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Abstract approved

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Ex-situ bioremediation of saturated soil contaminated with pentachlorophenol and 2,3,5,6-TeCP is commonly accomplished by landfarming or by treatment in a bioreactor. Treating saturated, low-permeability soils in bioreactors, without pre-treatment requires a reactor capable of promoting anaerobic and/or aerobic removal of chlorophenols without transferring these contaminants to the aqueous phase. A pilot-scale bioreactor was designed to treat 3.7 cubic meters of contaminated soil with a saturated hydraulic conductivity of 0.12 cm/day. The bioreactor demonstrated significant removal of chlorophenols when soil was infused with a treatment mixture containing imitation vanilla flavoring as an electron donor for reductive dechlorination and primary substrate for aerobic cometabolism. Bench scale studies showed greater overall removal when feed mixtures included an inoculated biomass, or when treatment mixtures were maintained anaerobically prior to use. The combined results of these studies suggest that concentrations of pentachlorophenol and 2,3,5,6-TeCP in soil can be significantly reduced using fill and draw batch reactors, operated for three to five week long cycles, using a variety of treatment mixtures.

Bioremediation of Low-Permeability, Pentachlorophenol-Contaminated Soil by
Laboratory and Full-Scale Processes.

by

Mark B. Havighorst

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Mark B. Havighorst, Author

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Bioremediation of Low-Permeability, Pentachlorophenol-Contaminated Soil by Laboratory and Full-Scale Processes.

Chapter 1

Introduction

For more than fifty years, chlorophenols have been used as industrial and agricultural chemicals throughout the world. Pentachlorophenol (PCP) has been widely used as an herbicide and as a molluscicide, but is employed primarily as a pesticide for wood preservation (Boyd et al., 1989). Its widespread use has made it a common contaminant of soils, sediment and groundwater. In contaminated soils at wood-preserving facilities, PCP has been found at levels as high as several thousand mg/kg (Boyd et al., 1989). Because of its pervasive use and its toxicity, pentachlorophenol has been classified as a priority pollutant by the U.S. Environmental Protection Agency (LaGrega et al., 1994).

Treatment of PCP-contaminated soil has been conducted by *ex-situ* and *in-situ* processes. Because of PCP's relatively low volatility and moderate solubility, most remediation efforts have taken the form of *ex-situ* biological treatment, such as landfarming and composting (Haggblom and Valo, 1995). The use of bioreactors in remediating PCP contaminated soils has been largely limited to laboratory-scale demonstrations. In 1996, the EPA Western Region Hazardous Substance Research Center approved the demonstration and testing of a novel permeable barrier reactor at a wood preserving facility in Eugene, Oregon (Woods and Williamson, 1995). The reactor was designed to bioremediate PCP-contaminated groundwater *in-situ* by sequential anaerobic-aerobic transformation. Testing this new technology required drilling a 24 inch diameter, 25 foot deep well. Generated as a by-product of the well-drilling operation was approximately 3.7 cubic meters of saturated, low permeability soil, contaminated with PCP and 2,3,5,6-tetrachlorophenol (TeCP). The design and implementation of a soil

bioreactor capable of treating the entire volume of contaminated soil was proposed as an option to stock bioremediation techniques or disposing of the soil as hazardous waste.

Biological treatment of PCP is possible under both aerobic and anaerobic soil conditions. Anaerobic degradation of PCP in soils occurs via reductive dechlorination. In this process, chlorines on the aromatic ring are replaced by hydrogens, ultimately resulting in dichlorophenols (DCP) (Mohn and Tiedje, 1992). PCP has been shown to be reduced to TeCP via *ortho*, *meta*, and *para* pathways, depending on the microbial consortia present; but *ortho* dechlorination predominates (Nicholson, 1990). In soils, a common sequence for PCP degradation is via ortho dechlorination to 3,4,5-trichlorophenol (TCP), followed by para dechlorination to 3,5-DCP (Haggblom and Valo, 1995). Dechlorination of PCP often leads to the accumulation of less-chlorinated phenols, but complete mineralization to CO_2, or CH_4 is possible (Haggblom and Valo, 1995).

Under aerobic conditions, PCP transformation pathways depend upon the type of biological population present. Although both *ortho* and *para* hydroxylation of PCP are common in aqueous experiments, bacterial strains transform soil bound PCP exclusively via *para*-hydroxylation to form tetrachloro-*p*-hydroquinone (TeCH) (Apajalahti and Salkinoja-Salonen, 1987). TeCH can then be hydroxylated at the *ortho* position, resulting in trichloro-1,2,4 trihydroxybenzene (THB); and after three reductive dechlorination steps, 1,2,4-trihydroxybenzene (Haggblom and Valo 1995). TeCH can also be dechlorinated without hydroxylation, resulting in trichlorohydroquinone (TCH) and 2,6-dichlorohydroquinone (DCH) (Steiert and Crawford, 1986). Fungal degradation of PCP proceeds by nonspecific oxygenations. Lignin-degrading fungi have been shown to oxidize PCP to tetrachloro-*p*-benzoquinone (Haggblom and Valo, 1995).

Biodegradation of PCP from soils by isolated microorganisms and mixed culture consortia has been well documented (Watanabe, 1973; Edgehill and Finn 1982, 1983; Apajalahti and Salkinoja-Salonen 1984, 1986; Saber and Crawford, 1985). Strains isolated from contaminated field sites have been most successful at oxidizing PCP and less chlorinated phenols (Stanlake and Finn, 1982; Edgehill and Finn, 1983). Isolating organisms capable of degrading chlorophenols anaerobically has proven more difficult. More commonly, anaerobic biodegradation of PCP has been demonstrated using a

microbial consortia found in wastewater sludge from anaerobic digesters (Boyd and Shelton, 1984; Mikesell and Boyd, 1985, 1986, 1988). Bioaugmentation with sludge inoculated with chlorophenols has been shown to be facilitate reductive dechlorination in numerous laboratory studies (Boyd et al., 1989; Bellin et al., 1990; Mikesell and Boyd, 1988). Bioaugmentation with similar sludge under aerobic conditions has been less widespread, but equally effective (Valo and Salkinoja-Solonen, 1986).

In field-scale demonstrations, bioremediation of soils by intrinsic bacteria and fungi, or by bioaugmentation, often results in incomplete removal of chlorophenols (Boyd et al., 1989) Residual chlorophenols remain in soil regardless of treatment methods or soil conditions. One approach to remedy this problem is soil treatment under sequential aerobic/anaerobic conditions. PCP is most readily removed from highly contaminated soil under aerobic conditions, while anaerobic treatment of large quantities of soil is less rapid, but results in greater removal (Boyd et al., 1989). This factor suggests that the most expedient method of soil treatment is to remove the bulk of PCP by oxidation, leaving the more recalcitrant contaminants to be removed by reductive dechlorination.

Sequential biological treatment of chlorophenols requires an electron donor for reductive dechlorination and a primary substrate for aerobic cometabolism. Transformation of PCP in soils using chlorophenols as the sole carbon and energy source is uncommon (Watanabe 1973; Haggblom and Valo, 1995). If there is no electron donor and primary substrate intrinsic to contaminated soil, they must be provided. In bench-scale studies, PCP removal from soils has been demonstrated in the presence of sodium glutamate and glucose as primary substrates for bacterial growth (Topp and Hanson, 1990; Hu et al., 1994). Information on extrinsic electron donors for soil remediation is limited, but in studies involving contaminated groundwater, the list of successful electron donors is extensive. Imitation vanilla flavoring (IVF) has been shown to promote complete mineralization of PCP to CO_2 by sequential treatment, serving as both an electron donor and a primary substrate (Roberts, et al., 1997). Unlike many carbon sources employed in chlorophenol remediation, IVF is a common food additive and is classified as GRAS (generally recognized as safe by the Food and Drug Administration).

The bioreactor designed for this treatment process is a fill and draw batch type, operated in weekly cycles. Constructed from a 6 cubic yard steel refuse container, it is intended to remove chlorophenols under both anaerobic and aerobic conditions. The reactor is separated into three soil treatment zones and four liquid mixing wells. Soil is infused with a liquid mixture containing a combination of nutrients, vitamins, and imitation vanilla flavoring (IVF). The purpose of the research presented here is to demonstrate the successful operation of this technology using the pilot-scale reactor, and similar bench-scale reactors. This study has four primary objectives:

1.   To demonstrate the effectiveness of fill and draw bioreactors at promoting removal of PCP from a large volume of low-permeability, contaminated soil without promoting soil washing,

2.   To evaluate the capability of imitation vanilla flavoring as an electron donor for anaerobic reductive dechlorination and as a primary substrate for aerobic cometabolism of PCP in contaminated soils,

3.   To determine the effect of bioaugmentation with PCP-inoculated anaerobic wastewater sludge on the transformation of chlorophenols in contaminated soil, and

4   To compare the effects of anaerobic and aerobic treatment mixtures on PCP removal and demonstrate removal of PCP from soil under sequential aerobic/anaerobic conditions.

This thesis presents the results and analysis of treating chlorophenol-contaminated soil in the pilot-scale reactor over 24 fill and draw cycles. Soil samples and treatment mixture samples were taken at the conclusion of each cycle to monitor the removal and mobility of chlorophenols. Soil samples were removed from each of the three soil treatment zones, and liquid samples were taken from each mixing well to monitor any patterns of preferential bioremediation or anomalies in reactor operation. Also included are the results and analysis of the concurrent bench scale study, conducted to optimize the performance of the pilot-scale reactor.

Chapter 2

Literature Review

Introduction

Bioremediation of contaminated soil can be an effective method for removing chlorinated compounds from hazardous waste sites. Halogenated aromatic compounds such as PCP can be completely mineralized to CO_2 under both anaerobic and aerobic conditions, using bioaugmentation, combined with composting and other ex-situ methods. This chapter examines the fate of chlorophenols in anaerobic and aerobic soils, and the potential for biodegradation using current field and laboratory techniques.

Anthropogenic Sources of Chlorophenols

Pentachlorophenol and tetrachlorophenols have been used extensively as agricultural and industrial biocides since the 1920's. In agricultural settings, they have been employed primarily as pesticides in rice paddies. Their industrial uses have been more varied, and have included use as a biocide in paints and oils and as a fungicide in freshly sawn timber. It is in the long-term preservation of timber products including power and telephone poles, and large scale structural members, where they have been utilized most extensively. The use of PCP and tetrachlorophenols in industry is universal. During the 1980's the annual word-wide production of PCP was estimated to range between 35 and 90 thousand tons (Haggblom and Valo, 1995). The production of all chlorophenols was estimated at 200 thousand tons per annum, with approximately 80 percent of total consumption attributed to the wood preservation industry (Haggblom and Valo, 1995).

Contamination of soil and groundwater at wood preservation facilities is common. There have been isolated cases in which wide-scale contamination was caused by an

industrial accident. Such an incident occurred in British Columbia in 1980, when 18,000 liters of a 7,500 mg/L chlorophenol solution leaked fom a dip tank (Haggblom and Valo, 1995). More often, contamination is a result of on-site treatment, handling, and storage methods. The treatment processes at wood preserving facilities vary among nations, but often do not include adequate features to prevent contamination of soil and ground water. In Scandinavian countries, a common method was to treat new timbers using dip tanks (Haggblom and Valo, 1995). Large bundles of lumber were submerged for a short time in a 1 to 2 percent solution of sodium chlorophenolate. In North America, wood treatment was accomplished using dip tanks and by using pressured vessels. Pressure vessels are still employed in wood treatment. Wood is pressure treated using a 3 to 6 percent solution of PCP dissolved in a petroleum based solvent. The solvent carrier liquid volatilizes under pressure making it easier for PCP to penetrate wood fibers. Unfortunately, volatile PCP readily penetrates small openings in treatment vessels. In the past, at the conclusion of the treatment process, timbers were left to drip dry on storage racks in areas unprotected from rain and snowfall. This drying process and leaching from storage areas during rainfall events has been a major cause or PCP contamination (Haggblom and Valo, 1995) PCP-contaminated soil sites are found throughout the world. In Finland, a study of wood treatment facilities showed soils to contain PCP in levels as high as several grams per kilogram of soil (Kitunen et al., 1987). Similar studies in the United States have shown PCP levels to range as high as several thousand milligrams per kilogram (Boyd et al., 1989). In response to their ubiquity, long-term use, and threat to human health, the USEPA has designated five chlorophenols, including PCP as priority pollutants.

Chlorophenol Interactions with Soils

Industrial and agricultural practices have led to wide-scale soil contamination by chlorophenols. Once in a soil environment, the fate of PCP is determined by several physical and chemical processes. PCP can be removed from soil by volatilization, or taken up by plants (Ferro et al., 1994). Degradation of PCP can occur via photodecomposition,

a process that includes reductive dechlorination, hydroxylation, and ring cleavage, or by microbial decomposition processes, including methylation, dechlorination, hydroxylation and mineralization (Boyd et al., 1989). PCP may sorb to soil organic matter or join with soil organic matter via oxidative coupling.

The transport and biological availability of chlorophenols in soil is most dependent upon the extent of sorption and binding of the compounds by soil humic matter (Boyd et al., 1989). Organic matter has been shown to act as a bulk phase solvent in solubilizing both ionic and non-ionic forms of chlorophenols in soil-water systems, making them more available to sorb and desorb from soils (Boyd et al., 1989). Sorption of PCP and 2,3,5,6-TeCP to soil is highly dependent upon environmental factors, and is most strongly influenced by soil pH and organic content (Kuwatsuka, S., and M. Igarashi, 1975; Schellenberger et al., 1984; Banerji et al., 1993; Galil and Novak, 1995). Sorption of non-ionic chlorinated phenols by organic matter, including PCP, is consistent with solute partitioning theory (Schellenberg et al., 1984). In laboratory studies using PCP and less chlorinated phenols, linear isotherms were observed for soil sorption over a wide range of relative solute concentrations, as was a linear relationship between K_{ow} and K_{oc}, (Chiou et al., 1985). In studies using moderate permeability Menfro series silt loam, Banerji et al., (1993) demonstrated a linear relationship between the sorption of PCP and the organic content of soil and an inverse relationship between sorption and soil pH.

Oxidative coupling is a microbial degradation reaction in which bound residues comprised of organic chemicals are incorporated into soil components (Sposito, 1989). Oxidative coupling enzymes can create stable covalent bonds between parent organic chemicals and soil organic matter, increasing the overall mass of organic matter (Dragun, 1988). Oxidative coupling is catalyzed by phenoloxidase and peroxidase enzymes produced by fungi, bacteria, and plants found in surface soils (Boyd et al., 1989). The process may also be autooxidative; catalyzed by soil minerals prevalent in soils such as clays (McBride, 1987).

Chlorophenol Biodegradation in Soils

Anaerobic Processes

Biodegradation and biological treatment of PCP is possible under both aerobic and anaerobic soil conditions. Anaerobic degradation of PCP in soils occurs almost exclusively via reductive dechlorination, a process in which chlorines on the aromatic ring are replaced by hydrogens, ultimately resulting in dichlorophenols (DCP) (Mohn and Tiedje, 1992). The anaerobic biodegradation pathways for PCP appear in Figure 1. Reductive dechlorination of PCP to TeCP can occur via *ortho*, *meta*, and *para* pathways. The dechlorination pathway depends upon the microbial consortia present; PCP is most often transformed via *ortho* dechlorination (Nicholson, 1990). A common degradation pathway for PCP in soils is via *ortho* dechlorination to 3,4,5,6-tetrachlorophenol (TeCP) and 3,4,5-trichlorophenol (TCP), followed by *para*-dechlorination to 3,5-dichlorophenol (DCP) and *meta*-dechlorination to 3-chlorophenol (CP) (Boyd et al., 1989). Another possible dechlorination pathway starts with *ortho* dechlorination to 3,4,5,6-TeCP, followed by removal of the *para* chlorine to form 2,3,5-TCP, or the *meta* chlorine forming 2,3,4-TCP (Boyd et al., 1989). Dechlorination of PCP often leads to the accumulation of less-chlorinated phenols, but complete mineralization to CO_2, or CH_4 is possible (Haggblom and Valo, 1995). A less common PCP degradation product in anaerobic soils is pentachloroanisole (PCP methyl ether). Methylation of PCP has been shown to account for up to 5 percent of degradation products in anaerobic soil (Murthy et al., 1979).

Aerobic Processes

Under aerobic conditions, PCP is transformed by hydroxylation and mineralization or by methylation (Boyd et al., 1989). The aerobic biodegradation pathways for PCP appear in Figure 2. In the hydroxylation process, a chlorine is removed from the phenolic
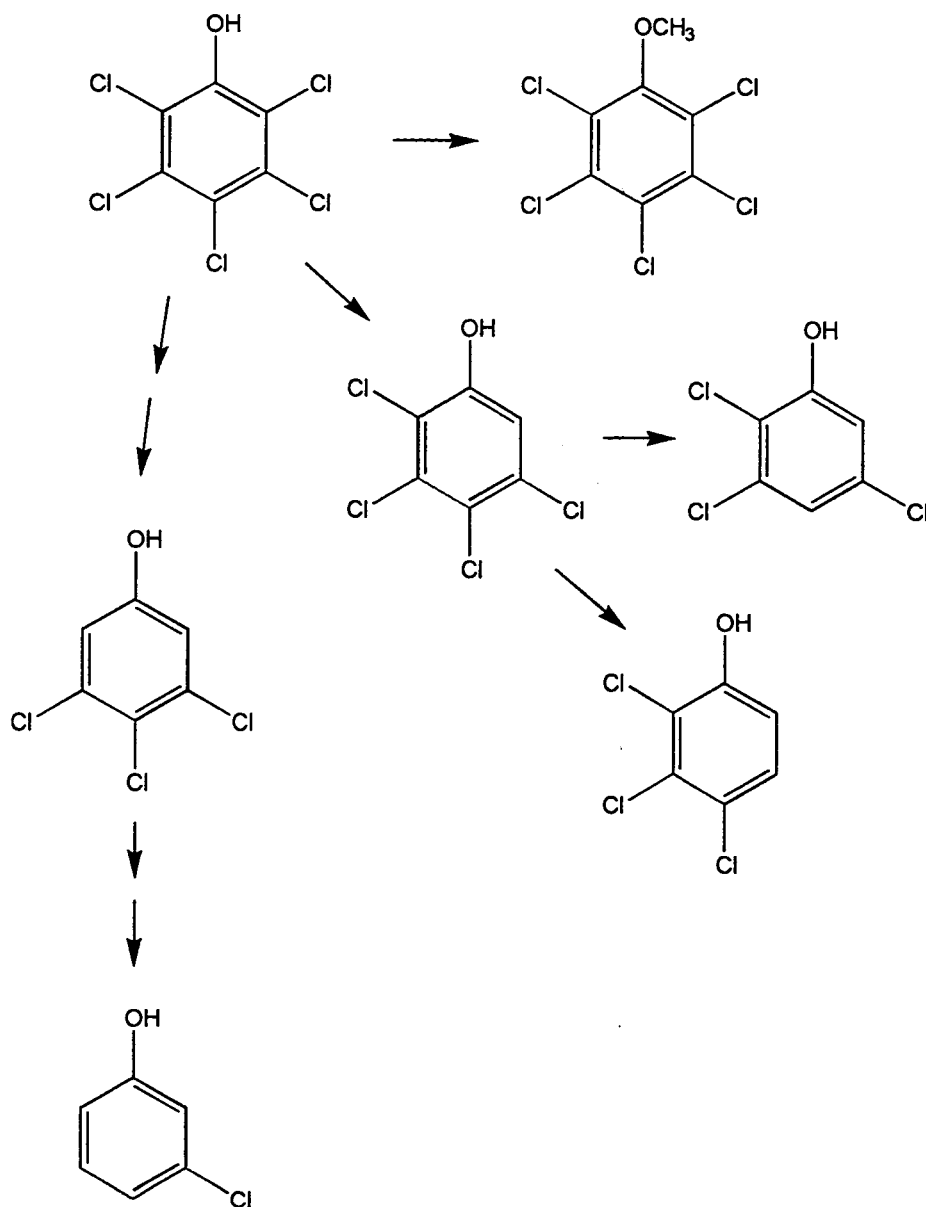
Figure 1. Anaerobic degradation pathways for PCP in soils
(adapted from Boyd et al., 1989)

ring and replaced by a OH⁻ radical. The hydroxylation transformation pathway under aerobic conditions, depends upon the type of biological media present. In aqueous environments containing bacteria, both *ortho* and *para* hydroxylation of PCP are common. Bacterial strains in soils transform PCP exclusively via *para*-hydroxylation to form
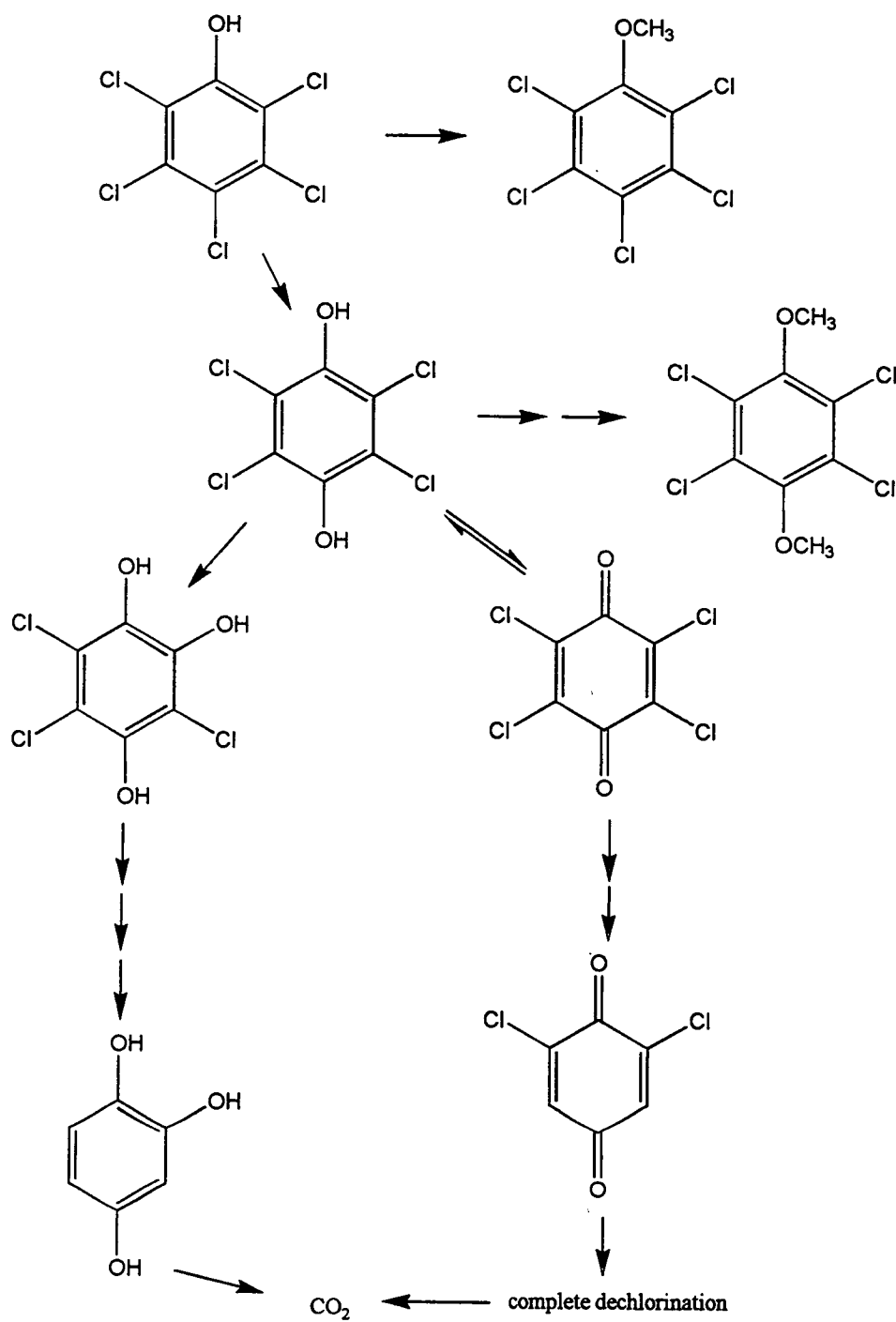
Figure 2. Aerobic degradation pathways for PCP in soils
(adapted from Boyd et al., 1989)

tetrachloro-*p*-hydroquinone (TeCH) (Apajalahti and Salkinoja-Salonen, 1987). TeCH can be hydroxylated at the *ortho* position, to form trichloro-1,2,4 trihydroxybenzene (THB), and ultimately, after three dechlorination steps, 1,2,4-trihydroxybenzene (Haggblom and Valo 1995). TeCH can also be dechlorinated without hydroxylation, resulting in trichlorohydroquinone (TCH) and 2,6-dichlorohydroquinone (DCH) (Steiert and Crawford, 1986). Fungal degradation of PCP proceeds by nonspecific oxygenations. Lignin-degrading fungi have been shown to oxidize PCP to tetrachloro-*p*-benzoquinone (Haggblom and Valo, 1995). TeCH can also be transformed to 2,3,5,6-Tetrachloroanisole. Methylation of PCP to pentachloroanisole is common in aerobic soils and can account for 50 percent removal of PCP in microcosm studies using a microbial consortia (Murthy et al., 1979).

Biodegradation by isolated microbes

Although it is highly successful as a general biocide, biodegradation of chlorophenols in soils has been reported since the 1970s. Biodegradation occurs in the presence of several different organisms, including bacteria and fungi. Bacterial strains capable of partial or complete mineralization of PCP and dechlorinated metabolites have been isolated by several researchers. In 1972 Chu and Kirsch demonstrated the mineralization of 75 percent of PCP to CO_2 using PCP as a sole source of carbon and energy. In early studies, strains of *Pseudomonas* sp. capable of growth using PCP were isolated from rice paddy soil (Watanabe, 1973; Suzuki, 1977). Using the isolated strain of *Pseudomonas* sp., Suzuki (1977) was able to demonstrate complete mineralization of PCP to CO_2. Four strains of bacteria in the genus *Arthrobacter* were isolated by Stanlake and Finn (1982). These strains were acquired from soil that was acclimated to PCP and from un-acclimated soil. Both samples demonstrated a consistent ability to metabolize PCP and trichlorophenols. The bacteria *Rhodococcus chlorophenolicus*, isolated from a mixed culture obtained from bark chips, was able to completely mineralize PCP and other polychlorinated phenol isomers to CO_2 (Apajalahti and Salkinoja-Salonen, 1986;

Middeldorp et al., 1990). In recent studies, multiple strains of *Flavobacterium* able to degrade PCP were isolated from Minnesota soils acclimated to between 12 and 800 mg/kg PCP. The bacteria promoted 73 to 83 percent mineralization of PCP at concentrations ranging from 100 to 200 mg/kg (Saber and Crawford, 1985).

