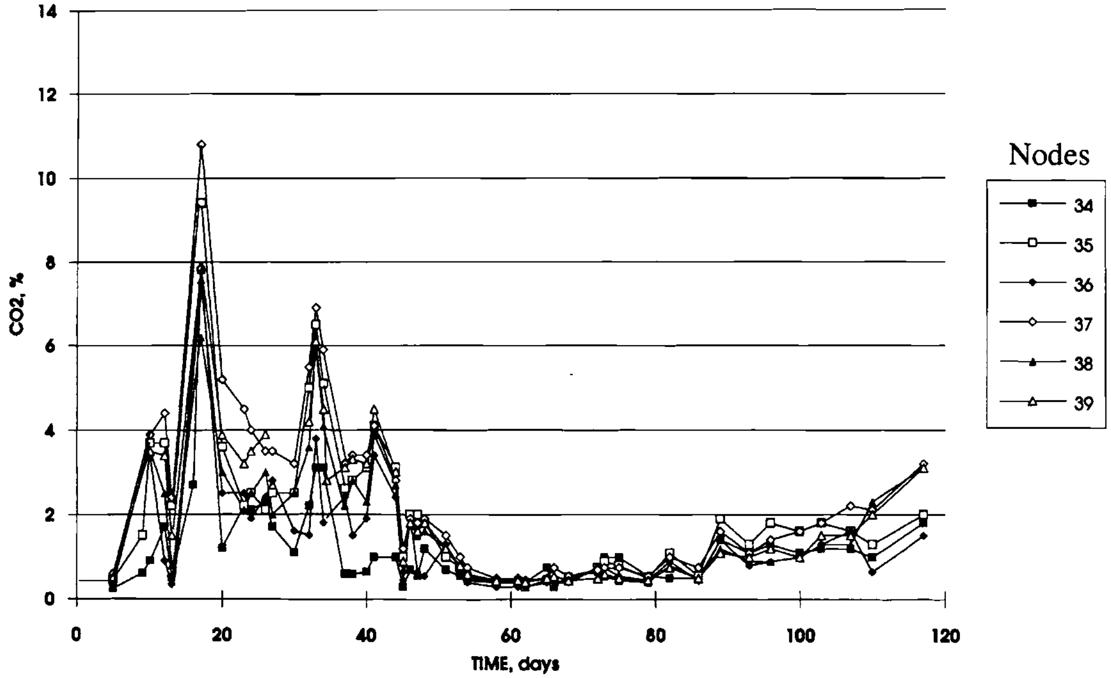

Figure 78. CO_2 Profiles at the 4 ft SP Elevation, Center Cross Section

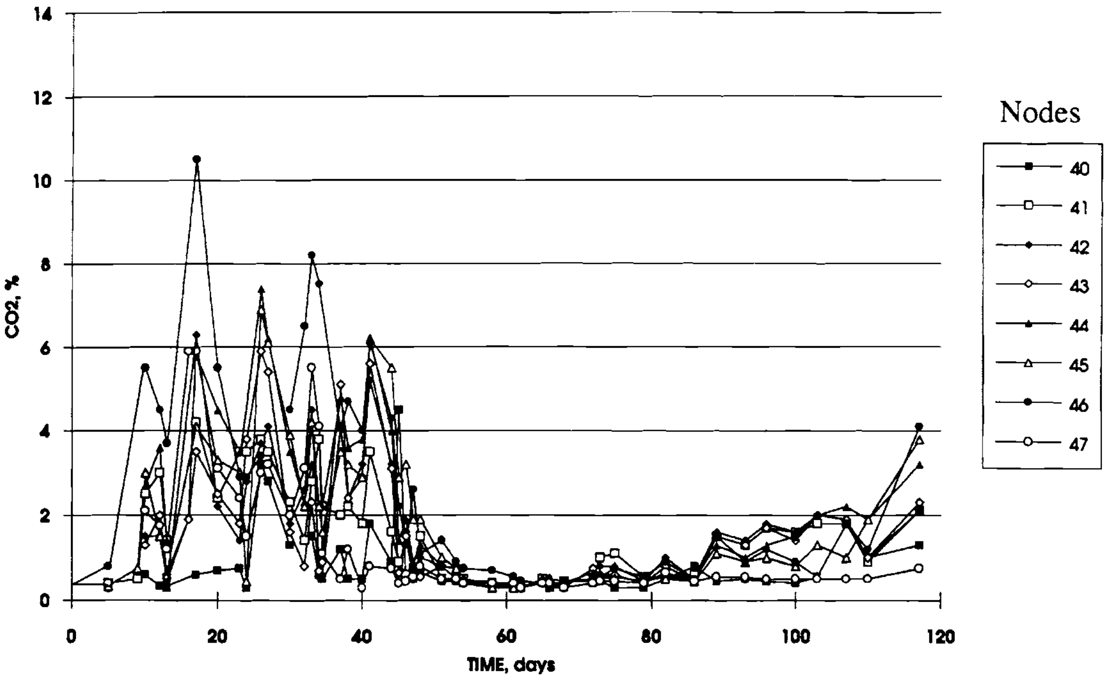

Figure 79. CO_2 Profiles at the 5 ft SP Elevation, Center Cross Section

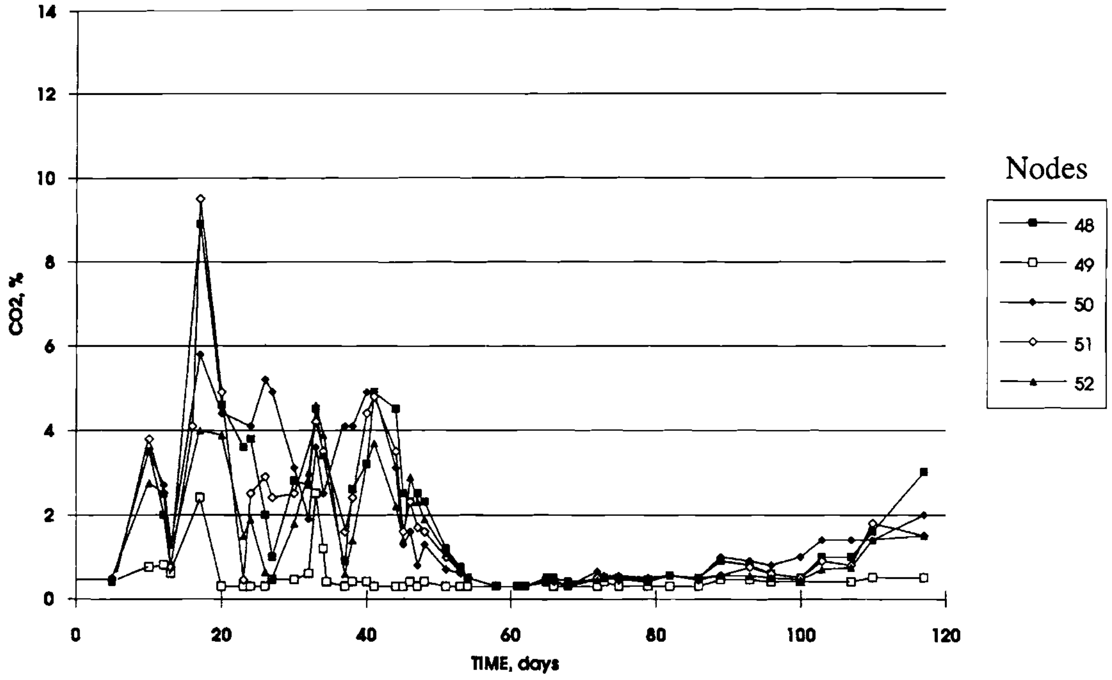

Figure 80. CO_2 Profiles at the 6 ft SP Elevation, Center Cross Section

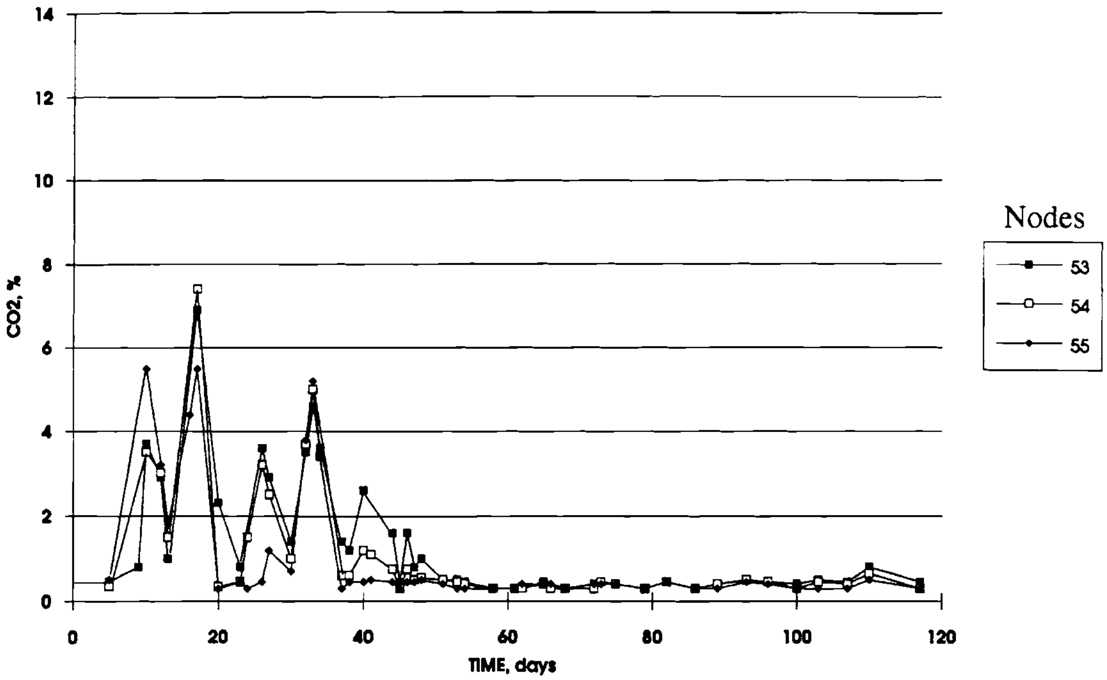

Figure 81. CO_2 Profiles at the 6.5 ft SP Elevation, Center Cross Section

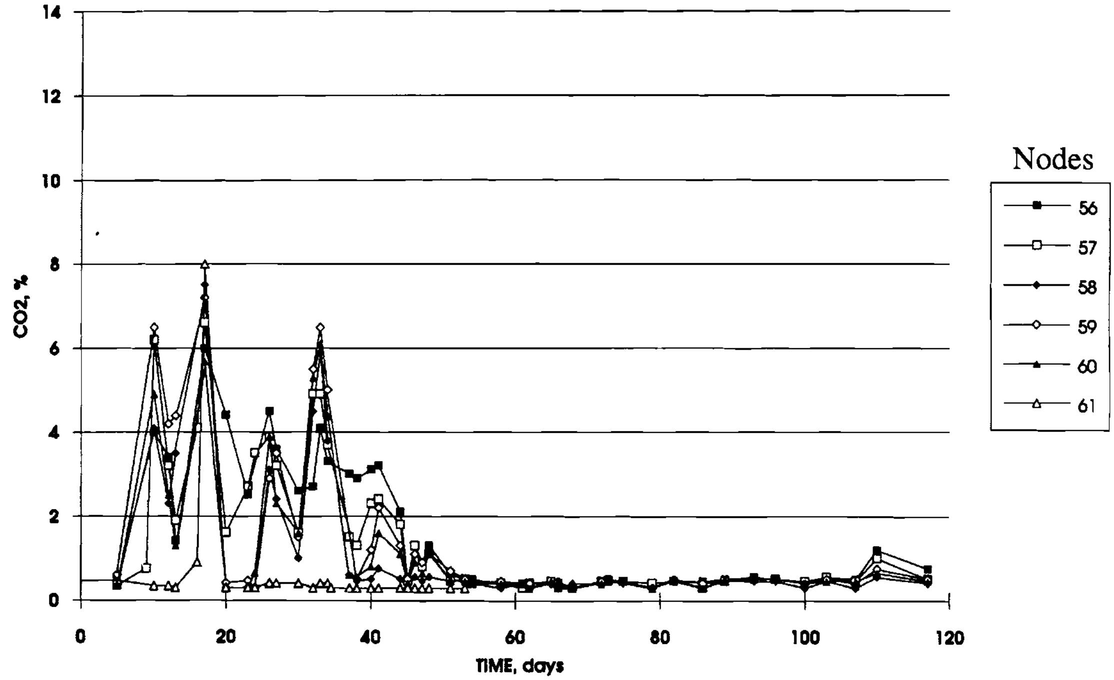

Figure 82. CO_2 Profiles at the 7 ft and Above Elevations, Center Cross Section

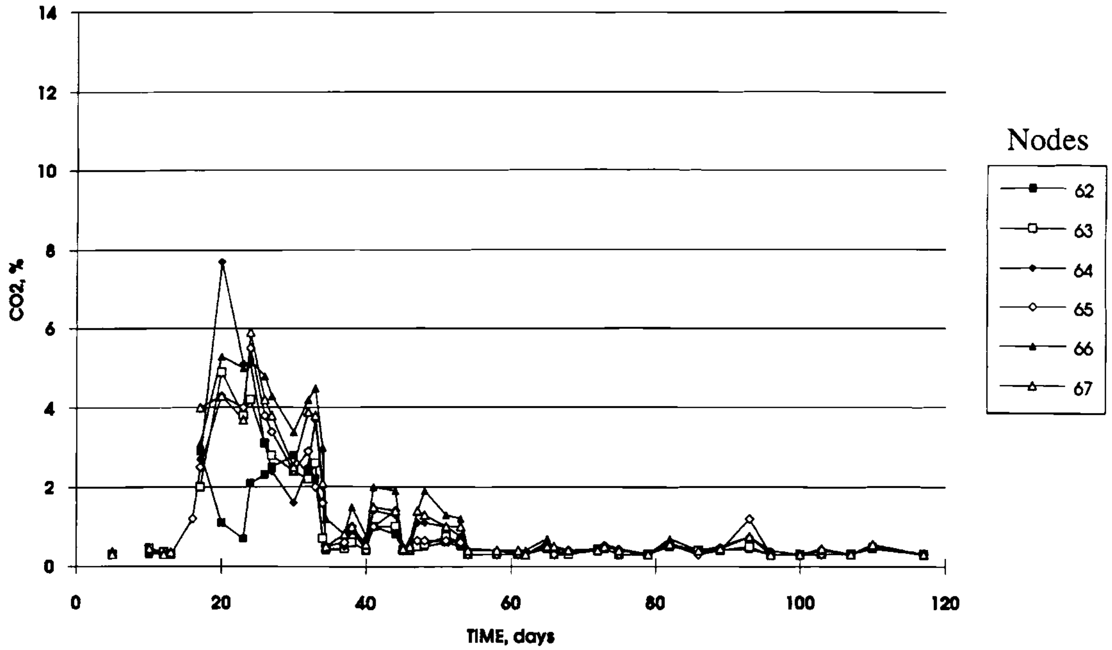

Figure 83. CO_2 Profiles at the 1 ft SP Elevation, End Cross Section

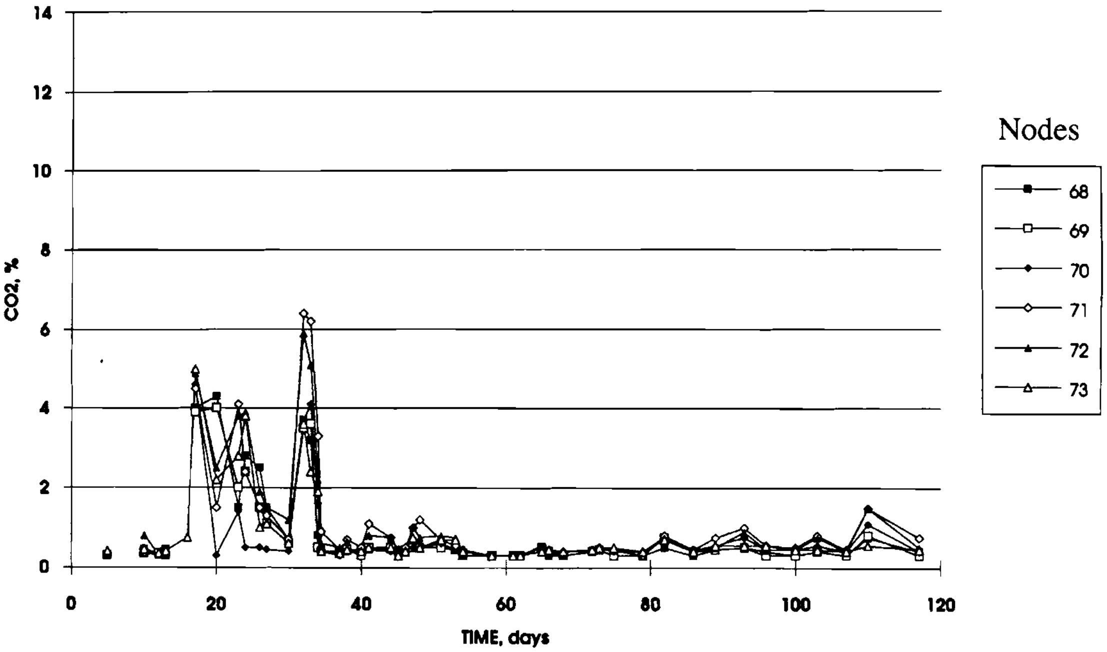

Figure 84. CO_2 Profiles at the 3 ft SP Elevation, End Cross Section

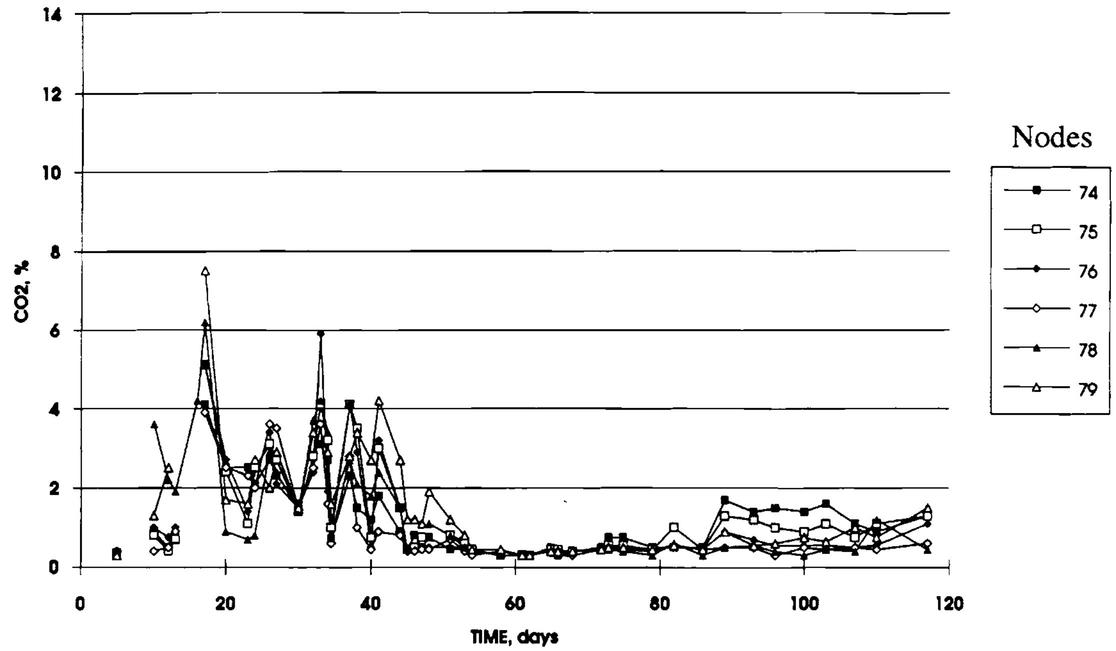

Figure 85. CO_2 Profiles at the 5 and 7 ft SP Elevation, End Cross Section

APPENDIX F

VOC GAS PROFILES IN THE STATIC PILE

The following Figures 86-97 show the VOC response at the sampling node locations shown in Figures 36 and Figures 37. These profiles correspond to the O_2 profiles in Figures 38-49 and the CO_2 profiles in Figures 74-85.