Several fungal strains capable of reducing PCP concentrations in soils have been isolated, including *Cephaloascus fragrans* and *Trichoderma* (Cserjesi, 1967; Cserjesi and Johnson, 1972). Similar studies demonstrated the PCP biodegradation capabilities of *Lentinula edodes* and *Phanerochaete chrysosporium* (Okeke et al., 1996) Numerous lignin degrading fungi have been found to promote chlorophenol biodegradation in soils (Bumpus et al., 1989; Lamar and Dietrich, 1990; Lamar et al., 1990; Loske et al., 1990; Mileski et al., 1988)) Three lignin-degrading fungi, *Phanerochaete chrysosporium*, *Phanerochaete sordida*, and *Trametes hirsuta* were found to promote 55 to 89 percent removal from soil containing 672 mg/kg PCP, depending on the mass of the inoculum and the type of fungi applied (Lamar et al., 1993; 1994).

Biodegradation by Bioaugmentation

One of the most successful field and laboratory remediation techniques for chlorophenol-contaminated soils is bioaugmentation. Removal of PCP via bioaugmentation occurs under both aerobic and anaerobic conditions, using isolated bacterial strains, bacterial consortia, and fungal consortia.

Aerobic bioaugmentation is a common and effective treatment process for field sites with high levels of PCP contamination. In most cases, aerobic treatment results in removal of the bulk of the mass of chlorophenols, but is unable to achieve complete biotransformation. Valo and Salkinoja-Salonene (1986) demonstrated removal of PCP from soils containing several hundred mg/kg PCP by composting and inoculation with *R. Chlorophenolicus*. In this case, 10-30 mg/kg of PCP remained after 500 days of treatment. Composting efforts conducted with lignin-degrading fungi, such as *Phanerochaete chrysosporium, Phanerochaete sordidam*, and *Trametes hirsuta* have been

shown to remove up to 90% of PCP in field demonstrations (Lamar et al., 1993). Similar levels of residual PCP were shown by Crawford and Mohn (1985). 10-30 mg/kg PCP remained in soils that originally contained 300 mg/kg.

Anaerobic bioaugmentation is most successful using wastewater sludge from anaerobic digesters. In laboratory studies, bioaugmentation with digester sludge inoculated with chlorophenols has been shown to be facilitate transformation of PCP by *ortho* dechlorination to 3,4,5-TCP, followed by *para* dechlorination to 3,5-DCP (Boyd and Shelton, 1984; Mikesell and Boyd 1986; Mikesell and Boyd, 1988). In a study using anaerobic digester sludge from a wastewater treatment facility in Jackson, Michigan, PCP was completely mineralized in microcosms initially containing 74 μmol/kg. Sludge was added to microcosms at a rate of 5 g per kg of soil and incubated for 28 days. At the conclusion of the incubation, PCP was completely removed from microcosms containing sludge acclimated to chlorophenols. Un-inoculated soil microcosms achieved only 45 percent removal of PCP. Flasks containing sterile sludge achieved no removal. In a parallel study using soil from an actual wood treatment facility, 77 percent removal of PCP with 38 percent recovery as lower chlorinated phenols was accomplished using soil with an initial PCP concentration measured at 30 mg/kg (Boyd et al., 1989). Some studies suggest a relationship between the rate of sludge addition and PCP transformation (Mikesell and Boyd, 1988). In these studies, however, it was impossible to quantify the percentage of dechlorinated metabolites resulting from transformation of PCP from acclimated inoculum.

Biodegradation under sequential aerobic/anaerobic conditions

Supporters of bioremediation under sequential aerobic-anaerobic conditions propose that soils contaminated with PCP can be bioremediated most effectively by varying the oxygen content of the soil. Aerobic treatment, although effective at removing chlorophenols from highly-contaminated soils, invariably results in some residual contamination. Anaerobic treatment, although relatively slow, can result in complete

mineralization of PCP and less chlorinated chlorophenols in highly-contaminated soils. Supporters of sequential treatment propose that contaminated soil be treated first under aerobic conditions, to remove the bulk of chlorinated phenols, then under anaerobic conditions, to remove more recalcitrant compounds.

Laboratory and field scale demonstrations of PCP transformation and removal under sequential conditions are rare. Mineralization of aqueous PCP to CO_2 under sequential anaerobic/aerobic conditions was recently shown by Roberts et al. (1996)., Mikesell and Boyd, (1988) tested the potential for treatment of PCP-contaminated soil using a sequential aerobic/anaerobic scheme and inoculation with unacclimated anaerobic digester sludge. Soil was amended with anaerobic wastewater sludge and incubated in flasks under aerobic conditions for 28 days. At the conclusion of the incubation, the contents of the flask was split and re-inoculated. The two flasks were incubated for 28 days, after which they were split, with one half returning to aerobic conditions, the other half remaining anaerobic. In the course of 84 days of treatment, PCP concentration decreased by 20 percent in aerobic bottles. In the first 28 day anaerobiosis the concentration of PCP steadily declined to near 0 mg/kg, while the concentration of dechlorinated metabolites rapidly increased. During the third 28 day period, anaerobic bottles showed continued transformation of PCP and dechlorinated metabolites, while those under aerobic conditions showed no additional removal.

Interactions with Chlorophenol Acclimated Wastewater Sludge

Anaerobic digester sludge is added to contaminated soil to increase the mass of chlorophenol-degrading bacteria in the system. Adding PCP inoculated sludge to bioreactors and composting applications has three significant, direct effects: it increases soil pH, increases the organic content of the soil, and changes the distribution of chlorophenols in the soil/water/colloid system (Banerji et al., 1993).

Coupled with the addition of organic matter to any soil bioreactor is an increase in pH (Banerji, 1993) and a corresponding increase in chlorophenol solubility. In high pH

systems, more chlorophenols are found in their ionic and more hydrophilic form, phenolate. In microcosm studies conducted with 846 mg PCP/kg soil at 1.38% TOC, Banerji et al. (1993) demonstrated an increase in PCP solubility, measured as PCP recovery from soils, (from 47.2% to 72.9%) when system pH was increased from 5.0 to 9.0. Christodoulatos and Mohiuddin (1996). demonstrated similar results for enhanced aqueous mobility of PCP in soil/water systems containing 2.96% TOC. In their analysis, equilibrium masses of PCP in soil decreased from 42.8% to 19.7% in soil/water microcosms containing 387.50 µg PCP when pH was increased from 4 to 10.

The most significant effect of sludge supernatant addition is an increase in the concentration of soluble organic matter in the soil reactor and a change in the distribution of PCP in the soil/water/colloid system. The organic fraction in soil increases as a direct result of augmented cell mass, but it also increases due to the addition of chlorophenols. When chlorophenols added as part of an inoculum contact soil, they induce a release of low molecular weight organic compounds and colloids from the soil surface (Galil and Novak, 1995). In microcosms containing 10g of soil at a pH of 5.35 comprised of 1.8% organic matter Galil and Novak (1995) demonstrated that the physical separation of colloids from saturated soils increases with PCP concentrations. Microcosms were spiked with 150 mL of 0 mg/L, 2.5 mg/L, 5 mg/L, and 10 mg/L PCP solutions and measured after an initial contact time of 24 hours. The concentration of soluble soil organic matter in microcosms containing 2.5 mg/L PCP increased from 8 mg/L to 65 mg/L, measured as TOC. PCP additions at 5 mg/L and 10 mg/L resulted in an increase to 85 mg/L. The same study conducted with soil at a pH of 7.25, comprised of 8.25 % organic matter showed an increase in soluble organic matter from 10 mg/L to 30 mg/L TOC at 2.5 mg/L PCP and 35 mg/L TOC at 5 mg/L PCP and 10 mg/L PCP. One third of the TOC measured was considered to be associated with colloids.

PCP introduced into bioremediation schemes does not distribute evenly in the soil/water/colloid system. Inoculated sludge contains chlorophenols in aqueous form and chlorophenols complexed with organic matter. Chlorophenols introduced in solution may complex with the solid phase soil or remain in the aqueous phase. Galil and Novak (1989) and Boyd et al., (1990) demonstrated that the fate of PCP added to flooded soil systems is

dependent upon soil pH and organic content. In soils with low concentrations of organic matter (1.8%) and pH of 5.35, PCP was more likely to be found in the aqueous form (Table 1). Galil and Novak (1989) found that the distribution of PCP among the three system components was similar at PCP concentrations of 2.5, 5 and 10 mg/L. In bottles containing soil and water, 87.1 percent to 90 percent of chlorophenols were found in aqueous form. 6 to 9.8 percent of chlorophenols were bound to soils, at levels increasing with initial PCP concentration. The concentration found in colloids ranged from 3.1 to 4 percent. At systems with pH greater than 7.25, and 8.25% organic matter, added chlorophenols were more likely to sorb to soils or bind with colloids. The increase in system pH resulted in increased chlorophenol availability. As a result of the increase in organic matter, more chlorophenols were found in the solid phase. Galil and Novak (1989) found that as initial PCP concentrations increase from 2.5 to 10 mg/L, solid phase chlorophenols decreased from 33.6 to 26.2 percent of the total (Table 1). The concentration found in colloids decreased from 13.2 to 26.2 percent.

Table 1. Percent distribution of PCP in a soil/water/colloid system as a function of pH, TOC, and PCP concentration[1]

Soil description	pH = 5.35 TOC = 1.8%		
Aqueous Phase	90	87.2	87.1
Colloids	4	3.8	3.1
Solid Phase	6	9.0	9.8
Total PCP (%)	100	100	100
Initial Conc. (mg/L)	2.5	5.0	10.0

Soil description	pH =7.25 TOC =8.25%		
Aqueous Phase	53.2	61.2	71.2
Colloids	13.2	6.8	2.6
Solid Phase	33.6	32	26.2
Total PCP (%)	100	100	100
Initial Conc. (mg/L)	2.5	5	10

[1]adapted from Galil and Novak, 1989.

Biodegradation in the presence of an extrinsic electron donor and substrate

Microbial biodegradation of chlorophenols under anaerobic and aerobic conditions requires a primary substrate for aerobic cometabolism and an electron donor for reductive dechlorination. In some cases, intrinsic carbon and energy sources exist to promote in-situ or ex-situ transformation. PCP and less chlorinated phenols can serve as carbon sources for microbial growth (Watanabe, 1973; Haggblom and Valo, 1995). PCP can also serve as an energy source to promote aerobic transformation and mineralization of up to 75 percent of PCP in contaminated soil (Chu and Kirsch, 1972; 1973). If there is no electron donor and primary substrate intrinsic to contaminated soil, one must be provided. Carbon sources used to promote anaerobic transformation of PCP in soil have included glucose (Middeldorp et al, 1990; Boyd et al 1989; Topp and Hanson, 1990; Zhong-Cheng et al., 1994), sodium glutamate (Topp and Hanson, 1990; Seech et al., 1991), and soybean residue (Boyd et al., 1989).

Information on extrinsic electron donors for soil remediation is limited, but in studies involving contaminated groundwater, the list of successful electron donors is extensive. Imitation vanilla flavoring (IVF) has been shown to promote complete mineralization of PCP to CO_2 by sequential treatment, serving as both an electron donor and a primary substrate (Roberts, et al., 1997). Unlike other carbon sources employed in chlorophenol remediation, IVF is a common food additive and is classified as GRAS (generally recognized as safe by the Food and Drug Administration).

Conclusion

Chlorophenols are halogenated aromatic compounds commonly found as contaminants in soil at hazardous waste sites throughout the world. PCP and less chlorinated phenols are readily biodegraded under both aerobic and anaerobic soil conditions. Laboratory and field-scale demonstrations have shown mineralization of PCP using numerous bacteria and fungal strains, and by bioaugmentation with anaerobic

digester sludge. The success of biological treatment of PCP-contaminated soil depends on the potential for microbial growth and the presence of an electron donor for reductive dechlorination. Soil treatment can be enhanced by adding a mixture capable of facilitating both aerobic and anaerobic biological processes.

Chapter 3
Evaluation of Bench-Scale and Pilot-Scale Bioreactors
Designed to Bioremediate Low-Permeability Pentachlorophenol-Contaminated Soil

Materials and Methods

The capability of fill and draw batch reactors to bioremediate chlorinated phenols' in low permeability soil was evaluated in bench-scale and pilot-scale systems. Bench-scale reactors were devised to assess the transformation of PCP and 2,3,5,6-TeCP in soil from a field site under sequential aerobic/anaerobic conditions. Treatment was evaluated under varying conditions by infusing soil with liquid treatment mixtures containing an extrinsic microbial population in the form of anaerobic wastewater sludge supernatant and/or an external electron donor in the form of imitation vanilla flavoring (IVF). A pilot scale reactor was designed to treat 3.7 m^3 of soil contaminated' with PCP and 2,3,5,6-TeCP using the processes demonstrated in the bench scale reactors.

Bench-Scale Fill and Draw Reactor Study

Configuration and Set-up

Five series of nine bench-scale reactors were filled with 50 g of soil from the McFarland Cascade pole treatment facility in Eugene, Oregon. Each reactor series was maintained under different conditions by supplying them with a distinct aqueous treatment mixture. The mixtures contained a combination of anaerobic wastewater sludge supernatant and IVF diluted in deionized water (Table 2). One series of reactors (series A), treated only with deionized water, was maintained throughout the treatment process. Series B was fed a deoxygenated treatment mixture containing IVF. Bench-scale reactors

were operated for nine, week-long fill and drain cycles. At the beginning of the first cycle, each reactor was flooded with its respective treatment mixture. At the conclusion of each week, the liquid contents of each reactor was drained and analyzed for chlorophenols. The saturated soil in one reactor from each series was removed at the conclusion of each fill and draw cycle, and analyzed for chlorophenols by Soxhlet extraction. To initiate the next cycle, the remaining reactors were then re-filled with fresh treatment mixtures.

Table 2. Bench-scale reactor set-up

Reactor Series	Deoxygenated/ Deionized H_2O	Vanilla Flavoring	Nutrient Mixture	Anaerobic Sludge Supernt.
A	NO	NO	NO	NO
B	YES	YES	YES	NO
C	NO	YES	YES	NO
D	NO	YES	YES	YES
E	NO	YES	YES	YES (4x)

Sequential Aerobic/Anaerobic Treatment

Bench-scale bioreactors were operated under sequential aerobic/anaerobic soil conditions with the intent of improving and expediting overall removal and transformation of chlorophenols. At the initiation of each treatment cycle, bench-scale reactors contained oxygen in the form of air dissolved in treatment solutions. Over the course of the treatment cycle, the dissolved oxygen content of the soil/water system could be reduced by aerobic biological activity and volatilization into reactor head-space or ambient air. Utilization of any air entrained in soil pore water and liquid treatment mixtures created an environment more favorable to anaerobic processes. Draining treatment solutions at the end of each week-long cycle introduced air back into the reactor, facilitating chlorophenol removal under aerobic conditions.

Design and Construction

Forty-five identical fill and draw batch reactors were used to conduct the bench-scale study. Each reactor was constructed of a 6 inch section of 1-1/4 in ID chlorine resistant poly-vinyl chloride pipe (CPVC), fitted with an end cap (Figure 3). A 1/4 inch x1/4 NPT, poly-vinyl chloride (PVC) barbed hose fitting was connected to each end cap to serve as a portal for injecting and drawing off treatment mixtures. One four inch section of Tygon tubing was affixed to each hose fitting. Small plastic tube clamps (McMaster-Carr, Los Angeles, CA) were fitted to the sections of tubing to act as check valves during treatment cycles. To promote an even distribution of feed solution, diffusers constructed of 1/8 inch glass beads encased in 1000 μm polyester filter paper were inserted at the base of each reactor. The diffusers were held in place by covering them with a small circular section of 200x200, 304 stainless steel mesh (McMaster-Carr, Los Angeles, CA), and securing the mesh to the reactor wall with epoxy. Reactors were rinsed once with reagent grade methanol and five times with deionized water after they were assembled.
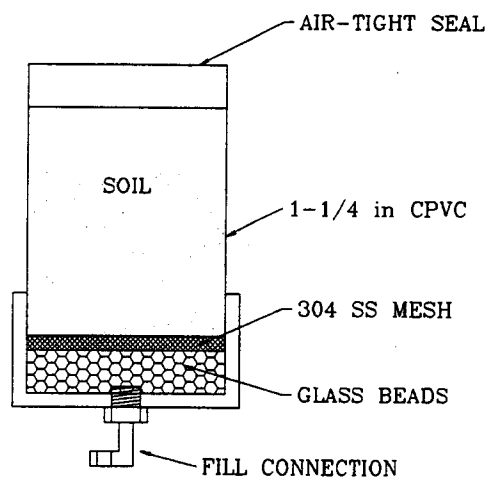
Figure 3. Bench-scale reactor

Soil Preparation

Soil was obtained in the form of saturated well cuttings from the McFarland Cascade pole treatment facility in Eugene, Oregon. Soil used in the pilot-scale study was a heterogeneous mixture of clay, sand, and gravel. Prior to filling the bench-scale reactors, the site soil was air dried and sorted using a #4 ASTM sieve to remove large cobble, twigs and other undesirable substances.

Liquid Feed Mixture Preparation

Bench-scale reactor feed mixtures were prepared in 1000 mL Nalgene® bottles at the beginning of each cycle. The mixtures were composed of a combination of deionized water, IVF, the supernatant extracted from PCP inoculated anaerobic wastewater sludge and a mixture of inorganic nutrients and vitamins. The contents of each mixture was as follows.

Reactor Series A - 1000 mL DI water.

Reactor Series B - 1000 mL deoxygenated DI water, 4.2 mL IVF, 17 µL S3, 84 µL S4, 55 µL S7.

Reactor Series C - 1000 mL deionized water, 4.2 mL IVF, 17 µL S3, 84 µL S4, 55 µL S7.

Reactor Series D - 750 mL deionized water, 250 mL anaerobic wastewater sludge supernatant, 4.2 mL IVF, 17 µL S3, 84 µL S4, 55 µL S7.

Reactor Series E - 1000 mL anaerobic wastewater sludge supernatant, 4.2 mL IVF, 17 µL S3, 84 µL S4, 55 µL S7.

The IVF was prepared at 23,600 mg/L COD, to specifications listed in Kaslik (1996) and contained 3.6 g/L guaiacol ($C_7H_8O_2$), 1.2 g/L ethyl vanillin ($C_9H_{10}O_3$), 7.8 g/L propylene glycol ($C_3H_8O_2$) and 0.8 g/L benzoate ($C_7H_5O_2$). Concentrations of organic constituents

in each liter of reactor feed mixture containing IVF were 15.0 mg/L guaiacol, 5.0 mg/L ethyl vanillan, 32.6 mg/L propylene glycol, and 3.3 mg/L benzoate. S3 and S4 are modifications of mineral mixtures and S7 is a vitamin mixture recommended by Owen et al., (1979) (Appendix M). The mineral and vitamin mixtures used in this study were 100:1 dilutions of Owen's stock solutions, prepared in DI water.

Anaerobic wastewater sludge was acquired from the Municipal Wastewater Treatment Facility, Corvallis, Oregon on October 3, 1995. Anaerobic conditions were maintained by flooding the storage carboy headspace with nitrogen. The carboy was maintained at 20°C. Sludge was acclimated to PCP by numerous additions totaling 0.75 μM (0.2 mg/L) PCP. Carboys received monthly injections of IVF at no more than 100 mg/L as COD and 100:1 dilutions of S3, S4 and S7 in DI water, as recommended by Owen et al. (1979). Supernatant was removed from the sludge suspension using a siphon device propelled by compressed nitrogen and stored in a 4 L amber glass container prior to feed solution preparation.

Reactor Operation and Sampling

The fill and draw reactors operated in weekly cycles. To start each treatment cycle, reactors were filled with 35 mL of feed solution using a 0.002 liter per minute peristaltic pump (McMaster-Carr, Los Angeles, CA). The discharge end of the pump tubing was secured to the reactor fill and drain tubing using a 1 inch section of 1/4 ID stainless steel tubing as a connector. Fill tubing was flushed with deionized water for 30 seconds between reactor series to protect against cross-contamination of reactors. To ensure anoxic conditions, the headspaces of anaerobic reactors were purged with oxygen-free nitrogen during the fill and drain process then securely capped with no. 7 butyl rubber stoppers.

At the conclusion of each one-week cycle, treatment solutions were removed from each reactor through the fill and drain ports, using the peristaltic pump. The quantity of liquid drawn off each reactor was recorded and 2 mL of each treatment mixture was

retained for analysis. The solid contents of one reactor from each series was destructively sampled at the conclusion of each fill and draw cycle. After drawing off the treatment solution, the saturated soil was removed from the subject reactor using a stainless steel spatula and placed in a 50 mL beaker in preparation for Soxhlet extraction.

Pilot-Scale Fill and Draw Reactor Study

Configuration and Set-up

The pilot-scale reactor was designed to treat large quantities of contaminated soil by periodically flushing the soil with a recirculating treatment mixture. The reactor was operated by pumping treatment mixtures into vertical liquid mixing zones. Through the hydraulic head supplied by the liquid column, the treatment mixture infiltrated adjacent soil treatment zones. After a one week incubation period, the treatment mixture was drained, supplemented as needed, and recycled into the reactor.

Sequential Aerobic/Anaerobic Treatment

Like the bench-scale bioreactors, the pilot-scale reactor was operated under sequential aerobic/anaerobic soil conditions. The dissolved oxygen content of the soil/water system increased at the beginning and conclusion of each fill and drain treatment cycle, with the intent of improving overall transformation and removal of chlorophenols.

Design and Construction

The pilot-scale bioreactor was constructed from a 6 cubic yard plain steel refuse container (DeWald Northwest, Salem, OR) finished with two-part polyurethane paint. The container was divided into two 41 cm and one 64 cm soil treatment zones and three 9 cm liquid mixing zones (Figure 4). Mixing zones were constructed using 14 gauge plain steel sheets with offset 1/8 inch perforations reinforced, with L 2x2x3/8 steel angles. Mixing sections created soil subdivisions and served as wells to supply treatment mixtures to contaminated soil.

LIQUID MIXING ZONES

SOIL MIXING ZONE   SOIL MIXING ZONE   SOIL MIXING ZONE
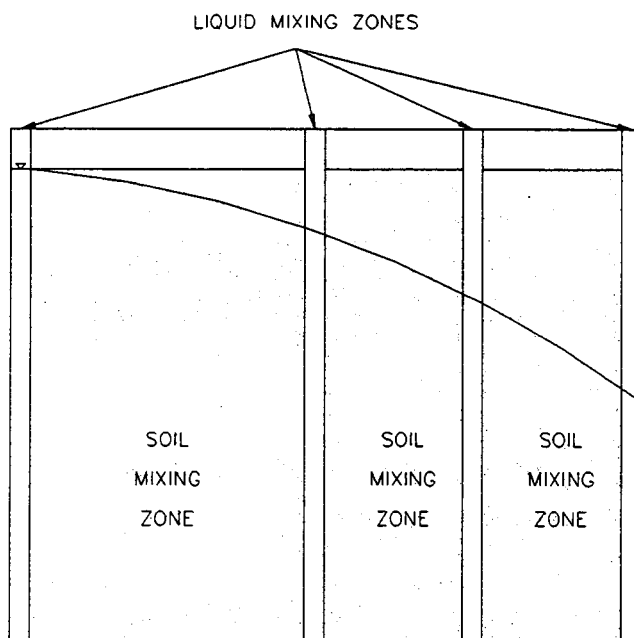
Figure 4. Pilot-scale bioreactor cross-section showing liquid mixing wells, soil treatment zones, and flow profile of treatment mixtures through soil zones

Operation and Sampling

The pilot-scale reactor was operated in weekly cycles and was fed the same mixture of nutrients and IVF as reactor series B and C. Soil was treated by filling well sections and allowing the treatment mixtures to slowly infiltrate the soil zone (Figure 5). Wells were filled by pumping the feed mixture, prepared with tap water, from a 55 gallon steel drum using a 2.8 gpm submersible pump (McMaster-Carr, Los Angeles, CA). Only one well section served as the fill port during each cycle. The well section was filled with the treatment mixture to the
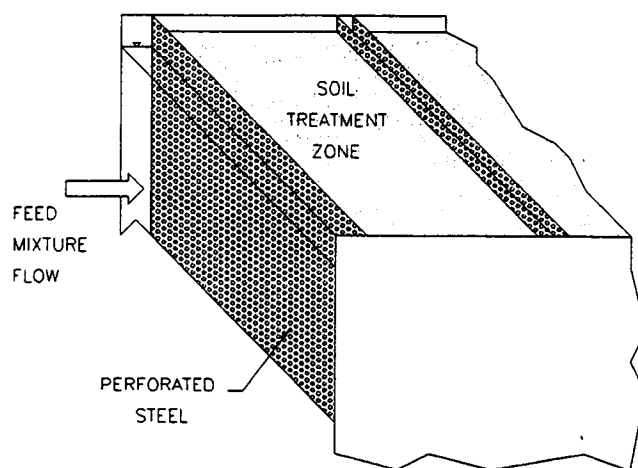
Figure 5. Operational cross-section of pilot scale reactor
showing influx of liquid mixtures through soil treatment zones

height of the soil present in the adjoining treatment zone. After the liquid levels in each well equilibrated, the single supply well was re-filled. This process continued until each well section was filled to the height of the adjoining soil treatment zone. This method promoted complete saturation of each soil section under the greatest possible gravitational head. Treatment mixtures were drained from the reactor by repeatedly pumping dry each well section in succession, until no liquid leached from soil zones. Drain mixtures were

temporarily stored in 55 gallon steel drums and analyzed for chlorophenol content. The mixtures were supplemented with IVF and nutrients and then pumped back into the successive well section. Fill mixtures were augmented such that the concentration of IVF remained between 150 mg/L and 220 mg/L IVF as COD.