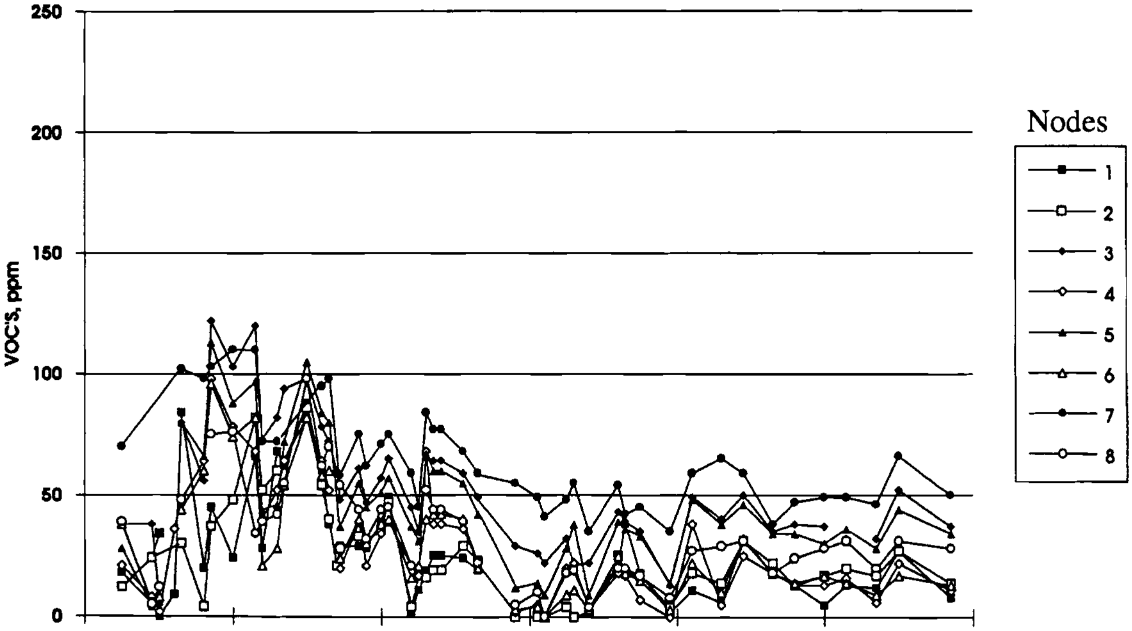

Figure 86. VOC Profiles at the 0.5 ft SP Elevation, Center Cross Section

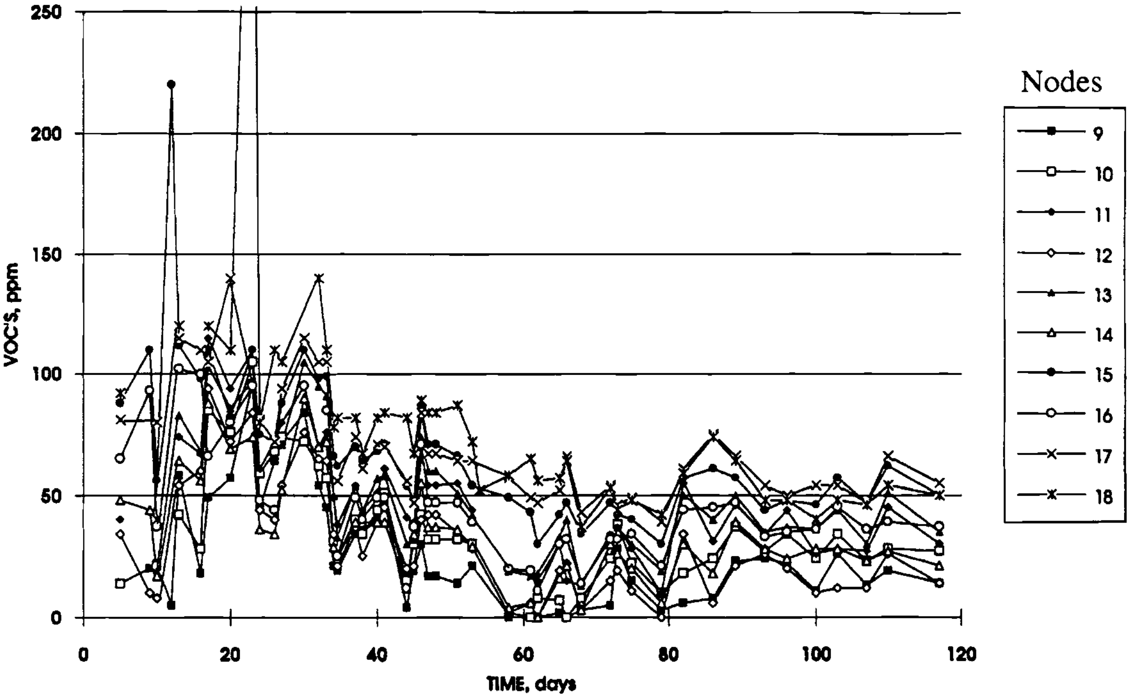

Figure 87. VOC Profiles at the 1 ft SP Elevation, Center Cross Section

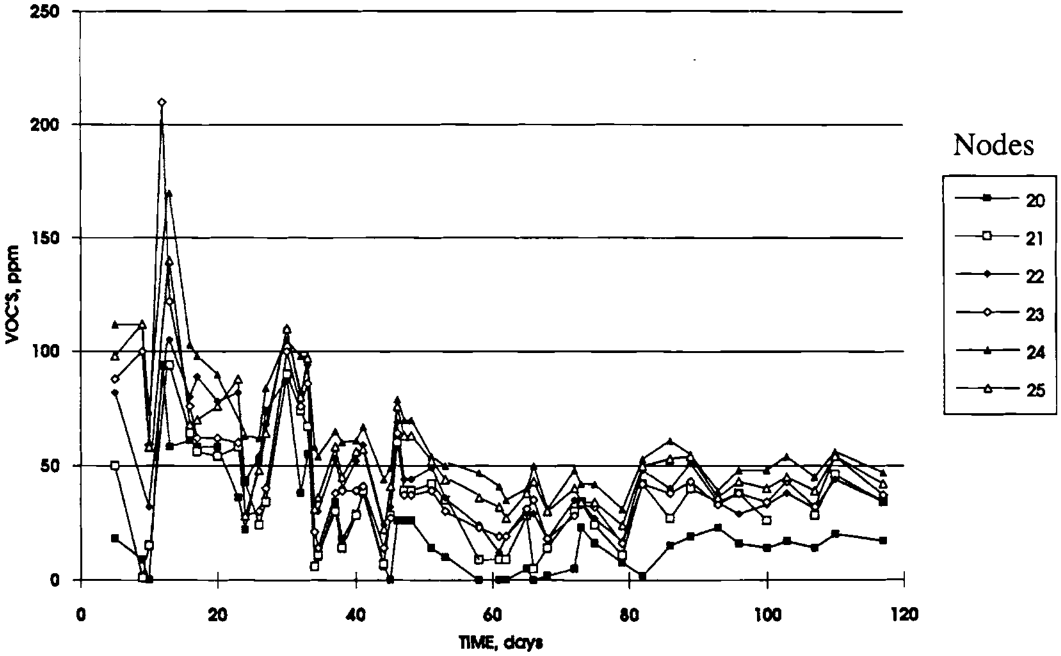

Figure 88. VOC Profiles at the 2 ft SP Elevation, Center Cross Section

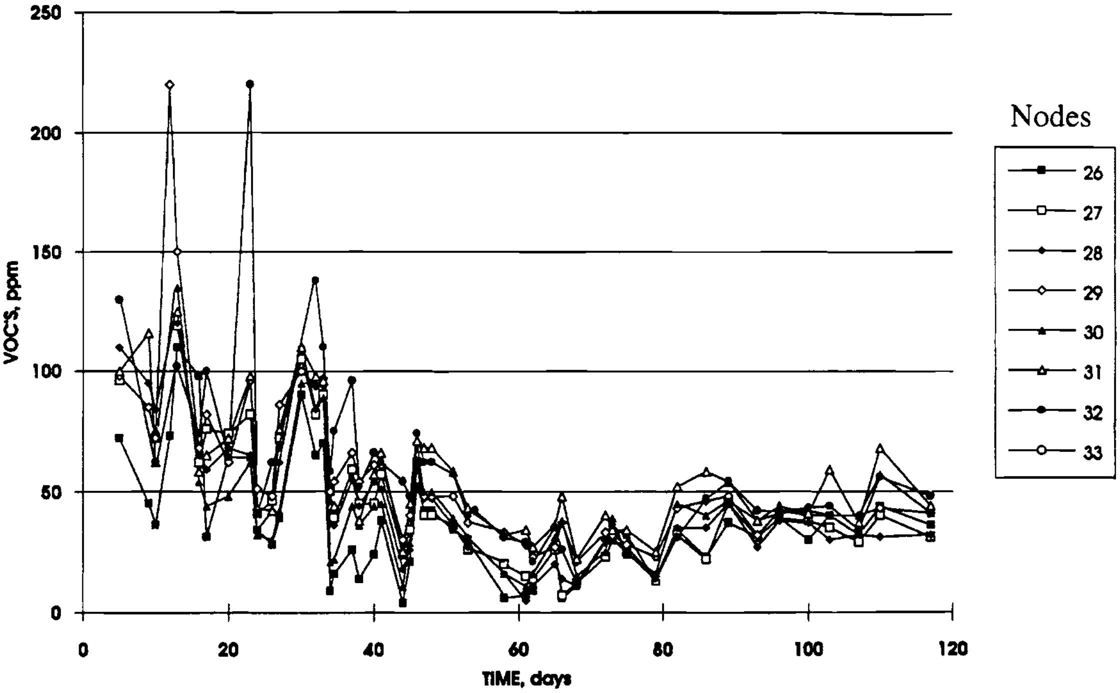

Figure 89. VOC Profiles at the 3 ft SP Elevation, Center Cross Section

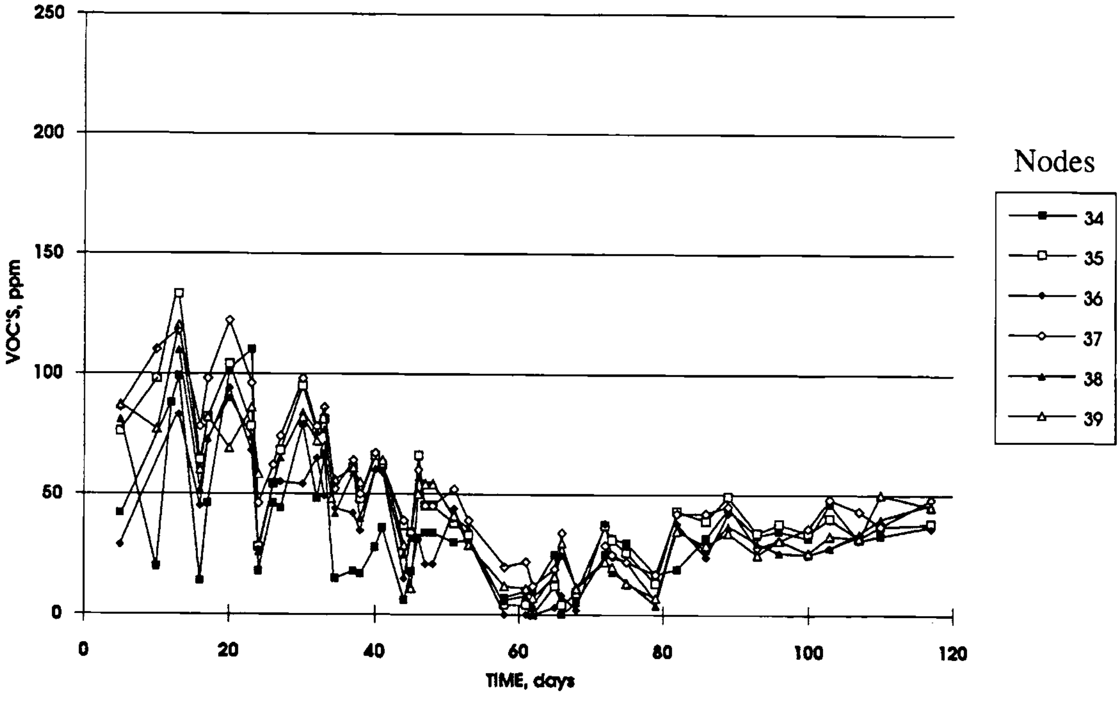

Figure 90. VOC Profiles at the 4 ft SP Elevation, Center Cross Section

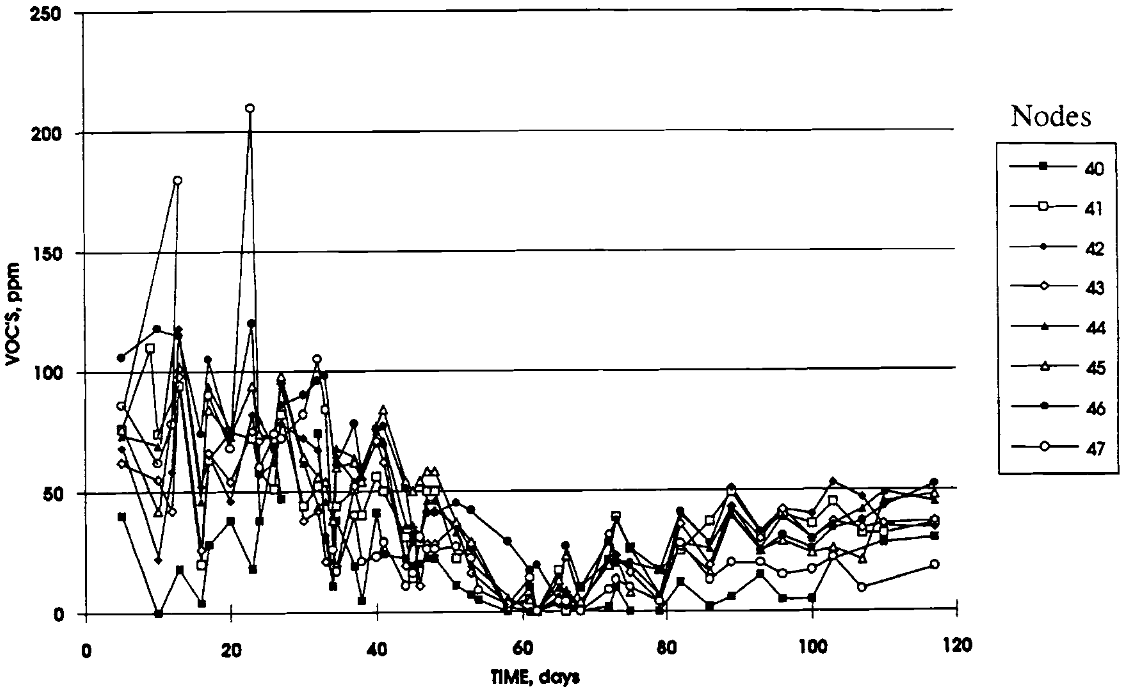

Figure 91. VOC Profiles a the 5 ft SP Elevation, Center Cross Section

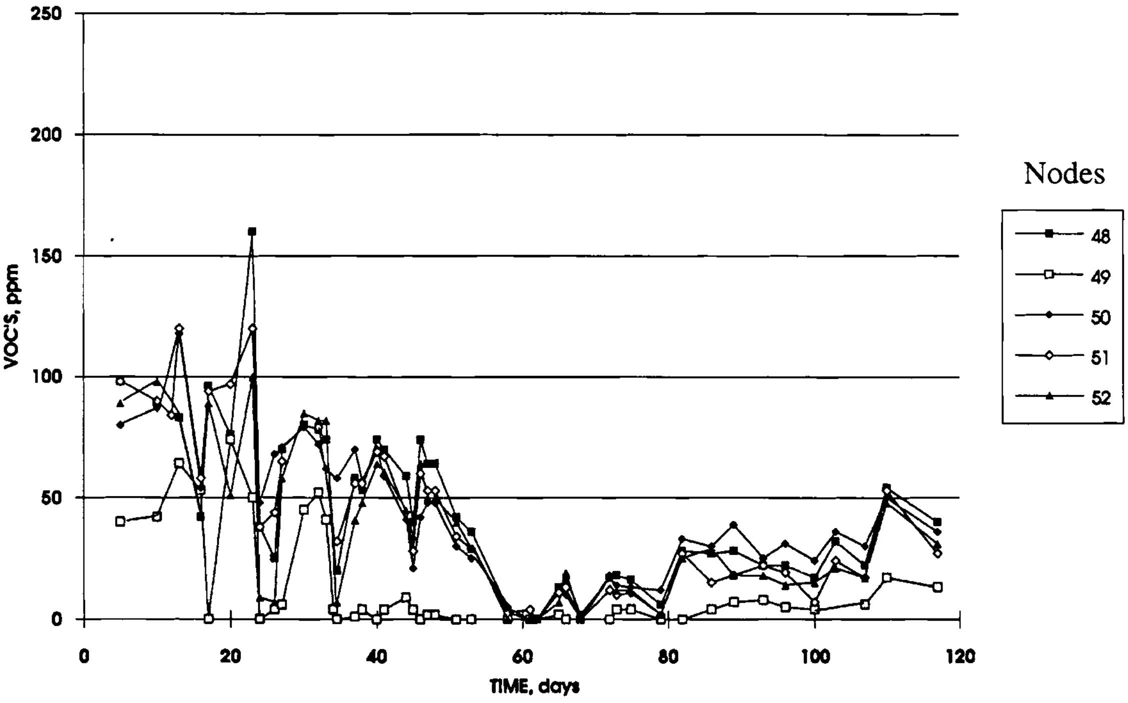

Figure 92. VOC Profiles at the 6 ft SP Elevation, Center Cross Section

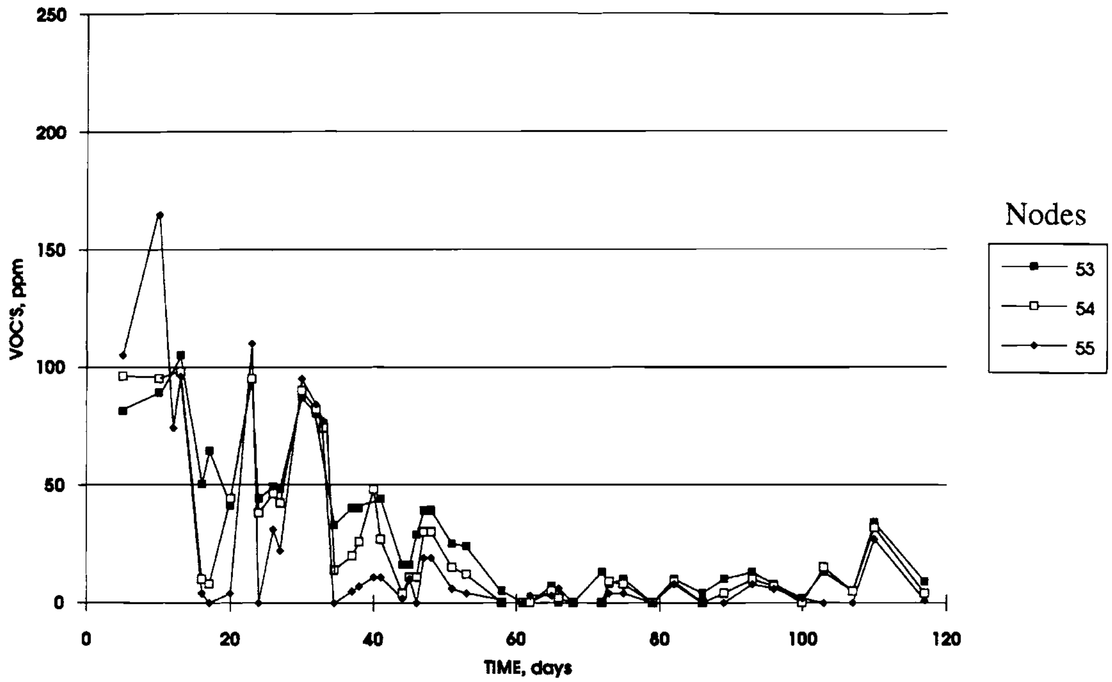

Figure 93. VOC Profiles at the 6.5 ft SP Elevation, Center Cross Section