Soil and pore water samples were drawn from treatment zones at the conclusion of each cycle. Soil samples were taken using a hollow, cone-shaped aluminum sampler screwed to the end of a 1.5 m long aluminum rod. Samples were captured by driving the sampler to the desired depth using a dead-blow mallet. The sampler was then removed and its contents emptied into a 50 mL flask. Soil sample locations within each zone varied over the course of the experiment, but were always taken from a depth of approximately 60 cm from the soil surface. Pore water samples were obtained from sampling wells placed at the center of each treatment zone. Samples were captured by inserting a glass tube into the well and siphoning its entire contents using a pipette bulb.

Analytical Procedures

Chlorophenols were removed from soil samples by Soxhlet extraction using the process described by Woods et al. (1985). Liquid samples from each fill and drain cycle were placed in 2 mL plastic centrifuge tubes and spun at 5000 rpm for 5 minutes using an Eppendorf 5415C micro-centrifuge to separate solids. Chlorophenol analyses were conducted on liquid samples using a hexane extraction procedure developed by Voss et al (1981) and modified by Smith (1993) (Appendix J). 100 μL water samples are mixed with 1 mL of an internal standard reagent prepared with 30.4 g/L K_2CO_3 and 500 mg/L 2,4,6-tribromophenol in a 10 mL test tube. 100 mL of acetic anhydride is added to the mixture; the tube was sealed with an air-tight Teflon® lined cap and shaken for 20 minutes. The tube was removed from the shaker and the cap was removed. 1 mL of HPLC grade hexane was added and the tube was shaken for an additional 20 minutes. Hexane was removed from the tube and placed in a 2 mL amber glass GC vial with a Teflon® lined septa and aluminum crimp cap.

Chlorophenols were quantified using a Hewlett-Packard 6890 gas chromatograph (GC) equipped with a [63]Ni electron capture detector and a J&W Scientific DB-5MS 30m column (J&W Scientific, Folsom, CA). The GC was controlled by a Hewlett-Packard Chemstation™ outfitted with Rev.A.05.01 [273]© software. The ECD was programmed to hold an initial oven temperature of 40°C for when minute then ramp at 25°C per minute to a mid point temperature of 140°C. From 140°C, the oven temperature increased to 250°C at a rate of 10°C per minute. The oven temperature was held for five minutes and injector and detector temperatures were maintained at 250°C and 350°C, respectively. Helium, at an initial flow of 2 mL per minute, served as the carrier gas. After fourteen minutes, the flow increased to 4 mL per minute at a rate of 4 mL per minute, where it was maintained for seven minutes. The detector auxiliary gas consisted of a 95:5 mix of Ar:CH$_4$ maintained at a flow rate of 60 mL per minute.

Results of Bench-Scale and Pilot-Scale Studies

The focus of this study was evaluation of the fill and draw batch reactor process as a method to bioremediate chlorophenols in low-permeability soils. Batch reactors were designed to infuse soil with a liquid treatment mixture, promoting aerobic respiration and reductive dechlorination under sequential aerobic/anaerobic conditions. The effectiveness of the reactor system was evaluated in bench-scale and pilot-scale experiments. Bench-scale studies assessed five different treatment mixtures and the liquid delivery system. Five series consisting of nine reactors each, treated chlorophenol-contaminated soil using mixtures composed of a combination of distilled water, imitation vanilla flavoring, and anaerobic wastewater sludge supernatant (Table 2). In the pilot-scale study, approximately 3.7 m^3 of contaminated soil were treated using a similar fill and draw bioreactor process.

Bench-Scale Fill and Draw Reactor Study

Reactors were operated for nine seven-day fill and draw cycles. The saturated soil contents of one reactor from each series was removed each week over the course of the experiment, and analyzed for chlorophenols. Fill and drain treatment solutions were also monitored for chlorophenol content. The molar masses of chlorophenols in saturated soil during the nine week treatment process are shown in Figures 6 through 11. These masses have been adjusted to account for losses or gains of chlorophenols from draining treatment solutions or by adding inoculated anaerobic wastewater sludge supernatant.

Background Concentrations of Chlorophenols in Soil and Soil Conditions

Soil treated in this study was a heterogeneous mixture obtained from a well cutting operation in the form of a slurry. Preliminary analyses showed that the soil, once dried and prepared for treatment, contained PCP in relatively low concentrations, ranging from 2.4 mg/kg to 3.0 mg/kg. By comparison, PCP contamination in soils at Superfund sites has been recorded at concentrations in excess of 100 mg/kg (Boyd et al., 1989). Tetrachlorophenols, occurring as a result of PCP transformation or as impurities in the grade of PCP used on site, were also present in soil samples. Concentrations of 2,3,5,6-TeCP ranged from 0.59 mg/kg to 0.73 mg/kg. 2,3,4,5-TeCP was found at trace levels, less than 0.08 mg/kg. Based on five soil samples, fill and draw reactors prepared with 50 g of soil contained an average of 0.50 μmol PCP (standard deviation $\sigma = 0.013$), 0.14 μmol 2,3,5,6-TeCP ($\sigma = 0.013$), and 0.014 μmol 2,3,4,5-TeCP ($\sigma = 0.0019$).

Laboratory studies were conducted to determine the physical and chemical characteristics of the site soil. Saturated hydraulic conductivity was measured at 1.4×10^{-8} m/day using a falling head permeameter (Appendix K). CEC was measured at 8.8 meq/100g and the pH was 8.2, indicating a moderately alkaline mixture. The organic content of the soil was approximately 1.2 percent. Soil conductivity, visual examination, CEC levels, and mineral content suggested that the soil was a clay-sand mixture (Sposito, 1989) common to the Willamette River valley.

PCP & 2,3,5,6-TeCP Removal

Fill and draw reactor technology promoted removal of PCP and 2,3,5,6-TeCP in low permeability soil under all test environments (Figure 6). Similar results were achieved using completely anaerobic and sequential aerobic-anaerobic reactors. Reduction of PCP and 2,3,5,6-TeCP levels in soil was demonstrated regardless of the presence of imitation vanilla flavoring or anaerobic wastewater sludge supernatant (Table 3). Most removal occurred during the first three cycles. Based on an average of soil samples taken between

cycles 4 and 9, approximately 70 percent to 81 percent of PCP (Figure 6) and 80 percent to 88 percent of 2,3,5,6-TeCP (Figure 7) were removed from soils over the nine week experiment duration. The average removal of PCP in all five reactor series was 74.1 percent by mass ($\sigma = 4.4$); average removal of 2,3,5,6-TeCP was 84.5 percent ($\sigma = 3.8$)

Table 3. Chlorophenol removal in fill and draw reactors over 63 days (percent by mass)

Reactor Series	PCP[1]	2,3,5,6-TeCP[1]
A	69.9±4.7	84.6±4.4
B	75.3±4.3	79.5±7.8
C	70.9±15.7	82.2±9.0
D	73.4±5.0	87.9±5.0
E	80.9±128	88.4±6.0

[1]Mean and standard deviation of removal efficiencies observed for the period between weeks four and nine
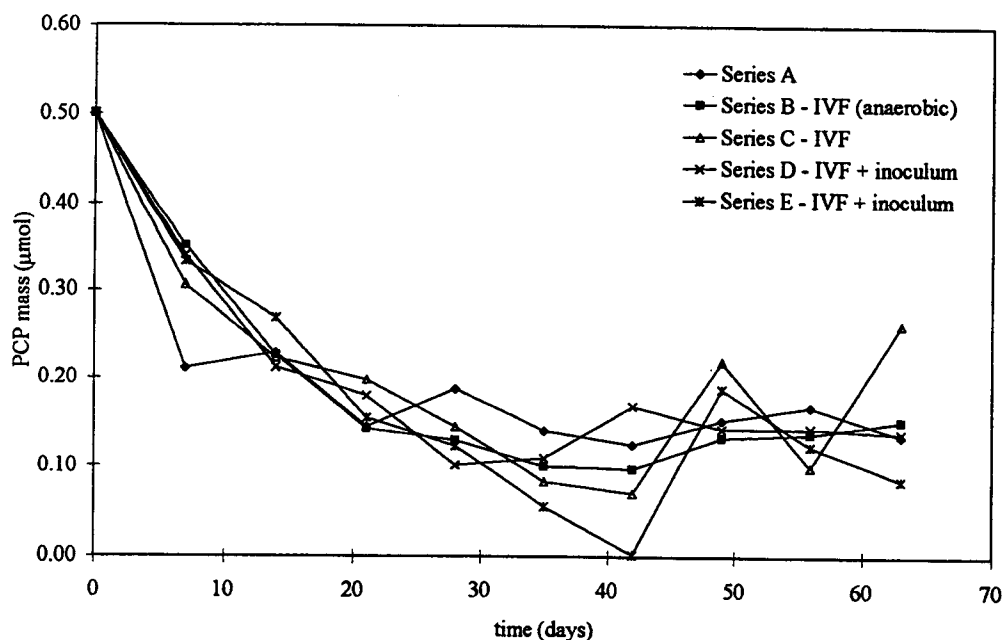
Figure 6. PCP removal from bench-scale reactor series A through E over nine fill and draw cycles.
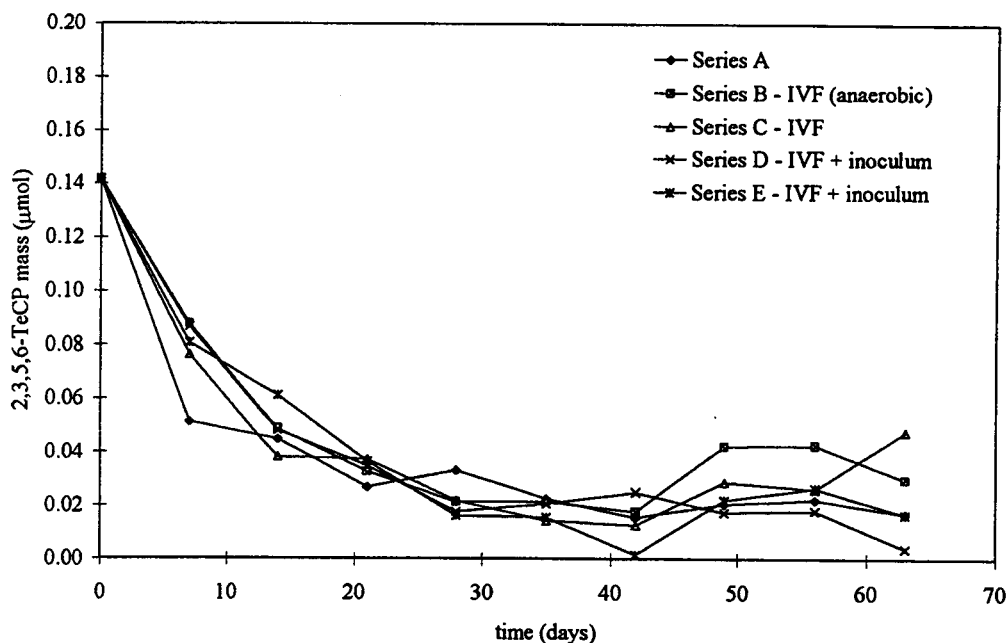
Figure 7. 2,3,5,6-TeCP removal from bench-scale reactor series A through E over nine fill and draw cycles.

Anaerobic PCP & 2,3,5,6-TeCP Transformation

The biological transformation of PCP and 2,3,5,6-TeCP in reactors treated with imitation vanilla flavoring, occurred under quasi-anaerobic conditions (Figure 6 through Figure 11). PCP and 2,3,5,6-TeCP were dechlorinated to 3,5-DCP in soil reactors inoculated with anaerobic wastewater sludge supernatant (series D and E) and in uninoculated soils, treated with completely anaerobic and anoxic fill solutions (series B). In uninoculated reactor series C, anaerobic treatment of PCP and 2,3,5,6-TeCP resulted in transformation to 2,3,5-TCP and 3,4,5-TCP, while 3,5-DCP was not observed. Anaerobic transformation of PCP and 2,3,5,6-TeCP was insignificant in reactor A, which showed no dechlorinated metabolites.

The appearance of anaerobic metabolites was dependent upon the biomass. Reactors containing a greater mass of anaerobic wastewater sludge generated anaerobic

metabolites within the first treatment cycle. Production of 2,3,4,5-TeCP was observed in the first cycle in reactor series E, the series containing the higher concentration of sludge. 2,3,4,5-TeCP was observed in the second cycle in reactors containing less inoculum (series D) (Figure 8). A lag time of five weeks occurred before 2,3,4,5-TeCP was observed in uninoculated reactor series C.
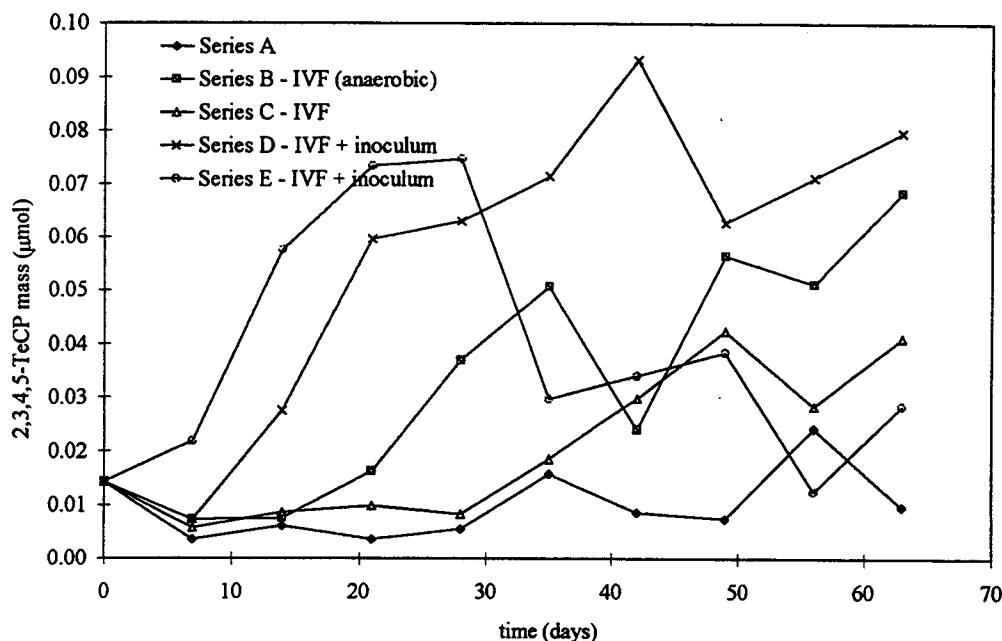
Figure 8. Production of 2,3,4,5-TeCP in bench-scale reactors from anaerobic transformation of PCP

The appearance of 3,4,5-TCP from *ortho* dechlorination (Figure 9) followed a trend consistent with the production 2,3,4,5-TeCP in inoculated and uninoculated soil reactors fed imitation vanilla flavoring. 3,4,5-TCP appeared first in E series reactors, followed by series D and C. Based on a comparison of reactors series B and C, the rate of production of 3,4,5-TCP was more rapid when anaerobic fill solutions were used. In reactor series B, 3,4,5-TCP first appeared during cycle 4 (Figure 9). In comparison, 3,4,5-TCP first appeared in reactor series C during the sixth cycle.
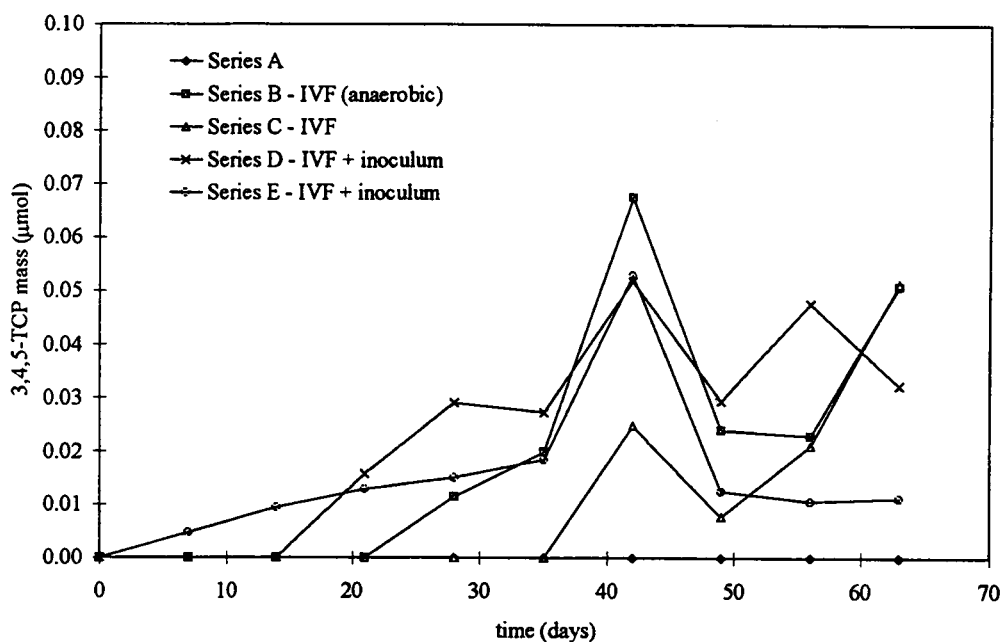
Figure 9. Appearance 3,4,5-TCP in bench-scale reactors from anaerobic transformation of 2,3,4,5-TeCP

2,3,5-TCP generated from *ortho* dechlorination of 2,3,5,6-TeCP and/or *para* dechlorination of 2,3,4,5-TeCP appeared during the first fill and draw cycle in soil reactors inoculated with anaerobic wastewater sludge (Figure 10). Production in uninoculated reactors did not occur until the third fill and draw cycle. In all reactor series, the appearance of 2,3,5-TCP preceded the emergence of 2,3,4,5-TeCP, suggesting that the 2,3,5-TCP was produced from *ortho* dechlorination of 2,3,5,6-TeCP. Production of 3,5-DCP was observed in the third cycle in the inoculated reactors, independently of biomass, and in the sixth cycle in the uninoculated anaerobic reactor series B (Figure 11). Dichlorophenols were never observed in soil samples taken from reactor series C.
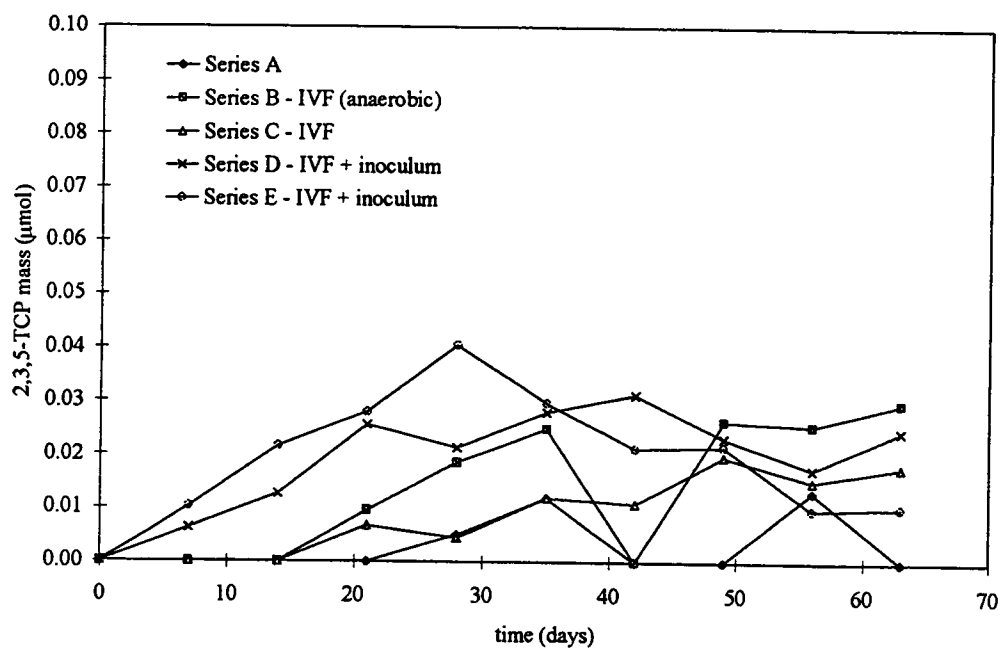
Figure 10. Appearance 2,3,5-TCP in bench-scale reactors from anaerobic transformation of 2,3,5,6-TeCP or 2,3,4,5-TeCP
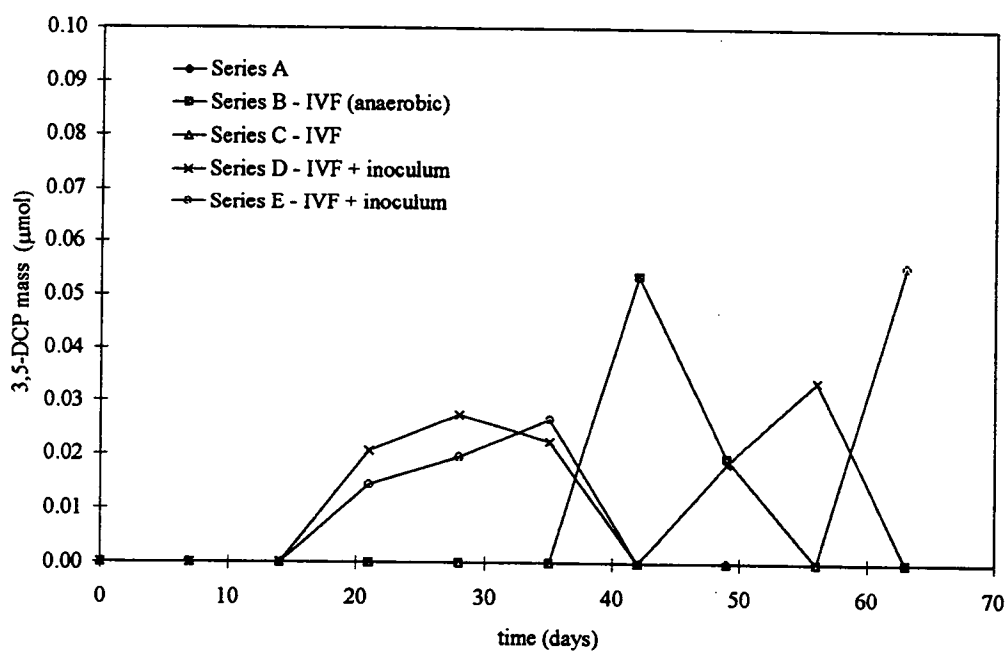
Figure 11. Appearance of 3,5-DCP in bench-scale reactors over nine fill and draw cycles

Chlorophenol Washout

One of the goals of fill and draw batch reactor technology was to treat contaminated soils and contaminated pore water without promoting soil washing. This was an important consideration in reactors where the mobility of chlorophenols was enhanced by the addition of anaerobic wastewater sludge supernatant. A comparison of the chlorophenol content of reactor fill solutions and subsequent drain solutions was used to determine the extent of soil washing that occurred each week, and over the nine week experiment duration. The change in chlorophenol mass in the aqueous phase over week-long cycles indicated the amount of chlorophenols added to or removed from reactors with each fill and draw cycle. The sum of these changes compared to the initial masses of chlorophenols in soil indicated the washout that occurred in each reactor series (Table 4). For example, at the conclusion of the fourth week of treatment, effluent treatment mixtures from reactor series A contained a total of 1.55 nmol of chlorophehnols (Series/Week A4). At the beginning of treatment, the fill mixture for series D reactors contained 2.58 nmoles of PCP. The effluent mixture at the conclusion of the first week of treatment contained 0.38 nmoles of PCP (D1).

The fill and draw process promoted nominal washout of PCP in bench-scale reactors, based on the initial mass in the soil-water system. The process resulted in 3.5 percent, 8.3 percent, 1.5 percent, and 1.5 percent losses of PCP in reactors A, B, C, and D respectively. In reactor series E, an overall increase in PCP mass of 2.8 percent resulted from fill and draw treatment with a PCP acclimated inoculum. Losses of more soluble 2,3,5,6-TeCP were more significant. Reactors A, B, and C lost 17.4 percent, 40.4 percent and 15.0 percent of their initial masses of 2,3,5,6-TeCP to washout over nine fill and draw cycles. Losses of 2,3,5,6-TeCP in inoculated reactors, D and E were, 2.1 percent and 32.5 percent, respectively. Reactors inoculated with wastewater sludge supernatant acclimated to chlorophenols showed significant changes of 2,3,4,5-TeCP in the aqueous phase. Series D reactors experienced a 21.9 percent gain in mass of 2,3,4,5-TeCP, while series E reactors lost 22.3 percent of their initial mass of 2,3,4,5-TeCP.