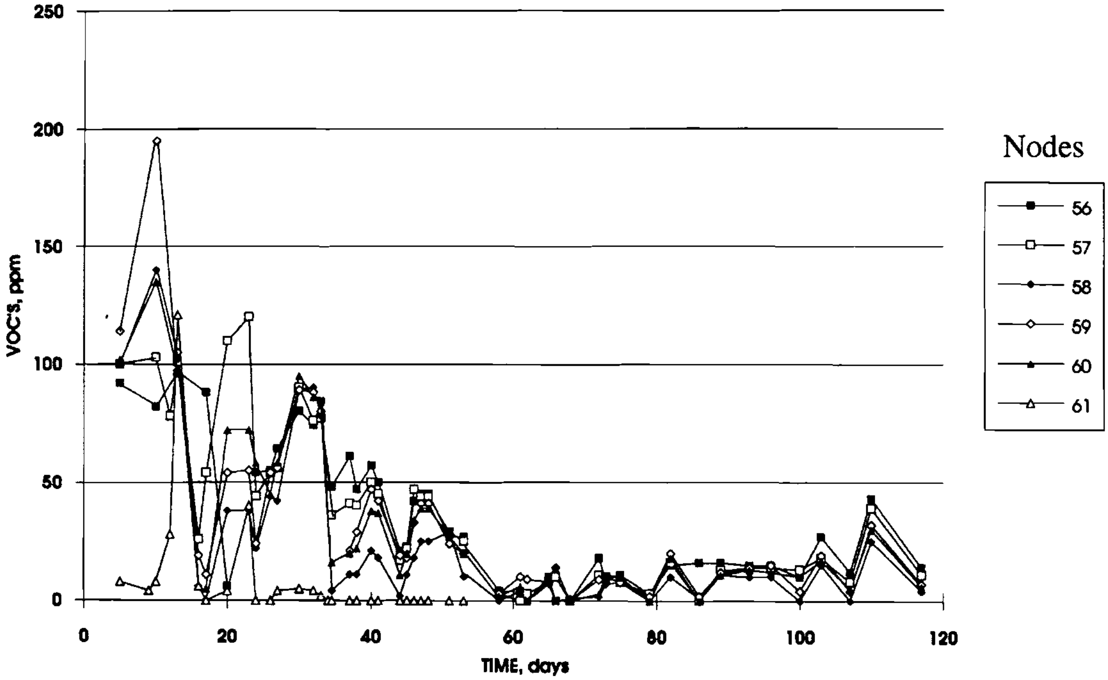

Figure 94. VOC Profiles at the 7 ft and Above SP Elevation, Center Cross Section

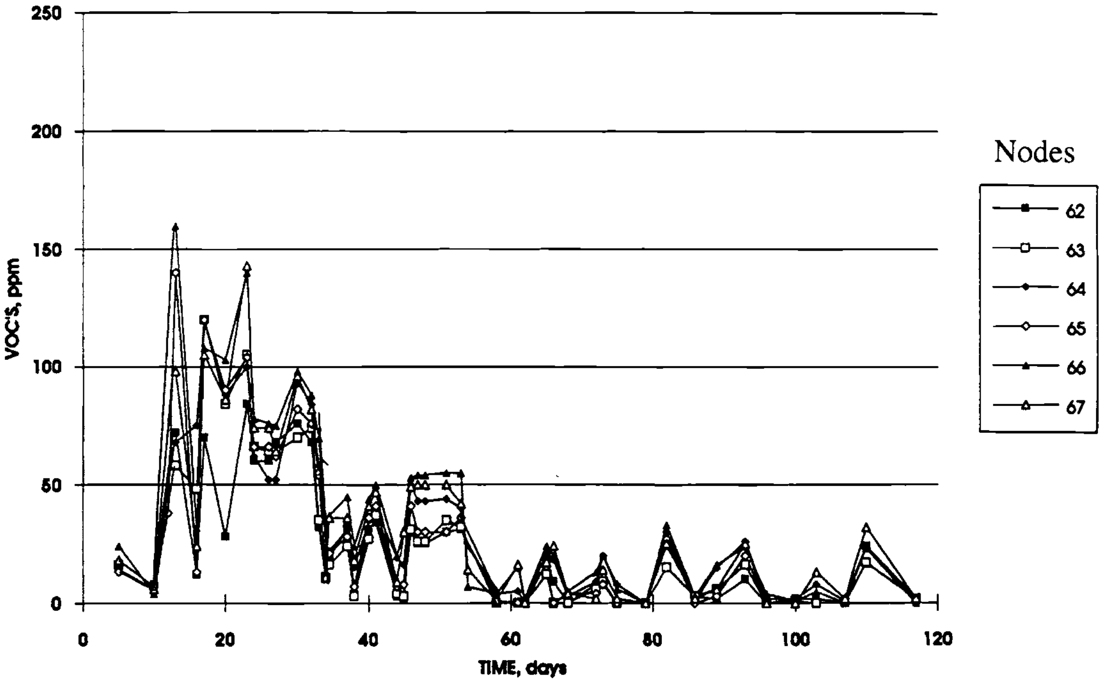

Figure 95. VOC Profiles at the 1 ft SP Elevation, End Cross Section

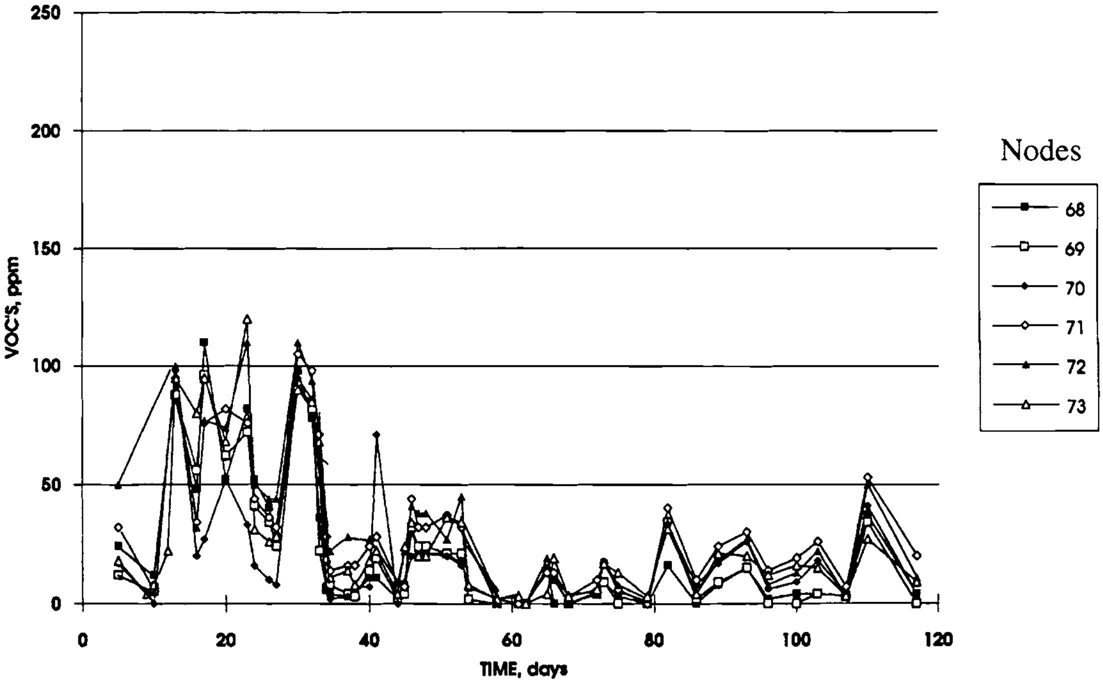

Figure 96. VOC Profiles at the 3 ft SP Elevation, End Cross Section

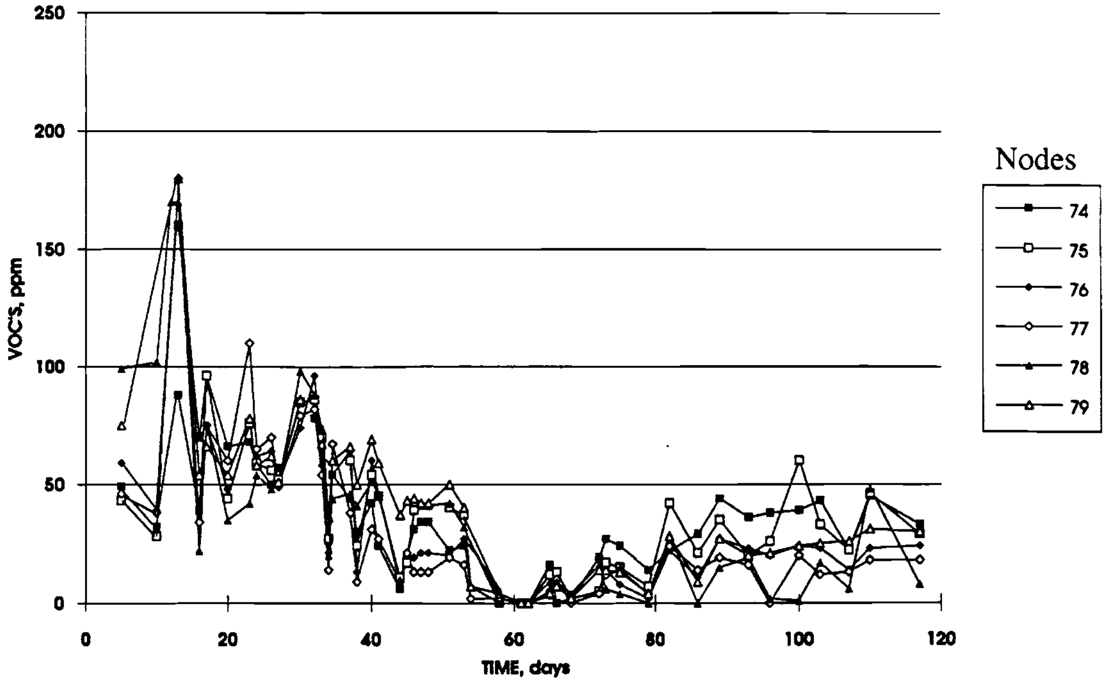

Figure 97. VOC Profiles at the 5 and 7 ft SP Elevations, End Cross Section

APPENDIX G

O&G SAMPLE DATA AND ANALYSIS METHOD

The following table is a compilation of all of the moisture and O&G analyses of the compost materials and samples. Samples marked with asterisks were not included in the average O&G calculation if they were not analyzed or their O&G values were more than 2 standard deviations from the mean value for their respective group. The table is followed by a detailed description of the O&G method used.

TABLE XXI

COMPOST OIL AND GREASE SUMMARY

1 **SAMPLE ID**	2 **% MC**	10 **% O&G, dry**	**AVG. O&G, dry**	**O&G SD**	**AVG. %MC**
12 Jan Soil Feedstock #1	13.31	3.77	**3.7660**	**0.0587**	**13.71**
** 12 Jan Soil Feedstock #1	13.31	**3.30**			
12 Jan Soil Feedstock #1	13.31	3.74			
12 Jan Soil Feedstock #1	13.31	3.78			
1/12 Soil Feedstock #1	13.78	3.90			
1/12 Soil Feedstock #2	13.93	3.75			
1/12 Soil Feedstock #3	14.74	3.84			
1/12 Soil Feedstock #4	13.91	3.71			
1/12 Soil Feedstock #5	13.97	3.71			
1/12 Soil Feedstock #6	12.81	3.68			
1/12 Soil Feedstock #7	13.48	3.74			
1/12 Soil Feedstock #8	14.67	3.79			
Compost Stock 1/29 #1	44.01	3.73	**3.4650**	**0.4066**	**39.44**
Compost Stock 1/29 #2	24.16	3.14			
Compost Stock 1/29 #3	51.66	4.44			
Compost Stock 1/29 #4	52.66	3.52			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
Compost Stock 1/29 #5	23.35	3.25			
Compost Stock 1/29 #6	43.87	3.04			
Compost Stock 1/29 #7	51.28	4.11			
Compost Stock 1/29 #8	29.43	3.19			
Compost Stock 1/29 #9	47.59	3.51			
Compost Stock 1/29 #10	44.15	3.48			
Compost Stock 1/29 #11	41.28	3.17			
Compost Stock 1/29 #12	28.26	3.57			
Compost Stock 1/29 #13	35.22	2.86			
Compost Stock 1/29 #14	35.22	3.47			
11 Feb SP Cmpst #1	35.31	3.14	**3.1650**	**0.1215**	**35.41**
11 Feb SP Cmpst #2	39.38	3.09			
11 Feb SP Cmpst #3	35.54	3.24			
11 Feb SP Cmpst #4	34.09	3.01			
11 Feb SP Cmpst #5	38.96	3.26			
11 Feb SP Cmpst #6	41.07	3.23			
11 Feb SP Cmpst #7	28.29	2.90			
11 Feb SP Cmpst #8	35.30	3.16			
11 Feb SP Cmpst #9	31.49	3.33			
11 Feb SP Cmpst #10	39.07	3.18			
** 11 Feb SP Cmpst #11	23.38	**2.28**			
11 Feb SP Cmpst #12	30.96	3.29			
2/17 SP #1	27.73	3.07	**3.2170**	**0.160**	**33.53**
2/17 SP #2	39.79	3.39			
2/17 SP #3	38.55	3.11			
** 2/17 SP #4	38.23	**3.80**			
** 2/17 SP #5	29.08	**3.65**			
2/17 SP #6	36.79	3.52			
2/17 SP #7	28.5	3.17			
2/17 SP #8	33.17	3.34			
2/17 SP #9	40.19	3.21			
2/17 SP #10	23.74	3.09			
2/17 SP #11	36.76	2.96			
2/17 SP #12	29.77	3.29			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
** 2/22 SP #1			**3.4463**	**0.404**	**33.69**
2/22 SP #2	35.26	3.35			
2/22 SP #3	39.95	3.85			
2/22 SP #4	25.41	3.50			
** 2/22 SP #5					
2/22 SP #6	36.99	3.74			
2/22 SP #7	37.77	2.72			
** 2/22 SP #8					
2/22 SP #9	30.14	3.39			
** 2/22 SP #10					
2/22 SP #11	37.77	4.01			
2/22 SP #13	26.25	3.01			
2/25 SP CMPST #1	29.37	3.60	**3.1023**	**0.3247**	**26.39**
2/25 SP CMPST #2	32.5	3.48			
2/25 SP CMPST #3	29.49	3.24			
2/25 SP CMPST #4	37.85	3.13			
2/25 SP CMPST #5	31.27	3.26			
2/25 SP CMPST #6	28.73	3.25			
2/25 SP CMPST #7	20.36	3.01			
2/25 SP CMPST #8	22.71	2.95			
2/25 SP CMPST #9	13.03	2.59			
2/25 SP CMPST #10	33.64	2.92			
2/25 SP CMPST #11	9.08	2.45			
2/25 SP CMPST #12	28.64	3.34			
3/1 SP CMPST #1	27.87	3.00	**3.2063**	**0.238**	**27.27**
3/1 SP CMPST #2	25.77	2.72			
3/1 SP CMPST #3	26.25	3.29			
3/1 SP CMPST #4	32.24	3.38			
3/1 SP CMPST #5	27.26	2.89			
3/1 SP CMPST #6	26.34	3.33			
** 3/1 SP CMPST #7	31.13	**3.94**			
3/1 SP CMPST #8	24.03	3.34			
3/1 SP CMPST #9	21.42	3.50			
3/1 SP CMPST #10	26.21	3.08			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
3/1 SP CMPST #11	26.67	3.29			
3/1 SP CMPST #12	32	3.45			
3/4 SP #1	24.66	3.11	**3.2045**	**0.523**	**22.75**
3/4 SP #2	31.75	3.34			
3/4 SP #3	28.11	3.62			
3/4 SP #4	34.18	3.01			
3/4 SP #5	32.46	4.49			
3/4 SP #6	21.64	3.29			
3/4 SP #7	9.34	2.40			
3/4 SP #8	20.6	3.19			
3/4 SP #9	12.8	2.90			
3/4 SP #10	9.31	3.55			
3/4 SP #11	23.44	2.49			
3/4 SP #12	24.75	3.07			
** 3/11 SP #1	22.09	**2.12**	**3.1917**	**0.346**	**25.94**
3/11 SP #2	29.08	3.41			
** 3/11 SP #3	31.48	**1.82**			
3/11 SP #4	26.57	3.60			
3/11 SP #5	23.44	3.02			
3/11 SP #6	22.41	3.58			
3/11 SP #7	23	2.60			
3/11 SP #8	12.71	2.65			
3/11 SP #9	23.55	3.38			
3/11 SP #10	31.61	2.94			
3/11 SP #11	23.35	3.33			
3/11 SP #12	41.96	3.41			
18 Mar SP Cmpst #1	25.99	2.88	**3.2224**	**0.1517**	**29.15**
18 Mar SP Cmpst #2	30.02	3.09			
18 Mar SP Cmpst #3	33.97	3.04			
18 Mar SP Cmpst #4	33.55	3.38			
18 Mar SP Cmpst #5	25.04	3.32			
18 Mar SP Cmpst #6	27.11	3.31			
18 Mar SP Cmpst #7	38.00	3.19			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
18 Mar SP Cmpst #8	29.28	3.37			
18 Mar SP Cmpst #9	28.25	3.39			
18 Mar SP Cmpst #10	21.77	3.33			
18 Mar SP Cmpst #11	16.68	3.22			
18 Mar SP Cmpst #12	40.17	3.15			
4/4 SP #1	33.9	3.40	**3.2287**	**0.304**	**23.35**
4/4 SP #2	26.45	2.83			
4/4 SP #3	23.49	2.60			
4/4 SP #4	25.12	3.32			
4/4 SP #5	24.24	3.45			
4/4 SP #6	29.67	3.60			
4/4 SP #7	24.2	3.36			
4/4 SP #8	22.48	3.43			
4/4 SP #9	18.27	2.80			
4/4 SP #10	12.69	3.31			
4/4 SP #11	21.24	3.50			
4/4 SP #12	18.48	3.15			
4/22 SP #1**	25.92	9.62	**3.3215**	**0.142**	**23.12**
4/22 SP #2	25.61	3.27			
4/22 SP #3	27.61	3.32			
4/22 SP #4	25.16	3.07			
4/22 SP #5	24.08	3.31			
4/22 SP #6	23.02	3.30			
4/22 SP #7	28.05	3.18			
4/22 SP #8	25.25	3.22			
4/22 SP #9	24.13	3.31			
4/22 SP #10	11.42	3.58			
4/22 SP #11	16.74	3.49			
4/22 SP #12	20.48	3.49			
5/13 SP #1	29.23	3.25	**3.0996**	**0.255**	**26.39**
5/13 SP #2	26.74	3.02			
5/13 SP #3	30.36	3.18			
5/13 SP #4	23.97	3.19			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
** 5/13 SP #5	20.18	**2.10**			
5/13 SP #6	30.01	3.16			
5/13 SP #7	29.83	3.48			
5/13 SP #8	30.53	2.87			
5/13 SP #9	21.63	2.49			
5/13 SP #10	28.47	3.41			
5/13 SP #11	20.94	3.00			
5/13 SP #12	24.84	3.05			
6/6 SP #1	22.04	3.14	**3.2791**	**0.120**	**21.68**
6/6 SP #2	23.28	3.18			
6/6 SP #3	16.74	3.31			
6/6 SP #4	20.94	3.26			
6/6 SP #5	23.58	3.43			
6/6 SP #6	23.05	3.08			
6/6 SP #7	23.64	3.48			
6/6 SP #8	26.59	3.33			
6/6 SP #9	28.1	3.38			
6/6 SP #10	25.6	3.32			
6/6 SP #11	8.61	3.13			
6/6 SP #12	17.97	3.30			
2/11 Windrow #1	35.08	3.50	**3.3466**	**0.2089**	**34.08**
2/11 Windrow #2	35.35	3.55			
2/11 Windrow #3	35.46	3.02			
2/11 Windrow #4	30.42	3.31			
** 2/11 Windrow #5 (surface)	7.00	**1.97**			
2/17 WR #1A	36.65	3.40	**3.3350**	**0.249**	**34.16**
2/17 WR #1B	34.23	2.98			
2/17 WR #2A	33.17	3.24			
2/17 WR #2B	34.32	3.69			
2/17 WR #3A	26.33	3.08			
2/17 WR #3B	29.29	3.12			
2/17 WR #4A	39.17	3.57			
2/17 WR #4B	40.08	3.59			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
2/25 WR #1	27.45	2.86	**3.1357**	**0.3199**	**26.78**
2/25 WR #2	29.53	3.30			
2/25 WR #3	26.77	3.35			
2/25 WR #4	27.42	3.33			
2/25 WR #5	31.27	3.62			
2/25 WR #6	28.73	2.61			
2/25 WR #7	20.36	2.81			
2/25 WR #8	22.71	3.21			
3/4 WR #1	33.41	2.83	**3.1502**	**0.242**	**29.90**
3/4 WR #2	23.86	3.26			
3/4 WR #3	30.79	3.10			
3/4 WR #4	34.57	3.24			
3/4 WR #5	25.84	2.73			
3/4 WR #6	26.33	3.51			
3/4 WR #7	31.13	3.32			
3/4 WR #8	33.24	3.21			
3/10 WR #1	29.26	3.17	**3.0817**	**0.075**	**26.74**
3/10 WR #2	28.48	3.14			
3/10 WR #3	21.67	3.08			
3/10 WR #4	22.39	3.15			
3/10 WR #5	29.54	2.97			
3/10 WR #6	31.43	3.10			
3/10 WR #7	30.03	2.97			
** 3/10 WR #8	21.08	**3.49**			
WR 3/17 SE #1	28.43	2.98	**3.0079**	**0.2256**	**23.93**
WR 3/17 SE #2	23.58	2.71			
WR 3/17 NW #3	25.06	2.89			
WR 3/17 NW #4	20.07	3.21			
WR 3/17 SW #5	21.89	3.36			
WR 3/17 SW #6	21.26	3.10			
WR 3/17 SW #7	26.94	2.67			
WR 3/17 SW #8	24.22	3.15			