Table 4. Chlorophenols added/removed from bench-scale reactors due to fill and draw treatment (nmol)

Series/ Week	2,3,5 TCP	3,4,5 TCP	2,3,5,6 TeCP	2,3,4,5 TeCP	PCP	ΣCP
A0	NA	NA	NA	NA	NA	NA
A1	0/0	0/0	0/1.35	0/0	0/0.88	0/2.22
A2	0/0	0/0	0/0.74	0/0	0/1.00	0/1.74
A3	0/0	0/0	0/2.75	0/0	0/3.84	0/6.59
A4	0/0	0/0	0/0.83	0/0	0/0.71	0/1.55
A5	0/0	0/0	0/1.62	0/0	0/1.36	0/2.98
A6	0/0	0/0	0/0.43	0/0	0/0.46	0/0.89
A7	0/0	0/0	0/2.58	0/0	0/2.90	0/5.48
A8	0/0	0/0	0/7.92	0/0	0/2.09	0/10.00
A9	0/0	0/0	0/6.15	0/0	0/4.05	0/10.20
Σ	0/0	0/0	0/24.37	0/0	0/17.29	0/41.65
B0	NA	NA	NA	NA	NA	NA
B1	0/0	0/0	0/1.40	0/0	0/0.75	0/2.15
B2	0/0	0/0	0/0.66	0/0	0/0.40	0/1.06
B3	0/0	0/0	0/2.70	0/0	0/3.76	0/6.46
B4	0/0	0/0	0/3.61	0/0	0/2.62	0/6.23
B5	0/0	0/0	0/7.35	0/0	00/9.64	0/17.00
B6	0/0	0/0	0/3.66	0/0	0/5.63	0/9.27
B7	0/0	0/0	0/11.8	0/0	0/10.4	0/22.10
B8	0/0	0/0	0/23.1	0/0	0/4.55	0/27.60
B9	0/0	0/0	0/2.23	0/0	0/3.66	0/5.89
Σ	0/0	0/0	0/56.51	0/0	0/41.41	0/97.76

Table 4 (continued). Chlorophenols added/removed from bench-scale reactors due to fill and draw treatment (nmol)

C0	NA	NA	NA	NA	NA	NA
C1	0/0	0/0	0/1.38	0/0	0/1.08	0/2.46
C2	0/0	0/0	0/1.29	0/0	0/0.73	0/2.02
C3	0/0	0/0	0/0	0/0	0/0	0/0
C4	0/0	0/0	0/0.93	0/0	0/0.75	0/1.68
C5	0/0	0/0	0/0	0/0	0/0.30	0/0.30
C6	0/0	0/0	0/0	0/0	0/0.27	0/0.28
C7	0/0	0/0	0/0	0/0	0/2.92	0/2.92
C8	0/0	0/0	0/12.36	0/0	0/1.02	0/13.38
C9	0/0	0/0	0/4.98	0/0	0/0.46	0/5.44
Σ	0/0	0/0	0/20.94	0/0	0/7.53	0/28.48
D0	NA	NA	NA	NA	NA	NA
D1	0/0	0/0	0/0.45	0/0	2.58/0.38	2.58/0.83
D2	0/0	0/0	0/5.05	0/0	2.56/5.02	2.56/10.07
D3	0/0	0/0	0/1.23	0/0	3.03/1.15	3.03/2.38
D4	0/0	0/0	0/1.87	0/0	1.98/2.34	1.98/4.20
D5	0/0	0/0	0/2.43	0/0	0/2.03	0/4.46
D6	0/0	0/0	0/1.77	0/0	0/1.76	0/3.53
D7	0/0	0/0	0/2.34	3.06/0	0/2.89	3.06/5.23
D8	0/0	0/0	20.78/3.83	0/0	0/1.78	20.78/5.61
D9	0/0	0/0	0/4.88	0/0	0/0.40	0/5.29
Σ	0/0	0/0	20.78/23.85	3.06/0	10.15/17.75	33.99/41.6
E0	NA	NA	NA	NA	NA	NA
E1	0/0	0/0	0/0.21	0/0	13.94/0.21	13.94/0.43
E2	0/0	0/0	0/2.96	0/0	13.89/2.49	13.89/5.45
E3	0/0	0/0	3.83/6.28	0/3.15	15.60/5.72	19.43/15.15
E4	0/5.00	0/0	2.28/2.23	0/0	11.88/1.02	14.16/8.25
E5	0/3.41	0/0	0/5.92	4.68/7.33	7.44/7.65	12.12/24.32
E6	0/6.71	0/3.62	4.48/5.31	9.23/8.23	8.50/2.55	22.21/26.42
E7	0/0	0/0.95	4.65/7.16	7.75/1.79	4.37/7.53	16.77/17.43
E8	0/0	0/0	0/13.05	4.17/2.81	0/13.91	4.17/29.77
E9	0/0	0/0.66	0/17.66	0/5.64	0/20.71	0/44.68
Σ	0/15.12	0/5.23	15.24/60.78	25.83/28.95	75.62/61.79	116.90/171.89

Pilot-Scale Fill and Draw Reactor Study

The pilot scale reactor was operated for twenty three seven-day fill and draw cycles. Soil samples from treatment zones, and water samples from liquid mixing zones and treatment zone sampling wells were extracted and analyzed for chlorophenols weekly. Soil was fed a treatment mixture similar to that used for batch reactor series C. The mixture was composed of IVF and nutrients, but no inoculum. Because the soil in the pilot-scale reactor was saturated at the inception of the treatment process, the concentration of imitation vanilla flavoring in feed mixtures was raised from 100 mg/L COD to 150 mg/L COD. The molar concentrations of chlorophenols in saturated soil during the treatment process are shown in Figures 12 through 16.

Background Concentrations of Chlorophenols in Soil and Soil Conditions

The pilot-scale reactor treated approximately $3.7m^3$ of a contaminated heterogeneous sand and clay mixture. Based on five samples, the soil mixture contained an average of 7.1 mg/kg (27 μmol/kg) PCP ($\sigma = 0.42$), and 0.012 mg/kg (0.052 μmol/kg) 2,3,5,6-TeCP ($\sigma = 0.0018$). Treatment zones A and B contained soil removed from well depths ranging from 18 to 22 feet and included small quantities of gravel and cobble. Treatment zone C was filled with soil taken from a well depth of 15 to 18 feet and was composed mostly of clay and other fines, with very little gravel. The soil was completely saturated at the inception of the treatment procedure. Concentrations of chlorophenols in five samples of soil pore water averaged 0.82 mg/L (3.1 μmol/L) PCP and 0.20 mg/L (0.86 μmol/L) 2,3,5,6-TeCP. Soil conditions, including CEC, pH and organic content were identical to those measured as part of the bench scale study.

PCP & 2,3,5,6-TeCP Removal

Over 160 days of treatment, PCP levels in soil zones A and B were reduced by approximately 90 percent, from 27 μmol/kg to less than 3 μmol/kg (Figure 12). The bulk of PCP transformation occurred during the first week of treatment. PCP concentration in zone C was reduced by more than 50 percent, and ranged from 5 to 10 μmol/kg. The concentrations of 2,3,5,6-TeCP in zones A and B were reduced by approximately 80 percent over the first one hundred days of treatment with most of the removal occurring during the first four treatment cycles (Figure 13). Chlorophenol analyses of zone C soil showed varying concentrations of 2,3,5,6-TeCP over the 160 day treatment period, ranging from 0 μmol/kg at the conclusion of week three to 0.065 μmol/kg in week fifteen.
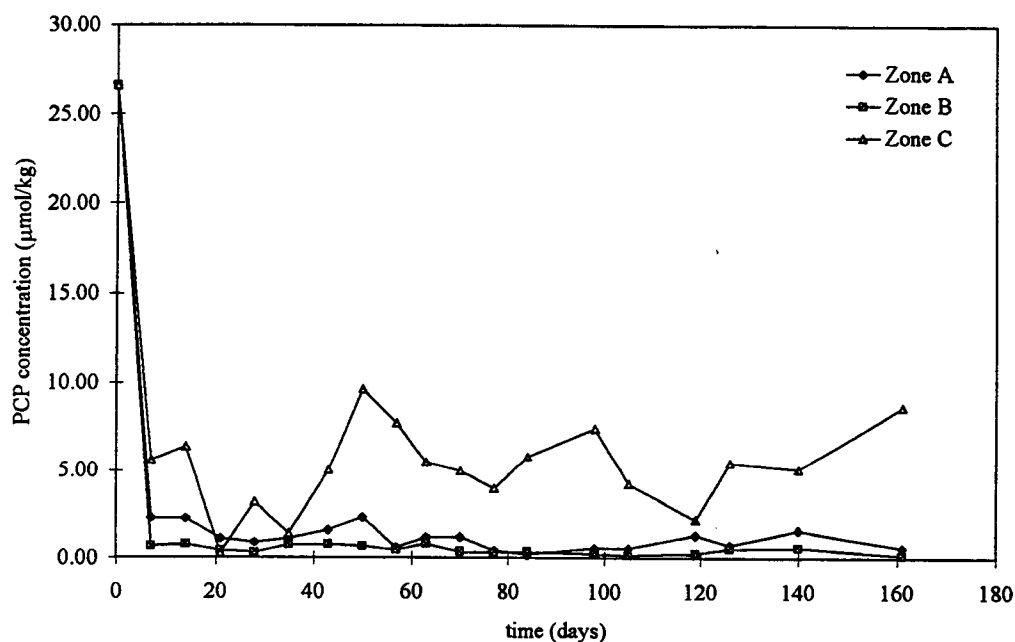
Figure 12. Concentrations of PCP in pilot-scale reactor soil treatment zones over 23 cycles
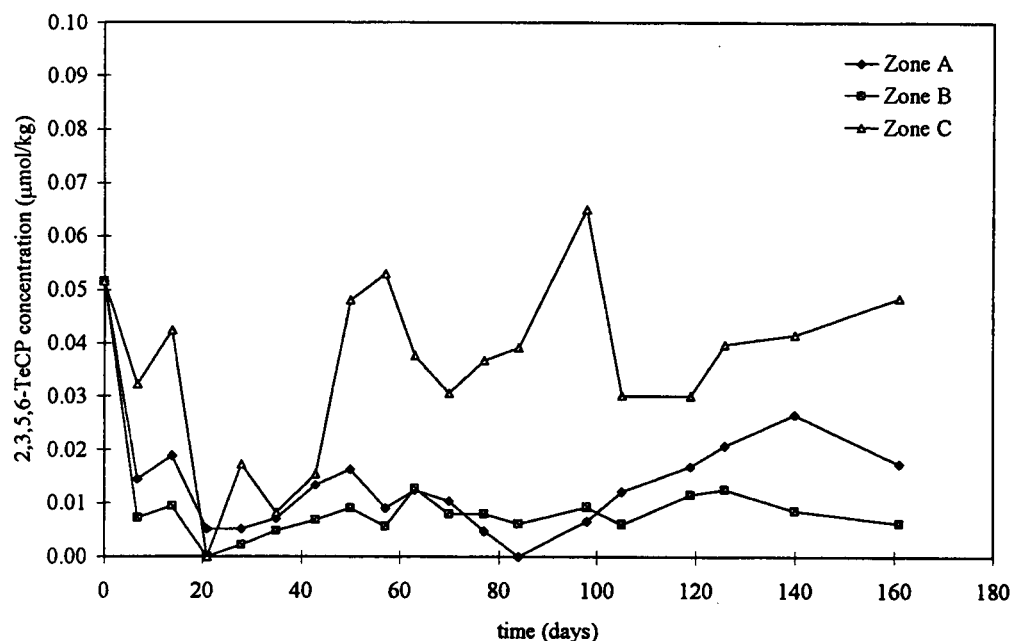
Figure 13. Concentrations of 2,3,5,6-TeCP in pilot-scale soil treatment zones over 23 cycles

Anaerobic PCP & 2,3,5,6-TeCP Transformation

The mass balance on chlorophenols indicated that soil treated in the pilot-scale reactor responded to treatment similarly to bench-scale reactor series C. PCP and 2,3,5,6-TeCP transformation in soils occurred almost entirely without the appearance of dechlorinated metabolites (Figure 14). The production of metabolites from anaerobic reductive dechlorination was sporadic in all treatment zones and accounted for less than approximately 10 percent of all PCP and 2,3,5,6-TeCP removal (by molar mass). Fill and draw treatment supported reductive dechlorination of PCP and 2,3,5,6-TeCP to 2,3,5-TCP in treatment zone C, and 3,4,5-TCP in treatment zones A, B and C (Figures 15 and 16).
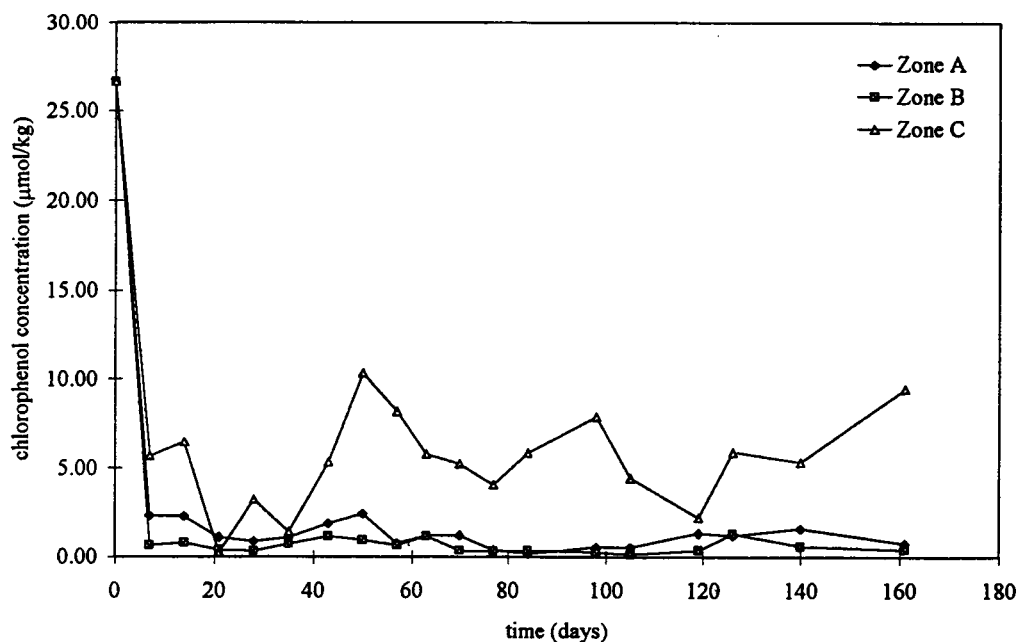
Figure 14. Mass balance of chlorophenols in pilot-scale reactor
Σ conc (PCP; 2,3,5,6-TeCP; 2,3,4,5-TeCP; 2,3,5-TCP; 3,4,5-TCP)
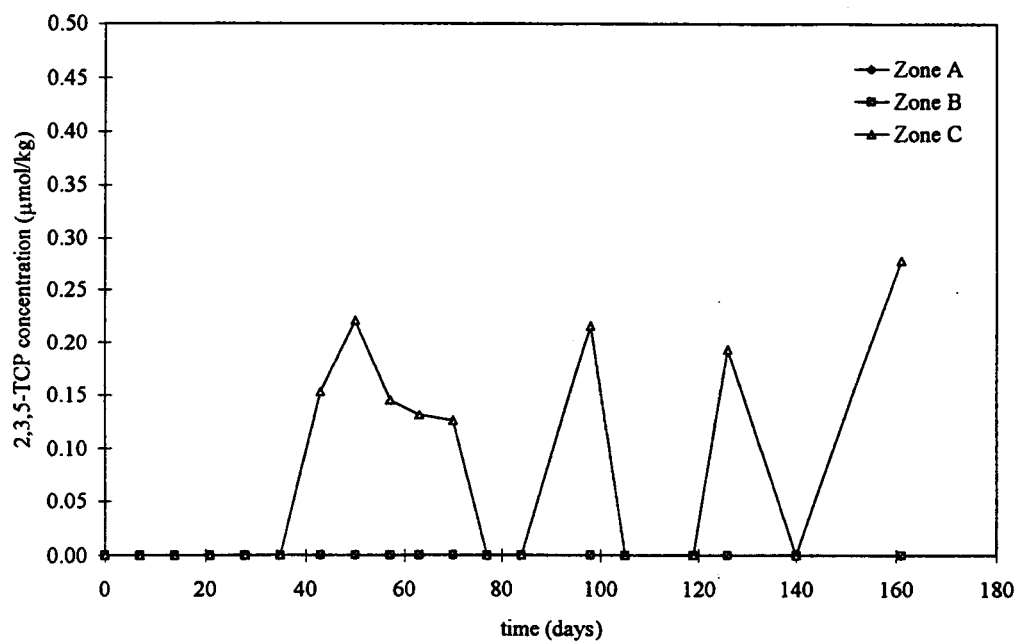
Figure 15. Production of 2,3,5-TCP in pilot-scale reactor soil treatment zones over 23 cycles
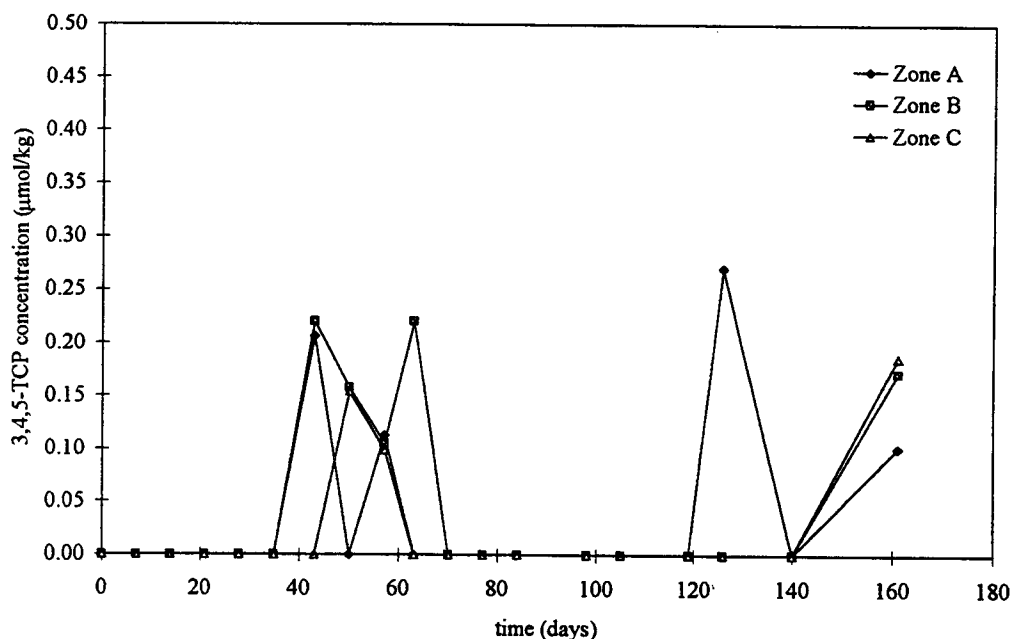
Figure 16. Production of 3,4,5-TCP in pilot-scale reactor treatment zones over 23 cycles

Chlorophenol Washout

As in the bench-scale reactors, fill and draw treatment in the pilot-scale reactor was successful at removing chlorophenols from soil without significant soil washing. To determine the extent of chlorophenol mobility in the pilot-scale reactor, reactor effluent from each fill and drain cycle was examined for PCP and lesser chlorinated phenols. The only significant appearance of chlorophenols in drain water occurred during the first five weeks of treatment. Through the fourth treatment cycle, 1.1 μmol 2,3,5,6-TeCP and 60.8 μmol PCP were removed from the reactor with fill and drain solutions. These losses comprised 0.29% of the 2,3,5,6-TeCP and 0.03% of the PCP present in the soil reactor at the inception of treatment (372 μmol 2,3,5,6-TeCP; 19187 μmol PCP). Based on an average of soil and drain water samples taken during the first four cycles, washout

contributed only 1 percent and 0.46 percent (by mass) to the overall removal of PCP and 2,3,5,6-TeCP, respectively, in the pilot-scale reactor (Table 5).

Table 5. Losses of PCP and 2,3,5,6-TeCP from soil (breakdown by soil treatment zone) and losses due to washout, during cycles 1-4

| | Removal from soil | | | |
| | mass (μmol) | | percent of initial mass | |
	PCP	2,3,5,6-TeCP	PCP	2,3,5,6-TeCP
A	32310	53	94	79
B	33748	61	98	91
C	104988	133	85	56
Σ	171046	247	89	66
	Removal in drain mixtures			
	mass (μmol)		percent of initial mass	
	PCP	2,3,5,6-TeCP	PCP	2,3,5,6-TeCP
Σ	61	1	0.04	0.40

Spatial Variability of Chlorophenols in Soil

PCP and 2,3,5,6-TeCP transformation during the treatment process varied by zone. The variability of chlorophenol degradation in the bioreactor was possibly due to the heterogeneity of soil and the effectiveness of the fill and draw batch process. To determine the extent of this variability, PCP and 2,3,5,6-TeCP concentrations were measured and compared with respect to horizontal and vertical position in the pilot-scale reactor. Samples taken at 30 cm intervals across soil treatment zone A at the conclusion of the fifth, sixth and seventh treatment cycles indicated no trend in the concentrations of chlorophenols over the area perpendicular to the treatment mixture flow field (Table 6).

Concentrations of chlorophenols also were evaluated with soil depth. At the conclusion of the fifth treatment cycle, soil samples were drawn from 60 cm and 120 cm depths at the same location in each treatment zone. Analyses of these samples were

inconclusive and could not ascertain the possibility of changes in biodegradation potential with vertical position in the pilot-scale reactor (Table 7).

Table 6. Variability of chlorophenol concentration with horizontal position in pilot-scale reactor soil treatment zone A (μmol/kg)

Distance from reactor wall (cm)	Week 5 2,3,5,6-TeCP	PCP	Week 6 2,3,5,6-TeCP	PCP	Week 7 2,3,5,6-TeCP	PCP
30	0.0068	0.63	0.001	0.18	0.0013	0.23
60	0.0023	0.75	0.0081	0.21	0.0008	0.18
90	0.0038	0.15	0.0007	0.11	0.0078	0.82
120	0.0072	0.57	0.0052	0.71	0.0031	0.62
150	0.0008	0.12	0.0081	0.41	0.0058	0.28
variance	0.0024	0.25	0.0032	0.19	0.0017	0.16
mean	0.0042	0.45	0.0046	0.32	0.0038	0.43
std dev.	0.0028	0.29	0.0036	0.24	0.003	0.28

Table 7. Variability of chlorophenols concentration with soil depth (μmol/kg)

Soil Zone	Depth (cm)	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A	60	ND	ND	0.0015	0.013	0.22
A	120	0.021	ND	0.0047	0.017	0.77
B	60	ND	0.016	0.0009	0.012	0.63
B	120	0.021	0.011	0.0049	0.017	0.81
C	60	0.022	0.015	0.0046	0.024	0.92
C	120	0.013	ND	0.0041	0.013	0.72

ND - none detected

Variability with horizontal and vertical position was inconsistent and was possibly due to soil heterogeneity and the preferential flow fields created by this condition.

Differences in biodegradation and chlorophenol content associated with vertical location could have been due to the method of fill solution delivery. The pilot-scale reactor was designed to deliver nutrients and imitation vanilla flavoring in an upflow manner. Soil at the top of the reactor was subject to a higher flux of fill mixtures, creating a higher concentration of electron donors and nutrients for chlorophenol transformation processes. Soil at the bottom of the reactor was more likely to remain anaerobic over the treatment period, creating a favorable environment for reductive dechlorination. The extreme range of chlorophenols indicated by the horizontal sampling study suggests that variability due to horizontal position is more likely due to soil heterogeneity than reactor operation.

Imitation Vanilla Flavoring Surfactant Microcosm Study

Imitation vanilla flavoring was added to weekly fill solutions at 100 mg/L COD to serve as an electron donor for reductive dechlorination and as a substrate for cell growth. The imitation vanilla flavoring used in this analysis contained four primary components: propylene glycol, ethyl vanillin, guaiacol, and sodium benzoate (Figure 17). Three of the components, guaiacol, benzoate, and propylene glycol are suitable primary substrates for aerobic

Propylene Glycol
(1,2-Propanediol)

Ethyl Vanillan
(3-Ethoxy-4-Hydroxybenzaldehyde)

Guaiacol
(2-Methoxy Phenol)

Sodium Benzoate
(Benzoic Acid)
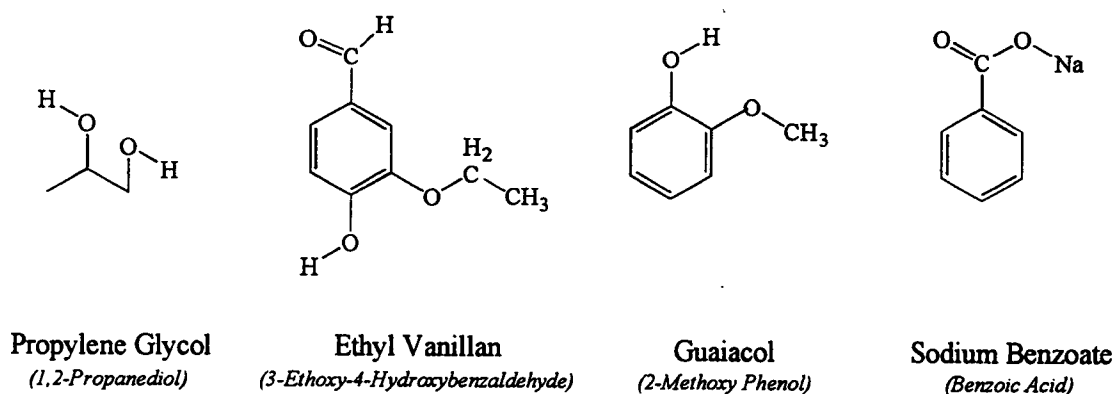
Figure 17. Structures of compounds composing imitation vanilla flavoring

degradation of some chlorophenols (Kaslik et al., 1996). A comparison of anaerobic metabolites present in reactor series A and C showed that imitation vanilla flavoring was an active electron donor for anaerobic reductive dechlorination as well (Figures 6 through 11). The presence of imitation vanilla flavoring was a critical factor in determining the transformation mechanisms in upflow reactors. Reactors containing no imitation vanilla flavoring showed no anaerobic transformation products.

Apart from facilitating biotransformations, literature suggested that imitation vanilla flavoring may also enhance the mobility of chlorophenols in saturated soil systems. Aromatic compounds have been shown to act as surfactants in saturated soils containing chlorophenols (Khodadoust et al., 1994). A microcosm study was conducted to determine the potential of imitation vanilla flavoring as a surfactant on soil from the McFarland-Cascade site (Appendix H). Soils were dosed with imitation vanilla flavoring at concentrations ranging from 0 to 470 mg/L (as COD). The presence of imitation vanilla flavoring resulted in slightly lower masses of PCP and 2,3,5,6-TeCP in the aqueous phase over the 96 hour experiment duration, when compared to reactors without IVF (Figures 18 and 19). At the termination of the analysis, microcosms exhibited an inverse correlation between the concentration of imitation vanilla flavoring and the mass of chlorophenols in the aqueous phase (Table 8). Masses of 2,3,5,6-TeCP and PCP in solution were lowest in microcosms treated with 470 mg/L imitation vanilla flavoring, and increased as the inoculation concentration approached 0 mg/L COD. No reductive dechlorination metabolites were found in the aqueous phase.
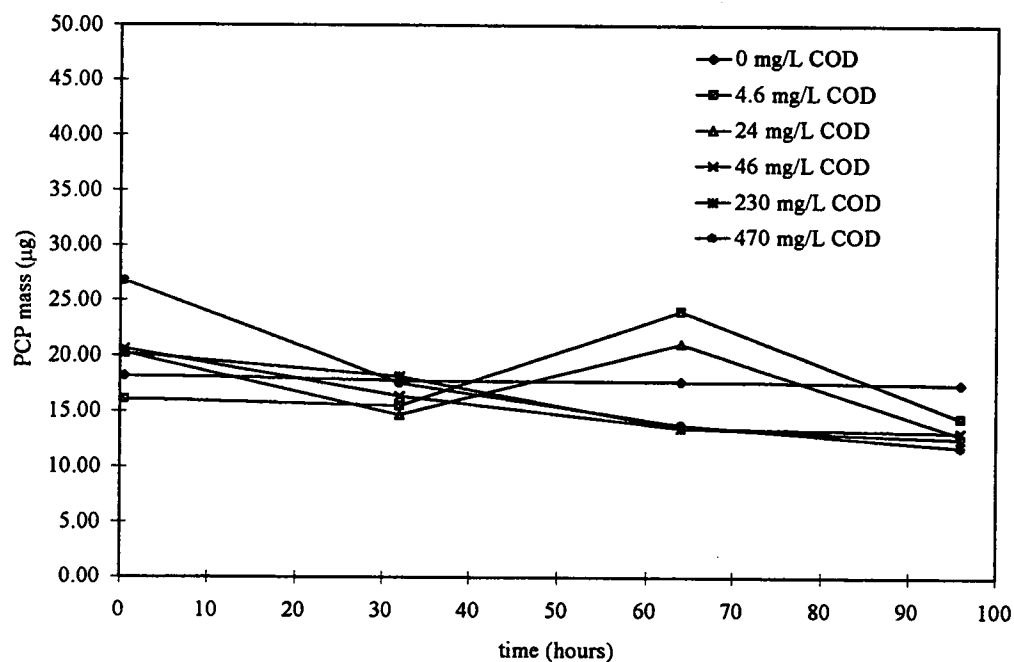
Figure 18. PCP mass in aqueous form in soil/water microcosms treated with IVF
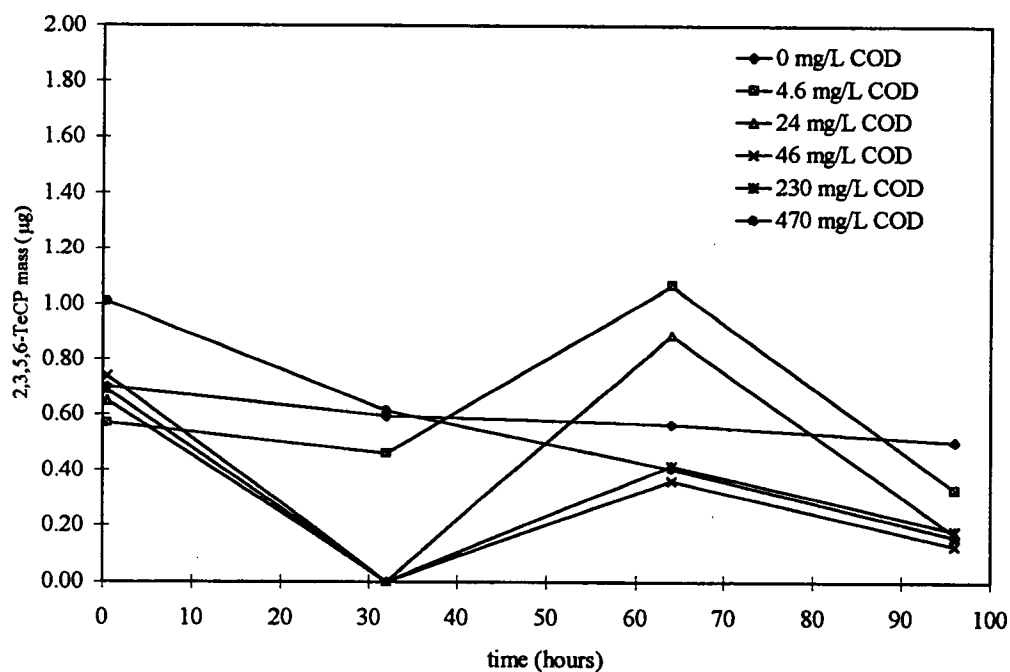
Figure 19. 2,3,5,6-TeCP mass in aqueous form in soil/water microcosms treated with IVF

Table 8. Imitation vanilla flavoring surfactant study - chlorophenols remaining in soil/water microcosms (µg)

Imitation Vanilla mg/L COD	mass in soil at 0 hrs		mass in liquid at 96 hrs		% in liquid	
	PCP	2,3,5,6-TeCP	PCP	2,3,5,6-TeCP	PCP	2,3,5,6-TeCP
0	81.4	20.1	17.6	0.51	21.6	2.53
4.6	81.4	20.1	14.5	0.33	17.8	1.64
24	81.4	20.1	13.0	0.17	16.0	0.84
46	81.4	20.1	13.2	0.13	16.2	0.65
230	81.4	20.1	12.6	0.18	15.9	0.89
470	81.4	20.1	11.9	0.16	14.6	0.79

Discussion - Evaluation of Fill and Draw Batch Reactor Technology

The reactor treatment mixtures were created to evaluate the effects of a number of conditions on the fill and draw batch process. Different reactor series were devised to assess and compare the biochemical processes promoted by fill and draw treatment in the presence of imitation vanilla flavoring, wastewater sludge supernatant, and an anaerobic feed mixture. The composition of reactors and the number of reactor series were limited by the desire to test a specific treatment process on soil. Reactor series A was designated as the base series and was fed only deionized water. Sterilized control reactors were not used in this analysis because their organic content would not accurately represent soil that might be treated using sequencing batch reactors. If accomplished through heat treatment, soil sterilization might significantly affect chlorophenol sorption and desorption, and soil hysteresis. Reactors B and C were configured to compare chlorophenol removal in the presence of similar feed mixtures maintained under anaerobic or sequential aerobic/anaerobic conditions. Series A and series C were used to evaluate the potential of IVF as an electron donor and primary substrate. Series C, D and E were used to evaluate the transformation and removal of chlorophenols in the presence of an acclimated biomass.

Effect of Imitation Vanilla Flavoring Addition

Laboratory analyses indicated that IVF served as an electron donor for reductive dechlorination of PCP and 2,3,5,6-TeCP in soils without significantly solubilizing these chlorophenols. This was true for bench-scale (Table 3), pilot-scale (Table 5), and microcosm (Table 8) experiments. A comparison of chlorophenol removal and transformation in bench-scale reactor series A and C was made to evaluate the reductive dechlorination of PCP in the absence (series A) and presence (series C) of an electron donor. PCP and 2,3,5,6-TeCP were removed similarly in series A and C (Figure 20a). Reductive dechlorination products of PCP and 2,3,5,6-TeCP were not observed in

significant quantities in series A, but did appear in effluent treatment solutions from reactor series C (Figure 20b).

The surfactant microcosm test suggested that there was no enhancement of chlorophenol mobility in bench scale reactors or in the pilot-scale reactor from the addition of IVF in the range of 46 mg/L COD to 230 mg/L COD. In microcosms containing IVF at 46 mg/L COD, masses of PCP and 2,3,5,6-TeCP found in solution were 13.2 µg and 0.13 µg, respectively (Table 8). PCP and 2,3,5,6-TeCP levels in the aqueous phase of microcosms treated with 230 mg/L COD were similar; 12.6 µg and 0.18 µg, respectively. These masses were slightly higher than series A, treated with no IVF, which showed 17.6 µg of PCP and 0.51 µg 2,3,5,6-TeCP in solution.

Effect of Anaerobic vs. Aerobic Treatment Mixtures

The oxygen content of feed mixtures had a nominal effect on the removal of 2,3,5,6-TeCP and PCP in bench-scale reactors treated with IVF. Reactors fed a deaerated feed solution (series B) achieved 75.3 percent removal of PCP and 79.5 percent removal of 2,3,5,6-TeCP (Table 3). When oxygen was not purged from feed mixtures, removal of PCP and 2,3,5,6-TeCP totaled 70.9 percent and 82.2 percent, respectively (series C). The presence of an anaerobic feed mixture facilitated reductive dechlorination of PCP and 2,3,5,6-TeCP to 3,5-DCP in series B reactors, while oxygenated treatment mixtures achieved reductive dechlorination to 3,4,5-TCP and 2,3,5-TCP (Figure 21). Deoxygenated treatment mixtures may have been more successful at transforming tetrachlorophenols. The levels of 2,3,5-TCP and 3,4,5-TCP measured in series B were noticeably higher than those measured in series C.
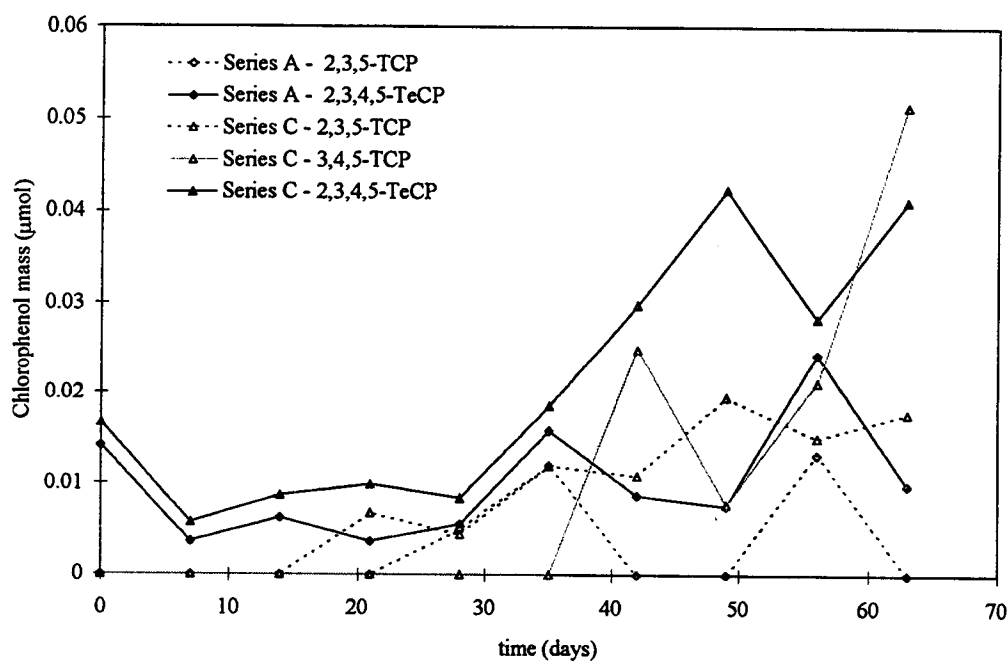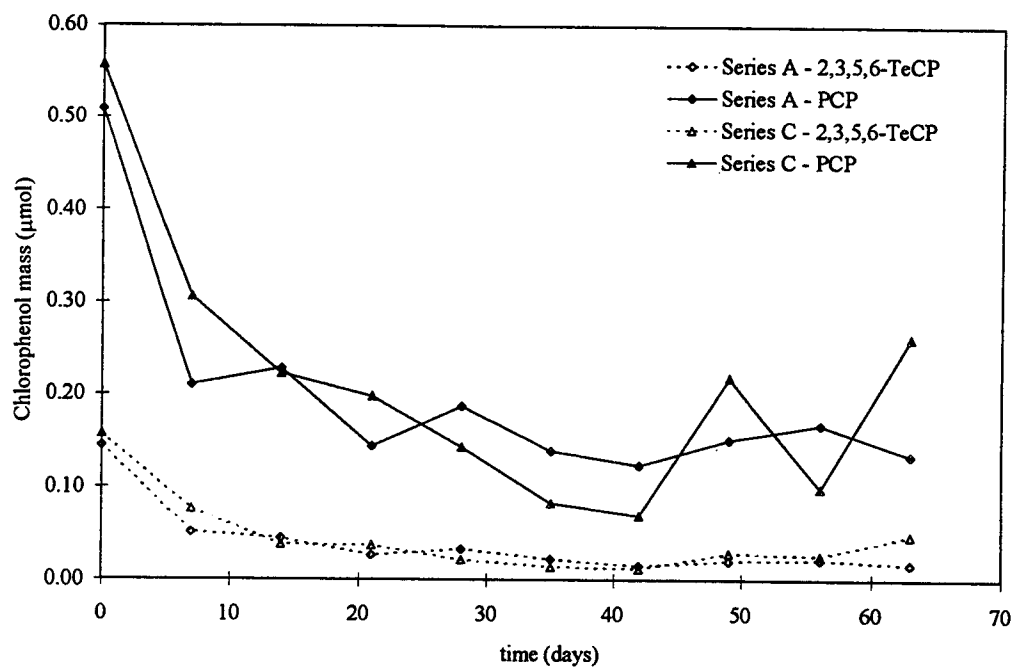
Figure 20a. PCP and 2,3,5,6-TeCP removal in bench-scale reactor series A and C
Figure 20b. Dechlorination metabolites in bench-scale reactor series A and C
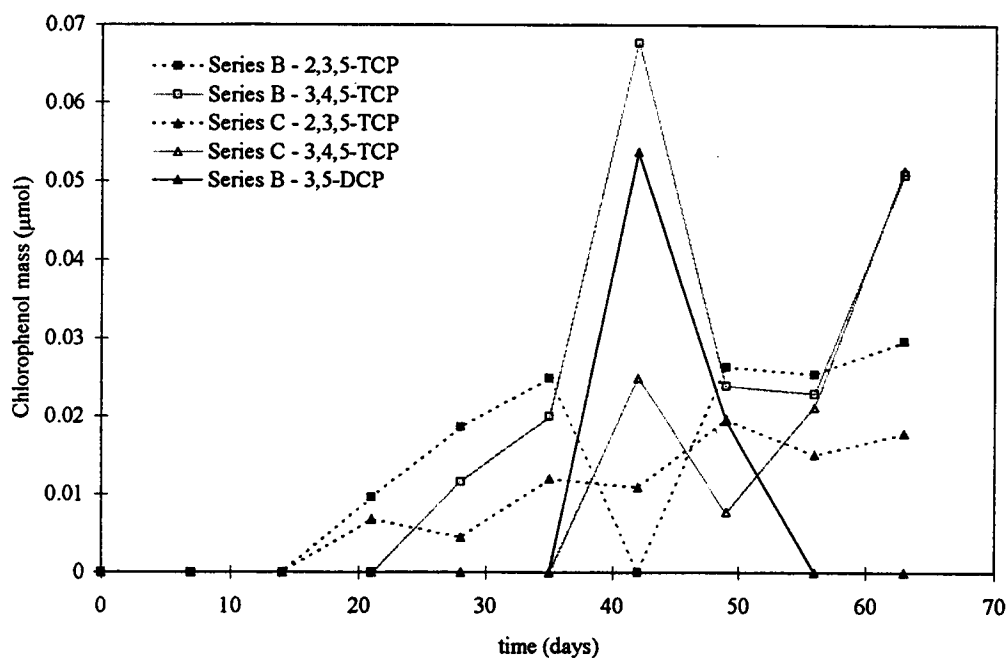
Figure 21. Production of reductively dechlorinated metabolites 2,3,5-TCP, 3,4,5-TCP and 3,5-DCP in bench-scale reactor series B and C

Effect of Anaerobic Wastewater Sludge Supernatant Addition

Inoculated anaerobic wastewater sludge supernatant was added to bench-scale reactor series D and E to increase the presence of chlorophenol-degrading bacteria in the saturated soil systems. The result was slightly enhanced transformation by reductive dechlorination and a closed reactor system in which nominal losses of chlorophenols occurred due to washout of chlorophenols in treatment mixtures. In comparison to un-inoculated reactors treated with IVF (series C), reactor series D demonstrated no greater removal of 2,3,5,6-TeCP (Figure 22). Series E reactors, which contained four times the concentration of sludge supernatant found in series D reactors, was no more successful at removing PCP. Because of the range of data standard deviation, comparing removal in reactors series C, D and E is difficult (Table 3). It is clear, however that reactors containing wastewater sludge supernatant demonstrated
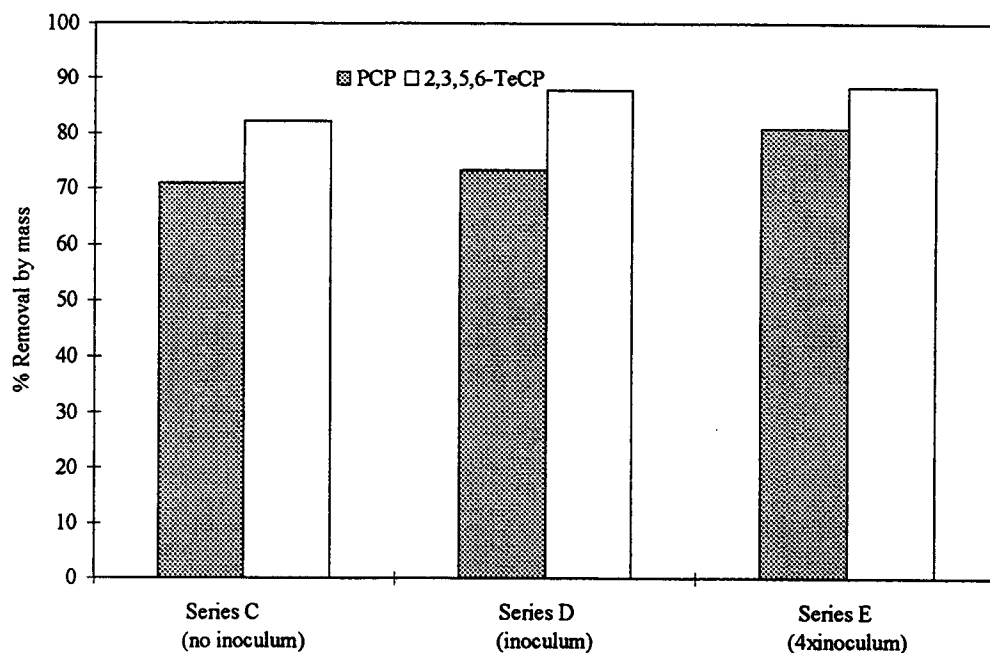
Figure 22. Chlorophenol removal efficiencies in bench-scale reactor series C, D and E

more complete anaerobic transformation of PCP and 2,3,5,6-TeCP compared to reactors containing no inoculum. Inoculated reactors (series D and E) were able to promote reductive dechlorination of PCP and 2,3,5,6-TeCP to 3,5-DCP over nine week experiment duration, while un-inoculated reactors produced only trichlorophenols, 2,3,5-TCP and 3,4,5-TCP (Appendix P, Figures 25 through 29). Feed solutions for reactors series D and E contained up to 4.16 µg PCP and 4.82 µg 2,3,5,6-TeCP, both complexed with organic matter and dissolved in water. Adding inoculated sludge increased the chlorophenol mass in the aqueous phase of these reactors. Additions of chlorophenols increased concentrations in reactor series D and E reactors such that they experienced no net loss of chlorophenols from fill and drain procedures (Table 9). The weekly change in chlorophenol mass from inoculated treatment mixtures was minimal. Measured as percent of the total mass in the soil/water system, variations in the total mass of chlorophenols due

to the inoculum ranged from -0.37 to +2.58 percent in series D and -7.59 to +2.30 percent in series E (Table 9).

Table 9. Cumulative change in mass of chlorophenols in sludge-inoculated bench-scale reactors due to fill and drain process (nmol)

Reactor Series/Cycle	drained	added	added - drained	% change of CPs
D0	0.00	0.00	0.00	0.00
D1	0.83	2.58	1.75	0.30
D2	10.07	2.56	-7.51	-1.28
D3	2.38	3.03	0.65	0.11
D4	4.20	1.98	-2.22	-0.38
D5	4.46	0	-4.46	-0.76
D6	3.53	0	-3.53	-0.60
D7	5.23	3.06	-2.17	-0.37
D8	5.61	20.78	15.17	2.58
D9	5.29	0.00	-5.29	-0.90
E0	0.00	0.00	0.00	0.00
E1	0.43	13.94	13.51	2.30
E2	5.45	13.88	8.43	1.43
E3	15.15	19.43	4.28	0.73
E4	8.25	14.16	5.91	1.00
E5	24.32	12.12	-12.20	-2.07
E6	26.42	22.21	-4.21	-0.72
E7	17.43	16.77	-0. 66	-0.11
E8	29.78	4.17	-25.61	-4.35
E9	44.67	0.00	-44.67	-7.59

The primary drawback to treatment with inoculated sludge supernatant was the creation of chlorophenol-contaminated effluent solutions. Any full-scale application of fill and draw technology must be designed to compensate for the production of process byproducts,

including hazardous wastes. Drain solutions from each reactor were collected and examined for chlorophenol content at the conclusion of the nine-week long treatment cycle. Analyses show that drain solutions contained chlorophenols at concentrations below the Universal Treatment Standards for process wastewater (Table 10). Concentrations of chlorophenols in drain solutions were far below contaminant solubilities, suggesting that treating soil with a higher content of PCP and 2,3,5,6-TeCP via fill and draw reactors may require secondary treatment for drain solutions or the use of an inoculum free of chlorophenols.

Table 10. Concentrations of chlorophenols in bench-scale reactor effluent (µg/L)

Reactor Series	volume (mL)	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A	979	0.00	0.00	5.78	0.00	4.73
B	1103	0.00	0.00	11.92	0.00	10.00
C	978	0.00	0.00	4.97	0.00	2.05
D	951	0.00	0.00	5.82	0.00	4.98
E	888	3.36	1.16	15.91	7.56	18.54
UTS[1]				30	30	89

[1]Universal Treatment Standards USEPA Fed. Reg. Sec 268.48 v61, no. 68 pp 15565-15660.

Transformation of Soil-Bound Contaminants vs. Water Borne Contaminants

One of the goals of this study was to treat chlorophenol-contaminated soil without promoting soil washing. Adding sludge to reactors may have enhanced the solubility of chlorophenols in the saturated soil system. One of the concerns in adding inoculated wastewater sludge supernatant to soil reactors was that reductive dechlorination would occur for the aqueous phase chlorophenols present in the inoculum, leaving the soil-bound contaminants untreated. To verify the removal and transformation of chlorophenols

sorbed to soils, it was essential to evaluate the transformation of chlorophenols in un-inoculated reactors and the timeliness of the appearance of anaerobic metabolites of PCP and 2,3,5,6-TeCP in both un-inoculated and inoculated reactors. Chlorophenol data from fill and drain solutions and soil samples taken from bench-scale reactors indicated that most of the transformation of chlorophenols originated from PCP and 2,3,5,6-TeCP sorbed to soil or complexed in soil pore water.

In reactor series B and C, dechlorinated metabolites were found at quantities and during specific treatment cycles that indicated their origin was soil-bound PCP and 2,3,5,6-TeCP. Treatment mixtures for reactor series B and C contained no wastewater sludge, yet both reactor series demonstrated reductive dechlorination of PCP and 2,3,5,6-TeCP to trichlorophenols (Figures 9 and 10). The possibility of trichlorophenols being generated from PCP and tetrachlorophenols washed from soils was not likely. Although trace levels of PCP and 2,3,5,6-TeCP were found in drain solutions, concentrations were usually insufficient to generate molar masses of 2,3,5-TCP and 3,4,5-TCP found in soil samples (Table 11).

Table 11. Mass of 2,3,5,6-TeCP and anaerobic metabolites (nmol) in bench-scale reactors

time, days	mass in soil				mass in liquid	
	2,3,5- TCP		3,4,5- TCP		2,3,5,6- TeCP	
	series B	series C	series B	series C	series B	series C
0	0.00	0.00	0.00	0.00	0.00	0.00
7	0.00	0.00	0.00	0.00	1.39	1.38
14	0.00	0.00	0.00	0.00	0.66	0.85
21	9.63	6.77	0.00	0.00	2.41	0.00
28	18.61	4.41	11.54	0.00	0.00	0.00
35	24.90	11.90	19.92	0.00	0.00	0.00
42	0	10.92	67.63	24.91	0.00	0.00
49	26.34	19.63	23.92	7.71	0.00	0.00
56	25.42	15.13	22.92	21.12	17.81	8.46
63	29.61	17.81	50.82	51.44	0	1.65

An example of this relationship occurred during the first three treatment cycles, during which 2,3,5,6-TeCP measured in the aqueous contents of series B and C measured a maximum of 2.41 nmol and 1.38 nmol, respectively. Levels of 2,3,5-TCP in soil were measured at 9.63 nmol and 6.77 nmol in reactors B and C. This pattern continued through the following four cycles, when 2,3,5-TCP was generated in soil samples without any measurable 2,3,5,6-TeCP in the effluent or influent liquid treatment mixtures. The production of 3,4,5-TCP was similar. 3,4,5-TCP appeared in un-inoculated reactor soil samples in cycles five through nine, yet it's parent compound, 2,3,4,5-TeCP never appeared in drain solutions.

In sludge-inoculated reactors D and E, the appearance of trichlorophenols was a direct result of reductive dechlorination of PCP and 2,3,5,6-TeCP sorbed to soil or complexed with soil pore water. This conclusion was based on the timeliness of the appearance of parent compounds in drain solutions and the molar quantities of metabolites found in soil samples. 2,3,4,5-TeCP was introduced to reactor series D and E fill solutions as part of the chlorophenol-acclimated inoculum during the seventh and third fill and drain cycles, respectively. 2,3,5-TCP and 3,4,5-TCP appeared in soil samples from D and E series reactors by the third week of reactor operation, but never appeared in drain solutions. This suggested that 2,3,5-TCP and 3,4,5-TCP originated from soil bound tetrachlorophenols and remained sorbed to soil. 2,3,5,6-TeCP was measured in fill and drain solutions prior to the appearance of 2,3,5-TCP, suggesting that 2,3,5-TCP could have been generated from *ortho* dechlorination of aqueous 2,3,5,6-TeCP. This possibility is not probable. In inoculated reactor series, molar quantities of 2,3,5,6-TeCP appeared in drain solutions at levels which were insufficient to generate molar quantities of 2,3,5-TCP found in saturated soil during the first three weeks of treatment.