TABLE XXI (Continued)

1 SAMPLE ID	2 % MC	10 % O&G, dry	AVG. O&G, dry	O&G SD	AVG. %MC
3/24 WR #1	20.99	3.04	**3.0269**	**0.161**	**22.00**
3/24 WR #2	22.91	2.86			
3/24 WR #3	26.41	2.99			
3/24 WR #4	18.84	3.19			
** 3/24 WR #5	21.97	**-6.06**			
3/24 WR #6	20.39	3.31			
3/24 WR #7	24.02	2.96			
3/24 WR #8	20.49	2.83			
3/31 WR #1	20.11	3.07	**3.1333**	**0.118**	**20.66**
3/31 WR #2	23.58	3.38			
3/31 WR #3	22.55	3.21			
3/31 WR #4	22.15	3.05			
3/31 WR #5	23.35	3.09			
3/31 WR #6	19.17	3.22			
3/31 WR #7	13.97	3.04			
3/31 WR #8	20.39	3.01			
4/7 WR #1	26.51	2.96	**2.9560**	**0.135**	**23.97**
4/7 WR #2	26.27	2.96			
4/7 WR #3	25.77	2.80			
4/7 WR #4	30.73	3.25			
4/7 WR #5	21.28	2.94			
4/7 WR #6	22.46	3.03			
4/7 WR #7	16.68	2.78			
4/7 WR #8	22.05	2.94			
4/22 WR #1	22.59	3.20	**3.0241**	**0.196**	**23.27**
4/22 WR #2	19.97	3.00			
4/22 WR #3	22.63	2.99			
4/22 WR #4	17.27	2.92			
4/22 WR #5	21.44	2.92			
4/22 WR #6	30.83	3.31			
4/22 WR #7	28.05	3.20			
4/22 WR #8	23.39	2.65			

TABLE XXI (Continued)

1 **SAMPLE ID**	2 **% MC**	10 **% O&G, dry**	**AVG. O&G, dry**	**O&G SD**	**AVG. %MC**
5/13 WR #1	26.48	2.49	**2.7656**	**0.327**	**25.14**
5/13 WR #2	25.17	2.56			
5/13 WR #3	21.73	3.23			
5/13 WR #4	31.42	2.89			
5/13 WR #5	23.98	2.91			
5/13 WR #6	23.5	2.13			
5/13 WR #7	25.27	3.02			
5/13 WR #8	23.54	2.90			
6/6 WR #1	27.05	3.18	**2.9407**	**0.105**	**22.63**
6/6 WR #2	27.8	2.90			
6/6 WR #3	22.44	2.95			
6/6 WR #4	19.99	2.86			
6/6 WR #5	21.07	2.92			
6/6 WR #6	16.18	2.90			
6/6 WR #7	19.8	2.81			
6/6 WR #8	26.73	3.00			
Straw #1	8.43	0.92	**0.9797**	**0.044**	**8.43**
Straw #2	8.43	1.00			
Straw #3	8.43	1.02			
Background Soil #1	2.53	0.010	**0.0094**	**0.002**	**2.53**
Background Soil #2	2.53	0.007			
Background Soil #3	2.53	0.011			

**** VALUES NOT INCLUDED IN O&G AVGS AND SD**

EXTRACTION PROCEDURE FOR GRAVIMETRIC OIL AND GREASE DETERMINATION OF OILY SOLIDS

The following procedure is a modification of U.S. EPA Method 413.1 (1979a) and was used to extract oil and greases from a solid and quantifies the content by gravimetric determination. Extracts were redissolved in solvent for performing GC "type analyses" to obtain a qualitative picture of hydrocarbon distributions in the compost materials and samples. The method uses Soxtec extractors instead of separatory funnels, methylene chloride instead of freon as solvent, does not acidify samples, and uses anhydrous magnesium sulfate to sequester free water in the sample to eliminate water effects on extraction efficiency. The method closely resembles that investigated by Martin et al. (1990) which is a modification of U.S. EPA Method 9071 in SW-846 (1986).

1. Prior to running the following extraction, obtain the moisture content of the soil to be extracted. This data is used to determine the dry weight of solids in the final calculations.
2. Weight out a sample of sludge or compost. Record.
3. Weight an equivalent or slightly greater amount of anhydrous magnesium sulfate. Record.
4. Place these materials into a mortar and grind with a pestle so that the two are well mixed.
5. Place the mixture into a container and cover or seal.
6. Let the mixture stand overnight.
7. Turn on the power to the Soxtec System HT service unit and set the operating temperature to 105°C. Turn on the water to the Soxtec HT2 1045 Extraction Unit to facilitate reflux of the solvent. Ensure that the air pump is off on the service unit and the air valve is closed on the extraction unit.
8. Select a fresh and dry extraction thimble and label it with a graphite pencil.
9. Place the thimble on a 4 place balance and tare the balance.
10. Place as much of the mixture into the extraction thimble as possible while leaving enough room at the top to plug with glass wool. Record the amount of mixture added to the thimble.

11. Press a metal Soxtec ring into the top of the extraction thimble and plug the top with a piece of glass wool.
12. Place several glass beads in a clean and dry, labeled Soxtec boiling beaker, weigh, and record the total.
13. Fill the boiling beaker about 1/2 full with methylene chloride solvent.
14. Attache the thimble to the extraction carrier.
15. Place the boiling beaker on the heater under the extraction thimble.
16. Lower the thimble into the solvent (boil position).
17. Allow the solvent to come to a boil and leave the thimble in the solvent for 1/2 hour.
18. At the end of 1/2 hour, raise the thimble (rinse position) and let the solvent reflux and rinse the thimble contents for 5-1/2 hours.
19. At the end of the 5-1/2 hours, turn off the heat. Turn on the air pump on the service unit and open the air valve on the extraction unit. Evaporate the solvent from the beaker.
20. When the solvent has evaporated, remove the beaker and thimble from the extraction device. Recover the ring from the thimble and discard the thimble and mixture.
21. Place the beaker in a heated vacuum oven at 40°C for 24 hours or longer to eliminate residual solvents and water.
22. Remove the beaker from the oven and weigh it.
23. Determine the oil and grease percentage of the sample on a dry weight basis as follows:

$$\%\ \text{O\&G, dry wt} = \frac{\text{Extract}}{\text{Dry Extractable Solids}}$$

where, % O&G is on a dry wt basis and the extract and dry extractable solids are in the same units of mass.

The following columns for data entry and manipulation were used in a spreadsheet for performing the O&G calculation:

1. Sample ID
2. % Moisture Content of wet soil
3. % Dry wt = (1 - %MC)
4. Wet Soil wt -- basis for making a large enough sample to be divided into replicates if desired.
5. $MgSO_4$ wt added
6. Mixture wt -- actual amount in thimble to be extracted.
7. Dry Beaker wt -- includes weight of clean glass beads and labeled beaker.
8. Extract and Beaker wt -- dried extract, beaker, and glass beads.
9. Extract wt.
10. % O&G -- calculated using above equation.

The following equation was used to calculate the weight of the dry extractable solids for determining the percent oil and grease in the subsequent equation. The dry extractable solids included the soil and the hydrocarbons in the thimble sample without water or $MgSO_4$. The wet soil wt and the $MgSO_4$ wt were the materials mixed in the mortar (4 and 5). The mixture wt was the mixed sample amount weighed in the thimble (6). The % dry wt was the percent dry extractable solids determined by subtracting the measured moisture content of wet soil prior to mixing (2 and 3).

$$\text{Dry Extractable Solids} = \frac{\text{Wet Soil wt}}{MgSO_4\text{ wt added} + \text{Wet Soil wt}} \bullet \text{Mixture wt} \bullet \%\text{ Dry wt}$$

VITA

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