Removal and Transformation of PCP and 2,3,5,6-TeCP

Transformation of 2,3,5,6-TeCP and PCP in bench-scale and pilot-scale reactors was expected to occur under both anaerobic and aerobic conditions, but more readily in

anaerobic soil conditions (Boyd et al., 1990). Our expectation was that by alternating aerobic and anaerobic conditions we would remove the bulk of chlorophenols by oxidation, leaving the more recalcitrant contaminants to be transformed anaerobically. Fill and draw batch reactors were operated as flooded soil systems. Flooding soil results in a rapid decrease in oxygen content of soil pore water and an increase in CO_2 from microbial respiration. This creates an anaerobic environment in which chlorophenol biodegradation is dominated by reductive dechlorination (Sposito, 1989). Experimental results suggested that chlorophenols were transformed by anaerobic reductive dechlorination, but also by another unknown method. Examination of the mass balances indicated that in both bench and pilot-scale experiments, (Figures 21 and 22) a decrease in PCP and 2,3,5,6-TeCP occurred over the first two fill cycles without proportionate increases in dechlorinated metabolites. Clearly, dechlorination was not the dominant factor in removal of PCP and 2,3,5,6-TeCP from each bench-scale reactor or soil treatment zone.
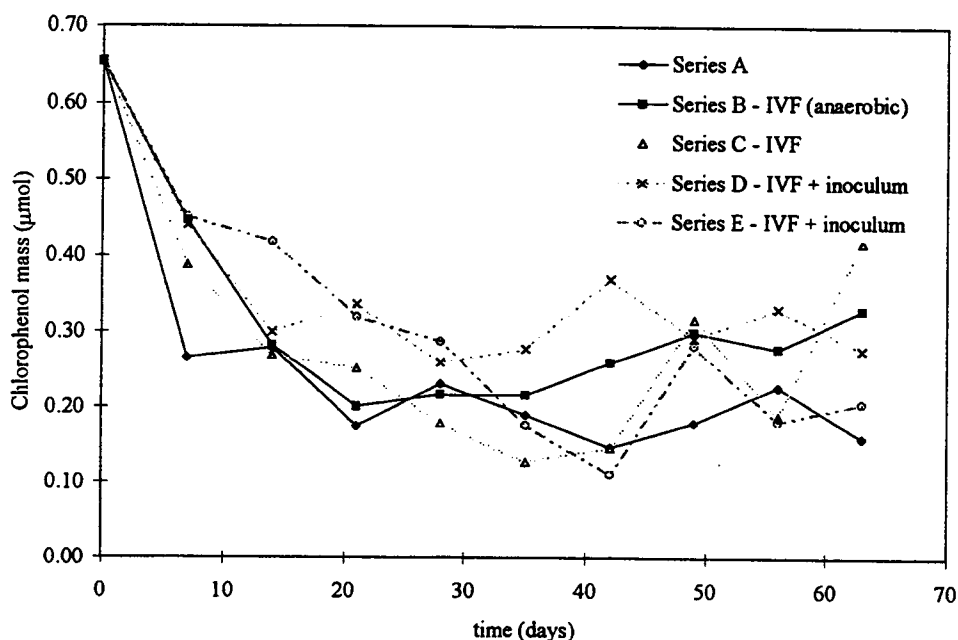
Figure 23. Mass balance on chlorophenols in saturated soil in bench-scale reactors
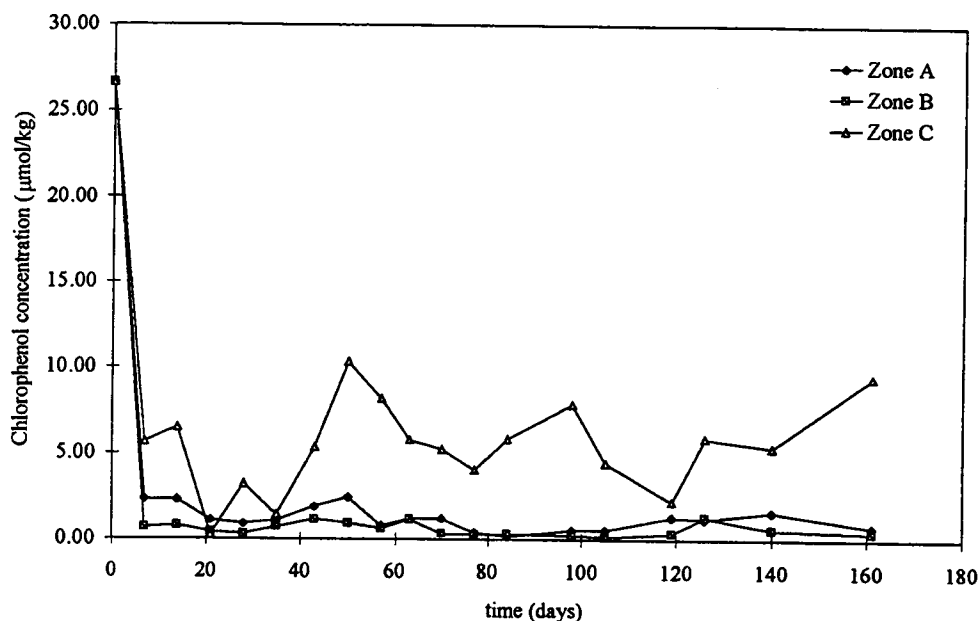
Figure 22. Mass balance on chlorophenols in saturated soil in pilot-scale reactor

Due to sampling variability, the extent of anaerobic transformation in the pilot-scale reactor was impossible to quantify, however, examination of the mass balance clearly showed that during the first six weeks, over 50 percent of PCP and 2,3,5,6-TeCP were removed from each soil treatment zone. This removal occurred without the observation of dechlorinated metabolites in soil, soil pore water, or recycled liquid treatment mixtures.

Several mechanisms for removal of PCP and 2,3,5,6-TeCP were possible, including biotransformation, volatilization and chemical complexation. A review of approximately two hundred articles showed no information regarding abiotic transformations of chlorophenols in flooded soils. Although aerobic metabolites of chlorophenols were not measured as part of this analysis, removal of PCP and 2,3,5,6-TeCP that could not be accounted for in the production of anaerobic metabolites was expected to be due to aerobic processes.

The possibility of aerobic transformation in bench-scale reactors was supported by a correlation between the appearance of anaerobic metabolites and the theoretical oxygen

content in soil reactors. Both oxygen dissolved in soil water, and molecular oxygen are known to promote aerobic metabolism of chlorophenols (Boyd et al., 1989). In bench-scale reactors, oxygen available from feed mixtures was minimal. The treatment mixtures for reactor series A contained a maximum of 385 µg of dissolved oxygen per cycle (based on the solubility of oxygen in water at 20°C). Reactor series B through E were treated with solutions containing only trace levels of dissolved oxygen and IVF at 100 mg/L as COD (3.5 mg/cycle), yet all demonstrated losses of chlorophenols without generating dechlorinated metabolites. Any possible aerobic transformation was likely supported by the oxygen entrained in soil at the initiation of treatment. Most of the PCP and 2,3,5,6-TeCP removal without dechlorination metabolites occurred during the first two treatment cycles when the oxygen content of bench-scale reactors was highest. During the first two cycles, oxygen was being flushed from the reactors by the incoming treatment mixtures, yet pockets of dried soil were likely to remain undisturbed. The proposed aerobic transformation in the pilot-scale reactor was similarly correlated with oxygen content. PCP and 2,3,5,6-TeCP removal without dechlorination metabolites occurred after the completely saturated soil of the reactor was initially drained and re-filled with treatment mixture.

Transformation and Mass Transfer Limitations

In bench-scale reactors, the presence of recalcitrant chlorophenols and the pattern of transformation over the course of treatment suggested that removal of PCP and 2,3,5,6-TeCP from soil was mass transfer limited. Bench-scale reactors were sucessful at removing PCP and 2,3,5.6-TeCP from saturated low-permeability soil, but as in many cases where soil has been exposed to contaminants over long periods of time, complete removal did not occur. Some chlorophenols remained entrained in the saturated soil system, unaffected by the treatment process. In reactor series A through E, between approximately 12 percent and 30 percent of PCP and 12 percent to 20 percent of 2,3,5,6-TeCP remained in soil through nine weeks of treatment. Removal in bench-scale reactors

occurred predominantly during the first four weeks of treatment (Figure 6 and Figure 7). The masses of PCP and 2,3,5,6-TeCP measured in each reactor similar from the fourth through ninth treatment cycle, suggesting that the bioavailability of PCP and 2,3,5,6-TeCP was limited.

Removal Kinetics

The rates of removal of chlorophenols in soil systems can be modeled using standard reaction kinetics. Reaction kinetic modeling is used to determine rate constants to represent several operational factors in soil reactor systems, including sorption/desorption phenomena, and biodegradation. Data from the first four treatment cycles were used to determine the reaction kinetics of the bench-scale reactors. This data suggested that the removal of PCP and 2,3,5,6-TeCP from bench-scale reactor series B through E followed first order kinetics (Figure 6 and Figure 7). The first order kinetic models for bench-scale reactor operation appear in Appendix P (Figures 46 through 55). The removal rate constants for PCP ranged from .047 day^{-1} to .055 day^{-1}. Rate constants for 2,3,5,6-TeCP varied from .70 μmol/day to .072 μmol/day. The removal and transformation of soil contaminants in series A did not resemble zero, first or second order kinetics.

Summary and Conclusions

Fill and Draw Batch Reactor Evaluation

In the past, *ex-situ* treatment of large quantities of chlorophenol-contaminated soil has been conducted mostly by landfarming or similar techniques. Landfarming requires the use of costly earth moving equipment and a substantial amount of space. At industrial complexes where space and equipment are readily available, and the volume of contaminated soil is extremely large, landfarming is a viable treatment method. In wood treatment facilities it is sometimes necessary to treat smaller-sized quantities of contaminated soil. Once excavated for *ex-situ* treatment, soils become a commingled mess of several different soil and gravel types . Because of their heterogeneous nature, soil mixtures can be highly impervious to fluids. Although the use of bioreactors for ex-situ treatment of chlorophenol contaminated soils has usually been limited to laboratory-scale endeavors, a self-contained bioreactor designed to treat these smaller volumes or low-permeability soil would appeal to facility owners and operators.

The objective of this study was to design, build and implement a pilot-scale fill and draw batch reactor capable of bioremediating 3.7 cubic meters of low permeability, chlorophenol-contaminated soil from a wood treatment facility. The reactor was designed to infuse soil with a liquid treatment mixture, promoting aerobic respiration and reductive dechlorination under sequential aerobic/anaerobic conditions. A concurrent bench-scale study was conducted to optimize the remediation process by comparing the effects of augmenting treatment mixtures with imitation vanilla flavoring as an electron donor and primary substrate, and the supernatant from anaerobic wastewater sludge. Chlorophenol removal from soil in the presence of an anoxic treatment mixture was also evaluated.

The results of the pilot-scale study indicated that treatment via the fill and draw process, using a recycled mixture containing imitation vanilla flavoring, resulted in approximately 90 percent removal of PCP and 80 percent removal of 2,3,5,6-TeCP (by mass) from soil treatment zones A and B, and 50 percent removal of PCP in soil treatment

zone C. Removal of PCP and 2,3,5,6-TeCP from contaminated soil occurred via anaerobic reductive dechlorination but was dominated by some unknown, probably aerobic microbial process. The transformation and removal of PCP and 2,3,5,6-TeCP occurred without washing chlorophenols from soils.

Bench-scale fill and draw batch reactors were successful at removing between 53 and 81 percent of PCP and 70 to 97 percent of 2,3,5,6-TeCP of from contaminated soil using treatment solutions of varied composition. A microcosm study showed that in addition to serving as an electron donor for reductive dechlorination, imitation vanilla flavoring did not enhance the solubility of chlorophenols in treatment mixtures. The addition of anaerobic wastewater sludge supernatant to treatment mixtures resulted in more complete anaerobic transformation of chlorophenols. As in the pilot-scale reactor, only small levels of anaerobic reductive dechlorination products were observed in bench-scale reactors. Treating soil with de-oxygenated feed mixtures resulted in more complete removal of chlorophenols and increased the rate of production of dechlorinated metabolites. Chlorophenols removed from bench-scale reactors originated almost exclusively from soil-bound contaminants.

The objective of this study was to develop and demonstrate a bioreactor capable of bioremediating 3.7 cubic meters of low permeability, saturated soil, contaminated with PCP and 2,3,5,6-TeCP. Remediation was to occur without soil washing. Results indicated that the pilot-scale reactor was successful at reducing PCP and 2,3,5,6-TeCP mass in soil without increasing contaminant solubility, using a feed mixture containing only imitation vanilla flavoring and a mixture or nutrients and vitamins. Bench-scale tests indicated that significant removal of chlorophenols could be achieved without soil washing, using a variety of treatment mixtures, including tap water.

Engineering Significance

The pilot-scale fill and draw reactor offers an viable alternative to other *ex-situ* remediation techniques including landfarming and more complex bioreactors. Fill and

draw batch bioreactors are a simple and effective means of treating low permeability soils contaminated with chlorophenols. They hold several advantages over alternative bioremediation and disposal schemes, including size, ease of operation and maintenance, and cost.

Fill and draw bioreactors of this type are an appropriate choice for biological treatment of soils when space is a primary concern. Reactors can be manufactured to a wide range of capacities from any non-permeable, non-reactive material, including inexpensive steel drums and poly-vinyl chloride tanks. Bioreactor size is limited only by the soil properties and the availability of low-flow, high pressure pumps capable of delivering and extracting feed solutions. Bioreactors can be stored in sheds away from plant operation and maintained at temperatures ideal for microbial performance, or they can be placed in yards, exposed to ambient environmental conditions. Reactors weighing up to several tons can easily by moved by materials handling equipment, such as forklifts, improving the versatility of the bioremediation scheme.

One of the main advantages of the fill and draw bioreactor scheme is its inherent simplicity and ease of operation. Fill and draw reactors can easily be constructed by a skilled welder. Operating the reactors requires no special knowledge or training and can easily be conducted by skilled or unskilled workers. Unlike landfarming, fill and draw reactors require no expensive equipment. Unlike similar types of similar bioreactors that employ a cross-flow technique to flush and treat contaminated soil, fill and draw reactors require no hard-piped equipment, making them easier to maintain and operate. For this study, the only equipment used in the treatment process was a small, submersible pump and a length of PVC hose. Using fill and draw reactors, soil treatment can be accomplished at almost any type of industrial facility, by personnel skilled in any one of a variety of disciplines.

The inexpensive construction, easy operation and low maintenance characteristics of fill and draw reactors make them a financially viable alternative to other bioremediation schemes. The reactor used in this analysis was constructed using materials costing less than two-thousand dollars. Operating costs for fill and draw treatment are defined by the type of feed mixture and the power and maintenance requirements of a pump. A total of

approximately twenty liters of imitation vanilla flavoring was used for this study. The IVF was formulated in-house, at a cost of under one dollar per liter. Purchasing a similar solution from a wholesale vendor would significantly increase treatment costs. Results of these analyses indicated that comparable treatment of chlorophenols can be achieved by treatment with a feed mixture augmented with anaerobic wastewater sludge and an extrinsic electron donor, and a feed solution containing only tap water. Using a simple feed mixture such as tap water could substantially reduce the cost of treatment.

In this baseline test of the pilot-scale fill and draw reactor, the total materials cost of treating 4 cubic yards of soil was approximately 2000 dollars. At a cost of 500 dollars per cubic yard, or approximately 100 dollars per 55 gallon drum, treatment via fill and draw bioreactors is comparable to transport and disposal of soil as hazardous waste. Transportation and disposal at a hazardous waste landfill or incineration can cost several hundred dollars per 55 gallon drum. But fill and draw reactors are re-usable and treatment costs on a per yard basis reduce significantly as the reactor is used, emptied and refilled. Fill and draw reactors are less expensive in the long-term.

Recommendations for Future Research

The success of this study suggests that even lower permeability soils and contaminated mixtures could be treated by the same process. Treatment of low permeability soils by fill and draw batch reactors was contingent on the dispersion of the fill mixtures. Distribution of electron donors and anaerobic bacteria, depended on the soil structure and supply pump characteristics. Soils treated in this analysis were of moderately-low permeability. With an augmented delivery and extraction system, or by using a pressurized vessel, treatment of contaminated mixtures such as wood debris from shop floors, sweepings from wood treatment vessels or extremely low permeability soils could be possible. Pre-treating soils could also improve the effectiveness of fill and draw bioreactors. In cases where soil permeability impairs the distribution of treatment mixtures, soil could be mixed with gravel. In other cases, soil could be augmented with

humic matter to increase organic content or a variety of chemicals to adjust soil pH, nitrate or sulfate content or a any number of parameters, as needed.

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Appendices

Appendix A. Pilot-Scale Bioreactor Soil Loading Protocol

Purpose:

To fill the bioreactor with a chlorophenol contaminated soil slurry in preparation for treatment.

Equipment:

- Approximately 3.7 cubic meters chlorophenol contaminated soil slurry obtained from MD McFarland Company, stored in 55 gallon drums
- 6 cubic yard metal container supplied by DeWald Northwest Company (Salem, OR); modified to conform to specifications listed in Appendix L
- Shovels, spades, scoops suitable for transferring soil slurries
- 1 Drum lifter (McMaster-Carr, Los Angeles, CA)
- Fork lift, cap. 4000 lb. or greater
- 2 wrenches, adjustable 3/4 - 1-1/4 inch, or similar
- Plastic sheets of sufficient length and width to cover 400 square feet
- 2 3/4 inch x 96 inch steel rod (or similar material) calibrated at 6 inch increments
- 3 sampling wells constructed to specifications listed in Appendix L.
- 12 foot measuring tape
- Paper towels
- 4 5 gallon plastic bucket
- 100 feet cotton duct tape
- Sampling/filling platform constructed from shipping pallets or plywood sheets
- Protective gear (Tyvek® coveralls, safety glasses, neoprene gloves, neoprene overboots)

Procedure:

1.   Place the bioreactor on a flat surface capable of withstanding a static load in excess of 5 tons per square foot. Level and support the bioreactor using shims where needed.
2.   Open the bioreactor clam-shell lids.
3.   Place filling /sampling platform so that it extends over bulkheads and provides a level working surface.
4.   Lay out plastic sheeting to cover any area which may be subjected to contamination by overspill. Tape plastic sheets to the sides of the bioreactor just below the bulkhead, draping the plastic down the sides of the reactor, and leaving enough slack to prevent puncture.
5.   Affix the drum lifter to the fork lift as needed.

6. Using the drum lifter, move one barrel onto the plastic sheeting.
7. Remove the drum cover using adjustable wrenches, and place the drum cover on the plastic sheet.
8. Raise the drum over the bulkhead and position it over the first treatment zone.
9. Carefully rotate the drum in the lifting cradle, and using a shovel empty the contents of the drum into the treatment zone. Continue filling the first zone to a uniform depth of 6 inches.
10. Tamp soil using steel bars to eliminate air pockets.
11. Fill soil zones B, C, and D in a similar manner, to a depth of six inches, then repeat the process starting with zone a, filling each zone successively in 6 inch increments until a depth of approximately 42 inches is reached.
12. Seal any drums which are not being unloaded.
13. Once filling is complete drive one sampling well into the middle of each soil treatment zone, locating the center of each zone using the 12 foot measuring tape. Wells can be driven by hand or by a small dead-blow mallet or wooden block.
14. Remove the filling/sampling platform and secure the clam shell lids.
15. Clean any tools by rinsing with tap water while holding the tools over an open drum to catch any run-off.
16. Disassemble the drum lifter and fork lift.
17. Remove and dispose of plastic sheeting and protective gear in an appropriate hazardous waste receptacle.

Appendix B. Pilot-Scale Bioreactor Fill and Drain Protocol

<u>Purpose</u>:

To demonstrate the nutrient and substrate delivery system for the pilot-scale bioreactor.

<u>Equipment</u>:

- 6 cubic yard metal container supplied by DeWald Northwest Company (Salem, OR); modified to conform to specifications listed in bioreactor construction guidelines (Appendix L) filled with approximately 3.7 cubic meters of chlorophenol-contaminated soil (Appendix A) obtained from MD McFarland Company
- 2 wrenches, adjustable 3/4 - 1-1/4 inch, or similar
- 1 20-50 gpm submersible pump supplied by McMaster-Carr (Los Angeles, CA)
- 2 55 gallon steel drums
- Plastic sheets of sufficient length and width to cover 20 square feet
- 12 foot measuring tape
- 50 feet 1/2 inch rubber or vinyl hose, and necessary fittings
- 4 1L Nalgene® bottles
- Feed mixture prepared with 0.424 L IVF, 83 mL S3, 284 mL S4, and 276 mL S7, per 100 L of liquid drained from treatment wells (IVF, S3, S4 and S7 are prepared in accordance with Appendix M
- 1 1 L graduated cylinder
- Sampling/filling platform constructed from shipping pallets or plywood sheets
- Protective gear (face shields, neoprene gloves, neoprene overboots)

<u>Procedure</u>:

Draining Procedure

1.   Open the bioreactor clam-shell lids.
2.   Place filling /sampling platform so that it extends over bulkheads and provides a level working surface.
3.   Lay out plastic sheeting to cover any area which may be subjected to contamination by overspill.
4.   Place two empty 55 gallon drums over the plastic sheeting near the bioreactor.
5.   Remove the drum covers using adjustable wrenches, and place the drum cover on the plastic sheet.
6.   Using the tape rule, measure and mark lines at 1/4, 1/2 and 3/4 the distance from the bottom of the drum.

7. Attach the 1/2 inch hose to the discharge end of the pump. Lower the pump to the bottom of mixing zone 1, holding it by the power supply cord.
8. Place the open end of the 1/2 inch hose into an open 55 gallon drum and engage the pump, emptying the contents of the mixing zone.
9. Repeat the process for mixing zones 2 through 4.
10. Allow the liquid level in the bioreactor to equilibrate for approximately one hour after the first draining is complete.
11. Repeat the draining procedure until no liquid remains in the mixing wells; use the second drum as a drain receptacle as necessary.

Filling the Bioreactor

1. Measure the height of the liquid in each storage drum and record the approximate volume.
2. Using the graduated cylinder measure a volume of treatment mixture such that the ratio of treatment mixture to the drained mixture volume is as listed in the material list.
3. Pour the measured contents of the cylinder into the appropriate drum.
4. Connect the hose to the pump and lower the submersible to the bottom of the drum, holding it by the power cord. Engage the pump and allow it to mix the contents of the drum for approximately five minutes. Repeat the procedure with the second drum.
5. Remove the pump from the second drum and disengage the power supply. Attach the 1/2 inch hose to the pump discharge and place the open end of the hose into mixing zone 1, ensuring that it reaches the bottom of the mixing zone.
6. Engage the pump and fill the mixing zone to the height of the soil in treatment zone A.
7. Repeat the procedure for mixing zones 2 through 4.
8. Allow the fill solution to penetrate the soil treatment zones for approximately one hour. Pump the remaining fill solution into zones 1 through 4 being careful not to cause any pooling on the mixing zones by over-filling.
9. All subsequent filling operations should be conducted with solution removed from the bioreactor, and stored temporarily and mixed in 50 gallon drums.
10. Remove the submersible pump from the mixing zone and flush the pump and hose with tap water to remove any solids. Retain the rinse water for disposal in an appropriate hazardous waste receptacle.
11. Cover the steel drums when not in use.
12. Remove and dispose of plastic sheeting and protective gear in an appropriate hazardous waste receptacle.
13. Remove the sampling/filling platform from the bioreactor and close and secure the clam-shell lids.

Appendix C. Pilot-Scale Bioeactor Sampling Protocol

<u>Purpose</u>:

To draw samples from the soil bioreactor to determine the concentrations of
chlorophenols in contaminated soil and soil pore water

<u>Equipment</u>:

- 3 foot section 1/4 inch O.D. glass tubing
- 6 inch section 1/4 inch O.D. glass tubing
- 3 feet 1/4 inch I.D. Tygon or vinyl tubing
- 1 pipette bulb
- 3 10 mL straight tubes with caps
- 1 100 mL Nalgene® bottle
- Analytical balance
- 3 100 mL beakers
- Soil sampling device built to specifications listed in Appendix L.
- 1 small stainless steel spatula
- 1 six foot x 3/4 in steel rod
- Safety equipment (neoprene or nitrile gloves and safety glasses)
- 100 mL tap water
- Parafilm™

<u>Procedure</u>:

Soil Sampling

1. Remove the clam-shell lid from the bioreactor and secure the sampling platform to the bulkheads over treatment zone A.
2. Using the soil sampling device with the cone end down, penetrate the soil to a depth of approximately two feet.
3. Raise the sampling device to a few inches above the soil. Remove any excess soil from the sampling cone by quickly spinning the sampler rod.
4. Remove the sampler from the bioreactor and empty its contents into one 100 mL beaker, using the spatula if necessary.
5. Using a steel rod, agitate the soil and fill the hole made by the sampling device.
6. Separate the sampling cone from the sampling rod and clean with tap water. Replace the cone when clean, and deposit the cleaning water/soil mixture in a hazardous waste receptacle for disposal.
7. Repeat the sampling process for soil treatment zones B and C, noting the sample names on each beaker.

8. Clean the sampling device and spatula with tap water when the procedure is complete.

9. Cover the soil samples with Parafilm™ and refrigerate until further analyses are conducted.

Pore Water Sampling

1. Assemble the pore water sampling device by placing one section of glass tubing in each end of the Tygon tubing. Insert the open end of the six-inch section of glass tubing into the pipette bulb.

2. Compress the pipette bulb and place the open end of the three-foot section of glass tubing into the sampling well.

3. Draw all water available from the treatment well A and deposit a 10 mL sample into a straight tube. Deposit the remaining water on top of the soil in the adjoining treatment zone. Cap and label the tube.

4. Clean the sampling device by placing the open end of the three-foot section of glass tubing into the 100 mL bottle filled with water. Flush the sampler by repeatedly drawing and expelling water from the glass tubing until no debris remains. Contain the cleaning water for disposal.

5. Repeat the sampling procedure for wells B and C.

6. When sampling is completed, clean the sampling device using the forementioned procedure. Remove the pipette bulb from the sampling device and flush the apparatus with RO water from a continuous pressurized supply for approximately one minute.

Appendix D. Bench-Scale Fill and Draw Batch Reactor Design and Construction Protocol

Purpose:

To construct upflow fill and draw bioreactors capable of treating low permeability soils contaminated with chlorophenols.

Materials:

- 25 ft 1-1/4 in ID CPVC pipe
- 45 1-1/4 in socket weld pipe end caps
- 45 1/4 in x 3/16 NPT PVC elbow barb fittings
- 45 1-1/2 in dia. 200x200 304 SS mesh discs
- 45 2 in id. 1000 μm polyester mesh discs
- CPVC adhesive and cleaner
- 1 L 1/8 in dia. glass beads
- 50 mL beaker
- Teflon pipe tape

Procedure:

1. Cut CPVC pipe into 45 six inch sections
2. Drill and tap 45 end caps to accept barbed fittings. Wrap barb fitting threads with Teflon tape and screw barbed fittings into end caps.
3. Clean the insides of the end caps using the CPVC cleaning solution.
4. Measure 20 mL of glass beads into each end cap. Wrap each stainless steel mesh disc in polyester filter paper. Place the disc and paper inside the end cap, covering the glass beads.
5. Prepare the pipe sections for assembly by cleaning a 1 inch section from one end of each pipe using the CPVC cleaning solution.
6. Swipe the pipe sections and end caps with CPVC adhesive. Assemble the reactors by inserting the pipe sections into the end caps as shown in Figure 1 below, being careful not to disturb the stainless steel mesh filter.
7. Allow the adhesive to dry for 30 minutes then rinse each reactor with a soap solution and rinse three times with tap water.

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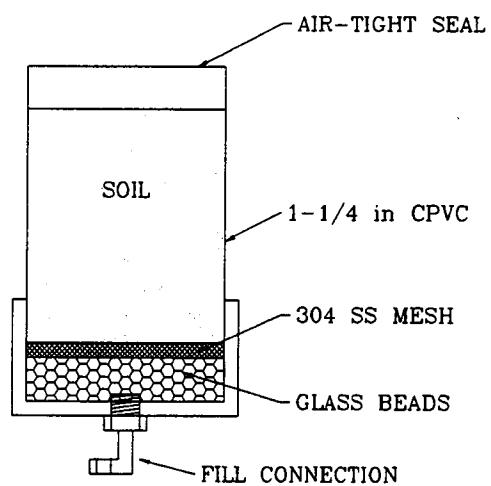
Figure 1. Bench-Scale Reactor Assembly

Appendix E. Bench-Scale Fill and Drain Batch Reactor Operation Protocol

Purpose:

To set up an upflow reactor study designed to demonstrate the anaerobic and aerobic transformation and removal of pentachlorophenol in soil using five series of batch reactors fed five different treatment solutions.

Materials:

- 45 Fully assembled upflow batch reactors, constructed to specifications listed in Appendix D
- 5 1000 mL Nalgene® bottles with screw tops
- 1 1000 mL Erlenmeyer flask
- 9 No. 7 butyl rubber stoppers
- Portable nitrogen cylinder with single-stage regulator
- 2 small bore hypodermic needles
- 8' 3/16" ID Tygon® tubing
- 15 L of PCP contaminated soil
- 25' 1/8" OD Teflon FEP tubing
- 3/16 in x 3/16 in nylon barb tube fitting
- Digital balance
- Weigh boat
- #4 ASTM sieve
- 25 L Polyethylene tub
- 1 gallon polyethylene bucket with lid
- 5 Yards 1000μm polyester filter paper
- 5 lb. sledge hammer or soil tamper
- 5 sq. ft. 1/8" perforated steel sheet
- 5 sq. yds. 2 mm plastic sheet
- 0.002 gpm, 20 psi peristaltic pump and necessary fittings (McMaster-Carr, Los Angeles, CA)
- 1/2 inch diameter diffusing stone
- 45 tube clamps
- 50 mL graduated cylinder

Procedure:

Soil Preparation

1. Air dry the soil and remove any large cobble and extraneous substances including leaves and twigs. Line the steel sheet with polyester filter paper. Set the steel sheet atop 25 liter polyethylene tub.
2. Distribute the saturated soil on top of the filter paper in a uniform one-half inch thick layer. Allow the soil to air dry for approximately three days, mechanically agitating and turning the soil each day.
3. Pick out and remove any small cobble from the soil. Using the filter paper, remove the dried soil from the perforated steel sheet. Remove the steel sheet from the top of the polyethylene tub and line the tub with a 3x3 ft plastic sheet.
4. Place the soil and filter paper inside the 25 liter polyethylene tub and fold the plastic sheet over the soil. Secure the plastic with tape. Using the sledge or soil tamper, break the soil into small-sized lumps suitable for sieving.
5. Sort the soil to a uniform size using a #4 sieve. Load the soil into the sieve and shake for 5 minutes. Soil that passes the sieve should be retained for analysis. Place this soil in 1 gallon polyethylene bucket.
6. Using the digital balance, measure and record the tare weight of the weigh paper.
7. Measure 50 grams of soil into each reactor being careful to agitate the fill bucket after each fill to ensure a uniform soil sample.

Fill Mixture Preparation

1. Label the 1 L Nalgene® bottles, 1 through 5.
2. Fill bottle 1 with distilled water and cap.
3. To bottles 2 through 5, add the following nutrient solutions: 17 μL S3, 84 μL S4, 55 μL S7, and 4.2 mL imitation vanilla flavoring (IVF). Vitamin solutions and IVF are prepared in accordance with Appendix M.
4. Fill bottles 2 and 3 with distilled water.
5. Cap and shake the bottles 2 and 3 vigorously.
6. Secure a diffuser stone to a three foot section of Tygon® tubing Secure the other end of the Tygon® tubing to the single stage nitrogen regulator.
7. Remove the cap on bottle 2 and purge the treatment mixture with nitrogen for approximately 3 minutes at 1-3 psi.
8. Fill the Erlenmeyer flask with distilled water. Remove the diffuser stone from bottle 2, screw the cap onto bottle 2 and place the diffuser stone into bottle 4. Purge the oxygen from bottle 4 with nitrogen for 3 minutes.
9. Measure 250 mL of anaerobic wastewater sludge into bottle 4, being careful not to introduce oxygen into the mixture.
10. Carefully fill the remaining space in bottle 4 with de-oxygenated distilled water. Slowly remove the diffuser stone from the mixture, purging the headspace in the bottle as the stone is extracted. Secure the screw cap to bottle 4 and gently shake the contents.

11. Siphon 1 L of anaerobic wastewater sludge into bottle 5. Purge the headspace in the bottle with nitrogen and secure the cap. Gently shake the bottle to mix its contents.

Reactor Fill Procedure

1. Measure 35 mL of the fill mixture from bottle 1 into the graduated cylinder.
2. Connect approximately 6 inches of Tygon® tubing to the discharge end of the peristaltic pump. Secure the remaining tubing to the suction side of the pump.
3. Connect the open end of the pump discharge tubing to an upflow reactor using the barbed fitting. Place the open end of suction tubing into the graduated cylinder. Open the tube clamp on the pig-tail tubing and engage the pump at the lowest flow rate, filling the reactor.
4. Remove the tubing from the reactor at the barb fitting and tighten the tubing clamp. Fill the remaining reactors in the series.
5. After the last reactor in the series is filled, remove the tubing from the reactor at the barbed fitting. Re-secure the tubing clamp on the reactor pigtail. Fill the 1000 mL Erlenmeyer flask with distilled water. Flush the pump tubing by inserting the suction tubing into the flask and engaging the pump for approximately 30 seconds, or until the tubing is free of debris. The flushing liquid can be drained into a sink.
6. Fill and rinse the graduated cylinder with distilled water 3 times, or until all debris is removed.
7. Fill the reactors in series 3 through 5 following this procedure using their corresponding feed solutions.
8. Remove the diffuser stone from the nitrogen feed line.
9. Fill the reactors in series 2 using the fill deoxygenated feed solution in bottle 2.
10. Carefully purge the headspace in each reactor with nitrogen and cap each reactor using a no. 7 butyl rubber stopper.
11. When the fill procedure is complete, retain a 10 mL of each fill solution for COD and chlorophenol analysis. Discard the remainder of the fill solutions and disassemble the peristaltic pump.

Appendix F. Bench-Scale Fill and Drain Batch Reactor Sampling and Draining Protocol

Purpose:

To sample a series of upflow reactors in preparation for soxhlet extraction and chlorophenol analysis.

Materials:

- 45 Fully assembled upflow batch reactors, constructed to specifications listed in Appendix D
- 1 1000 mL Erlenmeyer flask
- 2' 3/16" ID Tygon® tubing
- Digital balance
- Weigh paper
- 0.002 gpm, 20 psi peristaltic pump with necessary fittings and neoprene tubing (McMaster-Carr, Los Angeles, CA)
- 50 mL graduated cylinder
- 3/16 in x 3/16 in nylon male barb tubing fitting
- 45 2 mL microcentrifuge tubes

Procedure:

Liquid Sampling

1. Connect approximately 6 inches of Tygon® tubing to the discharge end of the peristaltic pump. Secure the remaining tubing to the suction side of the pump.
2. Connect the open end of the suction side tubing to an upflow reactor using the barbed fitting. Place the open end of discharge side tubing into the graduated cylinder. Open the tube clamp on the pig-tail tubing and engage the pump at the lowest flow rate, removing the solution from the reactor.
3. Continue pumping until no solution remains in the reactor. Measure and record the volume of feed solution removed from the reactor and carefully pour approximately 2 mL of the solution into a microcentrifuge tube. Label the centrifuge tube with the appropriate series designation.
4. Remove the tubing from the reactor at the barb fitting. Re-secure the tubing clamp on the reactor pigtail. Fill the 1000 mL Erlenmeyer flask with distilled water. Flush the pump tubing by inserting the suction tubing into the flask and engaging the pump for approximately 30 seconds, or until the tubing is free of debris. The flushing liquid can be drained into a sink.

5. Fill and rinse the graduated cylinder with distilled water 3 times, or until all debris is removed.

6. The reactor can now be filled with feedstock in accordance with the procedure described in Appendix E; or the contents can be removed and readied for soxhlet extraction, using the procedure outlined in steps 7 through 9.

Preparation For Soxhlet Extraction

1. Remove the tubing clamp from the pigtail and dislodge the reactor from its stand. Place the reactor upside-down on a weigh boat and place the entire assembly on a l ab bench. The contents of the reactor, soil and feed solution, should settle into the weigh boat.

2. Allow the contents of the weigh boat to stand for approximately 10 minutes, then decant the feed solution from the boat. The soil in the weigh boat can now be analyzed in accordance with the soxhlet procedure outlined in Appendix G.

3. Clean the reactor using a soap solution and rinse with distilled water in preparation for additional analyses.

Appendix G. Soxhlet Extraction Protocol

Purpose:

To determine the concentration of chlorophenols in soil by extraction with dichloromethane using a soxhlet device.

Materials:

- 1 500 mL round or flat bottom boiling flask
- 1 24" ring stand
- 2 4' 3/16" ID Tygon® tubing
- 2 right angle ring stand connectors
- 1 40/55 Soxhlet Extractor
- 1 45/55 Alihn Condenser
- 1 re-circulating constant temperature water bath
- 1 22 mm cellulose thimble
- 1 Heating coil
- 1 Shielded rheostat (0-240V)
- 1 inch Parafilm™
- 1 12 inch mercury or alcohol thermometer 0°-100°C range
- 150 mL HPLC grade Dichloromethane
- 1 250 mL separatory funnel
- 1 small plastic weigh boat
- 1 analytical balance
- 0.5 mL .72 g/mL K_2CO_3 buffer solution
- 0.5 mL pipette
- pipette bulb
- 50 mL graduated cylinder
- Deionized water
- 250 separatory funnel
- small glass funnel
- 1 10 mL glass culture tube with Teflon lined screw cap
- 15 g soil sample

Procedure:

1. Acid wash and thoroughly rinse and dry all glassware prior to use.
2. Fasten the Alihn condenser to the ring stand using the 2 right angle ring stand connectors.

3. Secure one end of the Tygon® tubing to the outlet port (top port) of the condenser. Using any necessary adapters, connect the open end of the tubing to the inlet port of the water bath re-circulating pump.

4. Connect the outlet port of the water bath re-circulating pump to the inlet port (bottom port) of the condenser.

5. Engage the pump, initiating flow through the condenser. Adjust the thermostat to ensure that the condenser water temperature is 5-10°C.

6. Measure 15 g of soil into a plastic weigh boat.

7. Deposit the soil into the cellulose thimble, making sure that the soil is distributed evenly on the bottom.

8. Insert the thimble in the bottom of the soxhlet apparatus.

9. Assemble the extraction device by inserting the end of the Alihn condenser into
the ` top of the soxhlet extractor. Seal the gap between fittings using Parafilm™.

10. Under a fume hood, measure approximately 150 mL of dichloromethane into the boiling flask. Connect the boiling flask to the bottom of the soxhlet extractor. Seal the gaps between the glassware with Parafilm™.

11. Place the soxhlet assembly including the boiling flask on the heating coil and secure the assembly to the ring stand.

12. Connect the heating coil to the rheostat and energize the rheostat. Adjust the rheostat such that the temperature of the heating coil is approximately 80°C.

13. Ensure that the heating coil temperature is sufficient to volatilize dichloromethane. Adjust the rheostat as needed.

14. Measure the cycling time of the soxhlet apparatus once it reaches steady state (after three cycles). Allow the soxhlet extractor to cycle 200 times.

15. After the cycles have been completed, turn off the heating coil and allow the assembly to cool for approximately ten minutes.

16. Gently shake the assembly to remove any residual solvent trapped in the condenser.

17. Disengage the cooling pump.

18. Remove the Parafilm™ and detach the boiling flask from the bottom of the extraction assembly. Carefully drain any dichloromethane remaining in the soxhlet device into the flask and cover the flask with Parafilm™.

19. Measure 14.5 mL distilled water into a graduated cylinder. Remove the Parafilm™ and deposit the water into the flask. Measure 0.5 mL of a 0.72 g/mL K_2CO_3 buffer solution into the flask. Re-attach the Parafilm™ to the boiling flask and gently shake the flask for two minutes. Allow the solution to settle and separate.

20. Transfer the contents of the boiling flask to the separatory funnel and allow the mixture to separate.

21. Drain the dichloromethane into an appropriate receptacle for recovery or disposal. Fill the 10 mL test tube with the water using a Pasteur pipette.

22. Disassemble the extraction device. Discard the Parafilm™ and place the cellulose thimble and soil sample in an appropriate hazardous waste container.

23. Rinse the glassware with tap water, then acid wash and dry the glassware in preparation for the next analysis.

Appendix H. Soxhlet Efficiency Protocol

Purpose:

To determine the removal efficiency of soxhlet extraction of chlorophenols using the procedure outlined in Appendix G.

Materials:

- 2 500 mL round or flat bottom boiling flask
- 1 24" ring stand
- 2 4' 3/16" ID vinyl tubing
- 2 right angle ring stand connectors
- 1 40/55 Soxhlet Extractor
- 1 45/55 Alihn Condenser
- 1 re-circulating constant temperature water bath
- 1 22 mm cellulose thimble
- 1 Heating coil
- 1 Shielded rheostat (0-240V)
- 12 ft 1 inch Parafilm™
- 1 12 inch mercury or alcohol thermometer 0°-100°C range
- 1 L HPLC grade Dichloromethane
- 1 small plastic weigh boat
- 1 analytical balance
- 0.5 mL .72 g/mL K_2CO_3 buffer solution
- 2 250 mL separatory funnels
- 0.5 mL pipette
- pipette bulb
- 50 mL graduated cylinder
- Deionized water
- 250 separatory funnel
- small glass funnel
- 1 10 mL glass culture tube with Teflon lined screw cap
- 15 g soil sample

Procedure:

1.	Following the procedure outlined in steps 1-22 of Appendix G, conduct a soxhlet extraction of one soil sample. After separating dichloromethane and water using the separatory funnel, drain the dichloromethane into the boiling flask and retain for analysis.

2.   Under a fume hood, measure approximately 200 mL of fresh dichloromethane into the second boiling flask. Connect the boiling flask to the bottom of the soxhlet extractor. Seal the gaps between the glassware with Parafilm™.

3.   Following steps 11 through 23 of Appendix G , conduct a second soxhlet extraction on the soil sample, retaining the dichloromethane for analysis in the second boiling flask.

4.   Conduct chlorophenol assays (Appendix J) on water samples from the first two extractions.

5.   Conduct a second extraction of chlorophenols into water from each dichloromethane sample.

6.   Perform chlorophenol assays on water samples derived from the second set of water extractions.

Appendix I. Imitation Vanilla Flavoring Surfactant Study Protocol

Purpose:

To quantify the potential of imitation vanilla flavoring (IVF) as a surfactant. This analysis
will test the ability of IVF to wash PCP and 2,3,5,6-TeCP from, low-permeability soil
removed from the McFarland Cascade wood treatment facility. This procedure is intended
to precede the Miniature Chlorophenol Assay Protocol described in Appendix J.

Equipment:

        5 125 mL serum bottles
        5 aluminum crimp caps with Teflon septa
        1 Liters deionized water
        Hand crimper
        Oscillatory shaker table
        5 Disposable weigh boats
        Stainless steel spatula
        Analytical balance
        2 50 mL beakers for cleaning glass syringes
        50 mL graduated cylinder
        10 µL glass syringe
        25 2 mL plastic centrifuge tubes
        Approximately 200 g McFarland soil prepared in accordance with Appendix E
        Approximately 10 mL IVF prepared in accordance with Appendix M
        wire test tube rack

Procedure:

1.   The 125 mL serum bottles should be prepared by soaking overnight in an
        ammonium persulfate and 50% v/v sulfuric acid bath. When removed, the bottles
        should be rinsed three times with tap water and three times with distilled water
        then placed in a 125°C drying oven for approximately 30 minutes, or until dry.
        Bottles should then be removed from the oven and allowed to cool to room
        temperature.
2.   The spatula and graduated cylinder should be rinsed with distilled water and
        allowed to completely dry.
3.   Measure 30 g of soil into each disposable weigh boats using the analytical balance.
        Carefully empty the contents of each boat into a serum bottle.

4. Measure 50 mL distilled/deionized water into a graduated cylinder. Carefully empty the contents of the cylinder into the first serum bottle. Repeat the procedure for the remaining bottles.

5. Using the 10 µL and 100 µL syringes measure the following quantities of IVF into the appropriately labelled serum bottles.

   Bottle 1 - 0 µL, Bottle 2 - 10 µL, Bottle 3 - 50 µL

   Bottle 4 - 100 µL, Bottle 5 - 500 µL, Bottle 6 - 1000 µL

6. Place one aluminum cap with Teflon septum on each bottle and crimp tightly.

7. Using a suitable felt tip pen, label each serum bottle for its respective contents.

8. Invert each bottle and shake for one minute.

9. Place bottles, septa side down, on the shaker table for approximately 30 minutes.

10. Place the 2 mL centrifuge tubes in a wire test tube rack.

11. Remove the serum bottles from the shaker table. Shake the bottle vigorously to remove any sediment which has settled on the Teflon cap.

12. Using the 100 µL syringe, draw 200 µL of liquid from serum bottle 1. Inject the contents of the syringe into a centrifuge tube. Rinse the syringe 3 times with DI water, emptying the contents into a 50 mL beaker for disposal. Repeat the sampling process for bottles 2 through 5.

13. Record the time when the sample was drawn, then place the serum bottle, septum side down, on the shaker table

14. Centrifuge the samples at 6000 rpm for 5 minutes to separate the solid contents. When centrifuging is complete remove the tubes and place them in the wire test tube rack.

15. Evaluate the PCP concentration in the liquid contents of the micro-centrifuge tubes using the miniature chlorophenol assay outlined in Appendix J.

16. Repeat the serum bottle sampling process, steps 10 through 16 at 72 hours, 216 hours and 336 hours from the time of the initial sampling.

Appendix J. Miniature Chlorophenols Assay

<u>Purpose</u>:

To analyze Pentachlorophenol (PCP) and its anaerobic metabolites on a gas
chromatograph (GC) equipped with an electron capture detector (ECD). This procedure
was developed by Voss et al., (1982) and has been adapted from Pete Kaslik (1996). The
method was later modified by Perkins (1992) and miniaturized by Dr. Mark Smith (Smith
1993).

<u>Equipment</u>:

- 2 100 mL metal syringes
- HP-6890 series II G-C equipped with a TCD and a J&W Scientific DB-5MS
  30m column
- Chemstation™ software: Rev A. 05.01 [273], Copyright© Hewlett Packard,
  1990-1997
- 1 x 1 mL fixed volume pipette
- 1 x 500 µL repeating pipette
- 1 x 100 µL repeating pipette
- 10 mL disposable culture tubes with Teflon™ lined caps
- 2 mL capacity Borosilicate amber glass crimp top vials
- 12 mm crimp caps with Teflon™ and silicone septa
- 2 x 500 mL beakers (DI rinse and waste water)
- 1 x 10 mL beaker (hexane)
- 1 x 50 mL beaker (methanol rinse)
- Disposable Pasteur pipettes and bulbs
- Hand crimper
- Wrist action shaker
- aqueous chlorophenol samples

<u>Chemicals</u>:

- Acetic anhydride, reagent grade
- hexane, HPLC grade
- Internal standard reagent (30.4 g/L K_2CO_3, 500 mg/L 2,4,6,Tribromophenol in
  DI water)

<u>Procedure</u>:

1. Label each test tube according to sample contents and date.
2. Add the following to each tube
    a) 500 μL of internal standard reagent
    b) 100 μL sample
    c) 100 μL acetic anhydride
3. Prepare a method blank for each sample set following a and c from step 2
4. Gently shake test tubes then place on wrist action shaker for twenty minutes
5. Remove test tubes from wrist shaker, place in test tube rack and remove cap. A small release of gas should occur when the cap is opened. This verifies the presence of acetic anhydride.
6. Add 1 mL of hexane to each test tube; cap the tubes and place on the wrist shaker for twenty minutes.
7. Remove test tubes from shaker, place in test tube rack and remove caps. Use disposable Pasteur pipettes to extract hexane sample from test tubes (upper layer of dual, non-aqueous phase liquid) and place hexane sample into crimp top amber vials. Cover with a crimp cap and secure the cap with a hand crimper.
8. Place the sample vials on the GC autosampler rack. Establish the sequence table and load the appropriate method (CP_321) and start sequence.
9. Loading Method CP_321 will establish the following parameters for the HP 6890 GC:
    a. Temperature Program
        i.   Initial Temp 40°C
        ii.   Initial Time 1.00 min
        iii. Rate 1 25°C/min
        iv.   Final Temp 140°C
        v.   Final Time 0.0 min
        vi.   Rate 2 10.00°C/min
        vii. Final Temp 2 250°C
        viii Final Time 2 5.00 min
    b. Injection Temperature 250°C
    c. Detector Temperature 350°C
    d. Helium Program
        i.   Initial Flow 2.00 mL/min
        ii.   Initial Time 14.00 min
        iii. Rate 1 4.00 mL/min/min
        iv.   Final Flow 4.00 mL/min
        v.   Final Time 7.00 min
    e. Argon Methane Program Continuous Flow 7.00 min

Appendix K. Soil Permeability Study

Purpose:

To determine the saturated permeability of chlorophenol contaminated soil used in bench-scale and pilot-scale bioreactors

Materials:

- 30 cm section 1-1/4 in ID CPVC SCH 40 piping
- 2 1-1/4 in CPVC socket weld end caps
- CPVC adhesive and cleaner
- 1.5 L of PCP contaminated soil
- 2 2 ft x 3/16 in ID Tygon® tubing
- 2 3/16 in x 3/16 in nylon barbed tube fitting
- 1000 μm polyester filter paper
- 2 3/16 in tube clamps
- 4 ft x 1 in ID glass cylinder
- 2 ring stands with 4 adjustable fork clamps
- 4 L plastic bucket
- no. 5 butyl rubber stopper with a 1/4 in hole bored on center
- 2 in x 1/4 in OD 304 SS tubing

Procedure:

1.   Assemble the permeameter in accordance with the figure shown below.
2.   Fill the permeameter with soil so the pipe section is completely full. Compact the soil by tapping the barbed tube fitting against a solid surface. When soil has completely settled, re-fill the soil column and tamp the soil again.
3.   Place the second end cap on the permeameter and secure it with pipe adhesive.
4.   Place the permeameter in 2 fork clamps and secure the clamps to a ring stand, orienting the permeameter vertically.
5.   Secure one section of Tygon® tubing to each barbed fitting. Insert the small section of stainless tubing into the open end of the Tygon® tubing fastened to the bottom barbed fitting. Insert the open end of the stainless tubing through a hole bored in the no. 5 butyl rubber stopper.
6.   Secure the glass column to the ring stand using the 2 remaining fork clamps. Orient the column vertically and place the butyl rubber stopper into the bottom end.
7.   Fasten the tube clamp to the section of tubing between the column and permeameter, and fill the column with water.

8. Place the open end of the section of tubing protruding from the top section of the permeameter into a bucket or another suitable drain receptacle.
9. Remove the tube clamp and allow the water contained in the soil column to fill the permeameter.
10. Secure the tube clamp approximately thirty minutes after water begins to drip from the permeameter. If the water column is emptied before the column is saturated, refill the column and repeat the process.
11. Once the column is saturated, refill the water column and record the height of the water.
12. Rotate the permeameter such that it is horizontal; check the orientation using a level.
13. Release the clamp and record the initial time. When approximately 10 cm of water has drained from the water column, re-secure the clamp and record the final time and the height of the water column.
14. Calculate the saturated conductivity of the soil column using the formula

$$K_{sat} = \frac{L}{t1 - to} \ln \frac{bo + L}{b1 + L}$$

where L is the length of the permeameter, bo is the initial height of the water column, b1 is the final height of the water column and t1 and to are the final and initial times, respectively.
15. Record K_{sat} as the saturated hydraulic conductivity.

Saturated Hydraulic Conductivity Data

An analysis of the saturated hydraulic conductivity of the chlorophenol-contaminated soil was conducted in accordance with Appendix K. Ksat refers to the saturated conductivity as calculated using the formula

$$K_{sat} = \frac{L}{t1 - to} \ln \frac{bo + L}{b1 + L},$$

where to refers to the initial time (0 minutes), t1 refers to the time in column 1, bo refers to initial water column height (29 inches), b1 refers to the height of the water column at any time, and L is the length of the permeameter (30 cm). Results of the study are shown in Table 1. The final K_{sat} was the average of the six K_{sat} calculations, based on measurements taken during the 180 minute duration of the experiment.

Table 1. Saturated hydraulic conductivity data

time (min)	water level (in)	K_{sat} (in/min)	K_{sat} (cm/day)
0	29		
30	27.1	0.00452	2.56
60	25.4	0.00442	2.50
90	23.8	0.00439	2.49
120	22.1	0.00453	2.57
150	20.5	0.00462	2.62
180	19.6	0.00435	2.47

Ksat (final) = 2.54 cm/day.

Appendix L. Construction Drawings

Pilot-Scale Reactor Liquid Mixing Wells

Notes - The four liquid mixing wells are constructed as modular units outside of the reactor. Each well is manufactured from perforated plain steel sheets, supported with structural angle iron and flat bar stock, all of which is black plain steel. Perforated sheets are secured to the structural member using spot-welds and mechanical fasteners where necessary. Once the wells are constructed, they can be placed into the reactor and secured in place by welding the angle iron to the reactor sides.
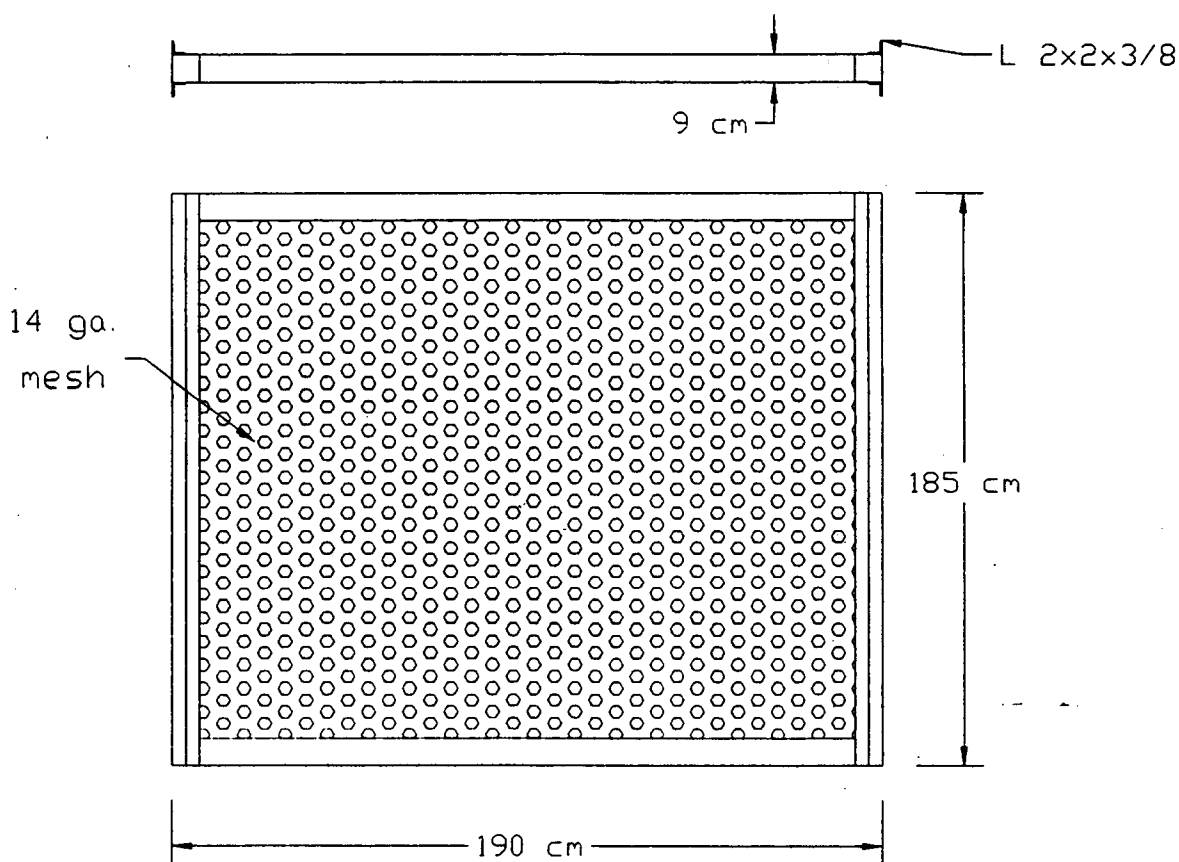
Pilot-Scale Reactor Sampling Well

Notes - The sampling well is constructed from a 52 inch long section of 1/2 inch black steel pipe. The pipe is closed off at one end using a cap and driven into the soil. Soil pore water penetrates the screened portion of the sampling well and is extracted using a siphoning device.

1/2in × 2in screen

52 in.

end cap
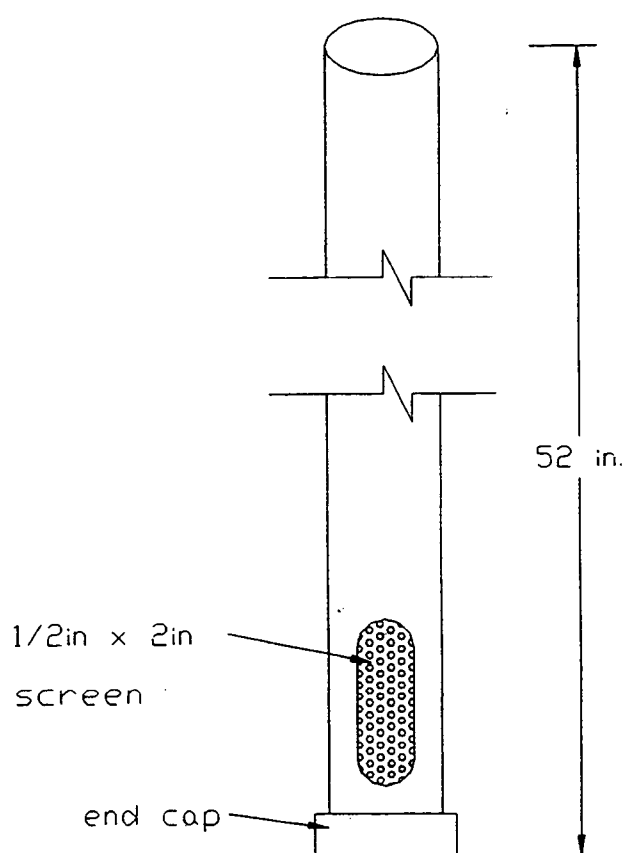
Pilot-Scale Reactor Sampling Apparatus

Notes - The sampling apparatus is a cone machined from a 5x5 cm aluminum bar. The cone is designed to penetrate a soil and cobble layer up to 5 feet in depth. The aluminum rod screwed into the cone serves a handle to force the coned through the soil reactor and retrieve soil samples.

1/2 in Aluminum rod

1.5 m

4.5cm dia.

5cm dia. x
8cm Aluminum
cone

drilled and tapped
for aluminum rod
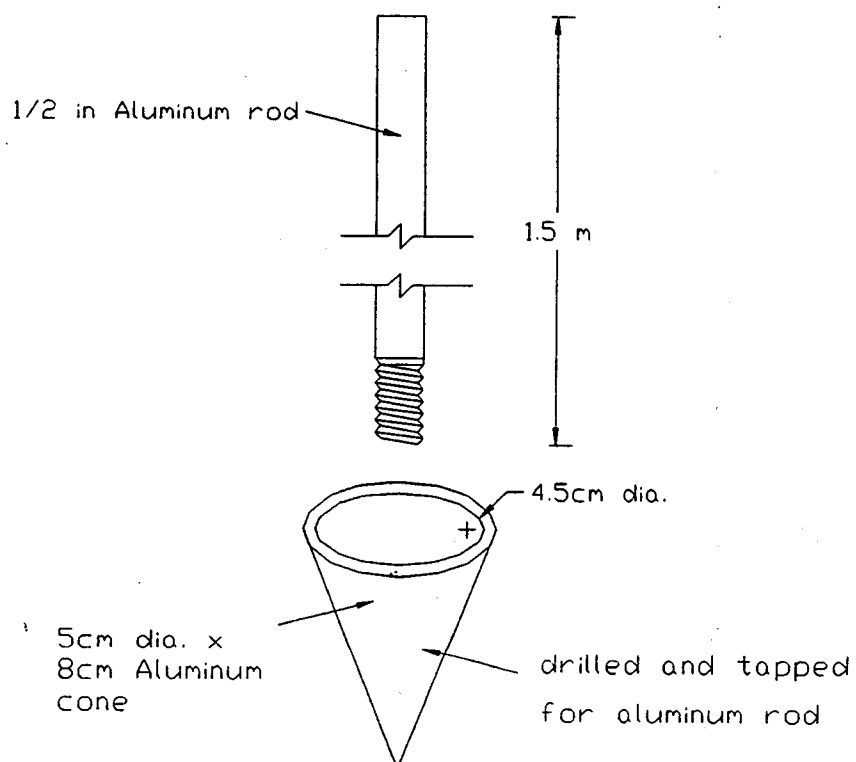
Appendix M. Composition of Treatment Mixture Components

Components of imitation vanilla flavoring

Compound	formula						
	conc. (g/L)	weight (g/L)	molarity (mol/L)	OD (mol/mol)	OD (g/mol)	COD (g/L)	TOC (g/L)
Guaiacol $C_7H_8O_2$	3.6	124	0.029	8	256	7.42	2.44
Ethyl Vanillan $C_9H_{10}O_3$	1.2	166	0.0072	10	320	2.31	0.78
Propylene Glycol $C_3H_8O_2$	7.8	76	0.1025	4	128	13.12	3.69
Benzoate $C_7H_5O_2$	0.8	144	0.0058	7.5	240	1.4	0.49
Total						24.25	7.40

Components of stock mixture S3

$(NH_4)_2HPO_4$ 26.7 g/L

Components of stock mineral mixture S4

Compound	Conc. (g/L)
$MgCl_26H_2O$	120.089
KCl	86.713
NH_4Cl	26.300
$CaCl_22H_2O$	16.707
$CoCl_26H_2O$	2.001
$MnCl_24H_2O$	1.338
$NiCl_26H_2O$	1.010
H_3BO_3	0.388
$CuCl_22H_2O$	0.181
$NaMoO_42H_2O$	0.173
$ZnCl_2$	0.141

Components of stock vitamin mixture S7

Compound	Conc. (mg/L)
Pyridoxine Hydrochloride	10.01
Riboflavin	5.08
p-Aminobenzoiuc acid	5.05
Thiamin	5.05
Thioctic acid	5.03
Nicotinic acid	5.01
Pantothenic acid	5.00
Folic acid	2.05
Biotin	2.01
B_{12}	0.12

Appendix N. Chlorophenol Extraction Efficiency and Statistical Considerations

The effectiveness of the fill and draw reactor treatment process was evaluated by the aerobic and anaerobic transformations of chlorophenols in soil occurring over each treatment cycle. The conclusions drawn from this study were dependent upon the accuracy and precision of the Soxhlet extraction process (Appendix G) employed to separate chlorophenols from contaminated soil. This extraction was evaluated using two procedures.

The first procedure determined the effectiveness of the Soxhlet soil washing procedure at removing chlorophenols from 15 g samples of low-permeability soil. Five soil samples were subjected to 240 consecutive washing cycles using HPLC grade dichloromethane as a solvent. The number of Soxhlet cycles used for these analyses was based on a similar procedure described by Woods et al., (1985). To determine whether this number of cycles could be effective for washing low permeability soils, dichloromethane was removed and analyzed after 240 cycles, and replaced with fresh dichloromethane. Soil was then washed for an additional 240 cycles. A comparison of dichloromethane from the first and second extraction procedures showed 100% removal of available chlorophenols from soil samples using a single stage Soxhlet extractions consisting of 240 cycles (Table 1).

Table 1. Removal Efficiency of Soil-Dichloromethane Extraction

Sample	2,3,5,6- TeCP		2,3,4,5- TeCP		PCP	
	1st/2nd	percent removed	1st/2nd	percent removed	1st/2nd	percent removed
A	0.95/0	100	0.08/0	100	2.04/0	100
B	0.81/0	100	0.07/0	100	1.92/0	100
C	0.11/0	100	0.05/0	100	2.07/0	100
D	0.11/0	100	0.07/0	100	2.15/0	100
E	0.26/0	100	0.08/0	100	2.05/0	100

A liquid-liquid extraction of chlorophenols from dichloromethane into water buffered with a potassium carbonate solution was the final stage of the Soxhlet analysis procedure. Consecutive dichloromethane/water extractions conducted on six contaminated soil samples indicated 100% transfer of 2,3,5,6-TeCP and 2,3,4,5-TeCP from dichloromethane to water, and 98% to 100% partitioning of PCP into water (Table 2).

Table 2. Dichloromethane to Water Extraction Efficiency

Sample	2,3,5,6- TeCP		2,3,4,5- TeCP		PCP	
	1st/2nd	percent removed	1st/2nd	percent removed	1st/2nd	percent removed
A	0.19/0	100	0.06/0	100	0.37/0	100
B	0.04/0	100	0.07/0	100	0.15/0	100
C	0.23/0	100	0.07/0	100	1.32/0.02	97.25
D	0.02/0	100	0.07/0	100	0.14/0	100
E	0.18/0	100	0.06/0	100	0.37/0	100
F	0.22/0	0	0.07/0	0	1.28/0.03	97.88

Appendix O. List of Filenames

Microsoft Word version 6.0 files

abstract.doc	Thesis abstract
title.doc	Title page
tablecon.doc	Table of contents and list of appendices
listfig.doc	List of figures
listab.doc	List of tables
litrev2.doc	Thesis literature review
intro2.doc	Introduction to Experimental Results Chapter 3
matmet2.doc	Materials and Methods Chapter 3
results2.doc	Experimental Results Chapter 3
discusn.doc	Discussion Chapter 3
summary.doc	Summary of Results Chapter 3
app_a.doc	Thesis Appendices
app_b.doc	
app_c.doc	
app_d.doc	
app_e.doc	
app_f.doc	
app_g.doc	
app_h.doc	
app_i.doc	
app_j.doc	
app_k.doc	
app_l.doc	
app_m.doc	
app_n.doc	
app_o.doc	
plots.doc	Appendix Figures
biblio.doc	Thesis Bibliography

Microsoft Excel version 5.0 documents

bench.xls	Data for bench-scale reactor experiment
pilot.xls	Data for pilot-scale reactor experiment
hycond.xls	Data for soil hydraulic conductivity analysis
soxeff.xls.	Data for Soxhlet extraction efficiency analysis
vansurf.xls	Data for imitation vanilla flavoring surfactant study

Appendix P.

Table 1. Mass of chlorophenols added to each reactor series with each treatment cycle

Week 1	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (µg)	area ratio	mass (µg)	area ratio	mass (µg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.009	0.699
Series E	0.000	0.000	0.000	0.000	0.048	3.714
Week 2	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (µg)	area ratio	mass (µg)	area ratio	mass (µg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.009	0.682
Series E	0.000	0.000	0.000	0.000	0.048	3.702
Week 3	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (µg)	area ratio	mass (µg)	area ratio	mass (µg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.010	0.809
Series E	0.011	0.889	0.000	0.000	0.053	4.157
Week 4	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (µg)	area ratio	mass (µg)	area ratio	mass (µg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.007	0.528
Series E	0.007	0.529	0.000	0.000	0.041	3.166
Week 5	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (µg)	area ratio	mass (µg)	area ratio	mass (µg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.000	0.000
Series E	0.000	0.000	0.012	1.086	0.025	1.983

Table 1 (continued). Mass of chlorophenols added to each reactor series with each treatment cycle

Week 6	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (μg)	area ratio	mass (μg)	area ratio	mass (μg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.000	0.000
Series E	0.013	1.040	0.023	2.142	0.029	2.264
Week 7	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (μg)	area ratio	mass (μg)	area ratio	mass (μg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.008	0.711	0.000	0.000
Series E	0.014	1.080	0.019	1.798	0.015	1.165
Week 8	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (μg)	area ratio	mass (μg)	area ratio	mass (μg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.061	4.821	0.000	0.000	0.000	0.000
Series E	0.000	0.000	0.010	0.966	0.000	0.000
Week 9	2,3,5,6-TeCP		2,3,4,5-TeCP		PCP	
	area ratio	mass (μg)	area ratio	mass (μg)	area ratio	mass (μg)
Series A	0.000	0.000	0.000	0.000	0.000	0.000
Series B	0.000	0.000	0.000	0.000	0.000	0.000
Series C	0.000	0.000	0.000	0.000	0.000	0.000
Series D	0.000	0.000	0.000	0.000	0.000	0.000
Series E	0.000	0.000	0.000	0.000	0.000	0.000

Table 2. Volume of treatment mixture drained from reactors in mL

series/ cycle	Week 1				
	A	B	C	D	E
1	9	8	9	8	4
2	7	8	9	16	12
3	9	9	7	10	9
4	9	11	7	11	10
5	7	7	11	10	13
6	7	6	7	10	9
7	7	5	7	10	6
8	10	7	7	7	7
9	7	4	7	8	8

series/ cycle	Week 2				
	A	B	C	D	E
2	30	20	35	26	15
3	27	11	12	15	15
4	15	13	30	24	13
5	16	13	13	20	15
6	12	12	15	25	16
7	28	34	13	22	25
8	11	30	14	26	27
9	27	22	14	16	24

series/ cycle	Week 3				
	A	B	C	D	E
3	18	45	33	20	34
4	30	40	31	22	28
5	29	50	28	10	20
6	33	47	39	23	23
7	21	32	30	20	13
8	43	30	22	15	25
9	24	25	28	32	22

series/ cycle	Week 4				
	A	B	C	D	E
4	33	17	20	23	19
5	32	14	31	36	21
6	33	31	23	29	25
7	24	34	28	23	21
8	28	26	27	32	25
9	19	15	25	25	22

Table 2 (continued). Volume of treatment mixture drained from reactors in mL

series/ cycle	Week 5				
	A	B	C	D	E
5	31	21	29	24	28
6	24	33	22	21	25
7	14	32	31	28	25
8	26	30	33	26	11
9	23	63	32	28	25
series/ cycle	Week 6				
	A	B	C	D	E
6	25	27	26	22	25
7	24	35	22	21	21
8	28	32	20	21	24
9	26	34	20	29	17
series/ cycle	Week 7				
	A	B	C	D	E
7	24	31	33	26	18
8	27	27	21	19	21
9	26	31	25	23	24
series/ cycle	Week 8				
	A	B	C	D	E
8	23	32	34	27	32
9	25	14	18	24	26
series/ cycle	Week 9				
	A	B	C	D	E
9	28	35	30	38	40

Table 3. Concentrations of Chlorophenols in reactor effluent (μg/L)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A1	0.000	0.000	0.000	0.000	0.000	0.035	0.000	0.026
A2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A3	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.015	0.000	0.015
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.018	0.000	0.017
A8	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.011
A9	0.000	0.000	0.000	0.000	0.000	0.021	0.000	0.017
B1	0.000	0.000	0.000	0.000	0.000	0.041	0.000	0.025
B2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.064	0.000	0.051
B5	0.000	0.000	0.000	0.000	0.000	0.029	0.000	0.029
B6	0.000	0.000	0.000	0.000	0.000	0.040	0.000	0.033
B7	0.000	0.000	0.000	0.000	0.000	0.067	0.000	0.046
B8	0.000	0.000	0.000	0.000	0.000	0.032	0.000	0.030
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C1	0.000	0.000	0.000	0.000	0.000	0.040	0.000	0.036
C2	0.000	0.000	0.000	0.000	0.000	0.011	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.014	0.000	0.012
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D1	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.013
D2	0.000	0.000	0.000	0.000	0.000	0.048	0.000	0.040
D3	0.000	0.000	0.000	0.000	0.000	0.019	0.000	0.018
D4	0.000	0.000	0.000	0.000	0.000	0.027	0.000	0.023
D5	0.000	0.000	0.000	0.000	0.000	0.034	0.000	0.026
D6	0.000	0.000	0.000	0.000	0.000	0.021	0.000	0.016
D7	0.000	0.000	0.000	0.000	0.000	0.014	0.000	0.013
D8	0.000	0.000	0.000	0.000	0.000	0.021	0.000	0.019
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E1	0.000	0.000	0.000	0.000	0.000	0.012	0.000	0.014

Table 3 (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

				2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	
E2	0.000	0.000	0.000	0.000	0.000	0.042	0.000	0.036
E3	0.000	0.000	0.000	0.000	0.000	0.026	0.000	0.021
E4	0.000	0.000	0.000	0.000	0.000	0.017	0.000	0.015
E5	0.000	0.000	0.000	0.000	0.000	0.023	0.000	0.022
E6	0.000	0.000	0.000	0.000	0.000	0.018	0.000	0.017
E7	0.000	0.000	0.000	0.000	0.000	0.016	0.000	0.015
E8	0.000	0.000	0.000	0.000	0.000	0.011	0.000	0.011
E9	0.000	0.000	0.000	0.000	0.000	0.028	0.000	0.024

Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A2	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.009
A3	0.000	0.000	0.000	0.000	0.000	0.019	0.000	0.031
A4	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.013
A5	0.000	0.000	0.000	0.000	0.000	0.017	0.000	0.016
A6	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.010
A7	0.000	0.000	0.000	0.000	0.000	0.017	0.000	0.023
A8	0.000	0.000	0.000	0.000	0.000	0.005	0.000	0.006
A9	0.000	0.000	0.000	0.000	0.000	0.025	0.000	0.036
B2	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.005
B3	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.008
B4	0.000	0.000	0.000	0.000	0.000	0.010	0.000	0.011
B5	0.000	0.000	0.000	0.000	0.000	0.009	0.000	0.009
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.006
B7	0.000	0.000	0.000	0.000	0.000	0.011	0.000	0.011
B8	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.007
B9	0.000	0.000	0.000	0.000	0.000	0.024	0.000	0.027
C2	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.006
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.007	0.000	0.007
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.006
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.005
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.006
C8	0.000	0.000	0.000	0.000	0.000	0.012	0.000	0.013
C9	0.000	0.000	0.000	0.000	0.000	0.010	0.000	0.009
D2	0.000	0.000	0.000	0.000	0.000	0.016	0.000	0.027
D3	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.008
D4	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.008
D5	0.000	0.000	0.000	0.000	0.000	0.011	0.000	0.014

Table 3 (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
D6	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.012
D7	0.000	0.000	0.000	0.000	0.000	0.018	0.000	0.021
D8	0.000	0.000	0.000	0.000	0.000	0.010	0.000	0.009
D9	0.000	0.000	0.000	0.000	0.000	0.006	0.000	0.007
E2	0.000	0.000	0.000	0.000	0.000	0.012	0.000	0.015
E3	0.000	0.000	0.000	0.000	0.000	0.007	0.000	0.009
E4	0.000	0.000	0.000	0.000	0.000	0.007	0.000	0.009
E5	0.000	0.000	0.000	0.000	0.000	0.024	0.063	0.036
E6	0.000	0.000	0.000	0.000	0.000	0.009	0.000	0.011
E7	0.000	0.000	0.000	0.000	0.000	0.018	0.000	0.029
E8	0.000	0.000	0.000	0.000	0.000	0.017	0.000	0.021
E9	0.000	0.000	0.000	0.000	0.000	0.033	0.000	0.051

Week 3	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.011
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.012	0.000	0.020
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.028	0.000	0.045
B6	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.026
B7	0.000	0.000	0.000	0.000	0.000	0.037	0.000	0.052
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.011
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.011
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.008
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 3 (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

				2,3,5-	3,4,5-	2,3,5,6-	2,3,4,5-	
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.009
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.008
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E3	0.000	0.000	0.000	0.000	0.000	0.033	0.021	0.035
E4	0.000	0.000	0.000	0.024	0.000	0.009	0.000	0.000
E5	0.000	0.000	0.000	0.008	0.000	0.036	0.028	0.052
E6	0.000	0.000	0.000	0.024	0.014	0.010	0.083	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.015	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.023	0.000	0.037
E9	0.000	0.000	0.000	0.000	0.000	0.030	0.009	0.053
Week 4	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.015
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.008
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.007
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E4	0.000	0.000	0.000	0.016	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.014	0.000	0.012	0.021	0.005
E6	0.000	0.000	0.000	0.012	0.006	0.000	0.000	0.000

Table (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

E7	0.000	0.000	0.000	0.000	0.000	0.016	0.000	0.025
E8	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.034
E9	0.000	0.000	0.000	0.000	0.000	0.015	0.007	0.034

Week 5	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.018	0.000	0.000	0.007	0.006
E6	0.000	0.000	0.000	0.000	0.000	0.013	0.000	0.014
E7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.006
E8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.006
E9	0.000	0.000	0.000	0.000	0.000	0.009	0.011	0.021

Week 6	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.016	0.000	0.000

Table 3 (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
B8	0.000	0.000	0.000	0.000	0.000	0.024	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.023	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.020	0.000	0.000
E6	0.000	0.000	0.000	0.019	0.010	0.015	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.017	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.012	0.012	0.000
E9	0.000	0.000	0.000	0.000	0.000	0.011	0.021	0.000
Week 7	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.019	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.012
C8	0.000	0.000	0.000	0.000	0.000	0.030	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.010	0.013	0.023	0.028
E8	0.000	0.000	0.000	0.000	0.000	0.042	0.017	0.026
E9	0.000	0.000	0.000	0.000	0.000	0.015	0.000	0.027
Week 8	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A8	0.000	0.000	0.000	0.000	0.000	0.072	0.000	0.017
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.129	0.000	0.019
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.058	0.000	0.000

Table 3 (continued). Concentrations of Chlorophenols in reactor effluent (µg/L)

C9	0.000	0.000	0.000	0.000	0.000	0.035	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.014	0.000	0.021
E9	0.000	0.000	0.000	0.000	0.000	0.026	0.000	0.029

Week 9	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A9	0.000	0.000	0.000	0.000	0.000	0.014	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.042	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.052	0.000	0.000
E9	0.000	0.000	0.000	0.000	0.014	0.073	0.035	0.029

Table 4. Mass of Chlorophenols Removed from reactors (µg)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A1	0.000	0.000	0.000	0.000	0.000	0.312	0.000	0.234
A2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A3	0.000	0.000	0.000	0.000	0.000	0.117	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.105	0.000	0.103
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.127	0.000	0.120
A8	0.000	0.000	0.000	0.000	0.000	0.127	0.000	0.109
A9	0.000	0.000	0.000	0.000	0.000	0.147	0.000	0.117
B1	0.000	0.000	0.000	0.000	0.000	0.325	0.000	0.200
B2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.700	0.000	0.561
B5	0.000	0.000	0.000	0.000	0.000	0.200	0.000	0.201
B6	0.000	0.000	0.000	0.000	0.000	0.241	0.000	0.195
B7	0.000	0.000	0.000	0.000	0.000	0.333	0.000	0.232
B8	0.000	0.000	0.000	0.000	0.000	0.224	0.000	0.211
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C1	0.000	0.000	0.000	0.000	0.000	0.321	0.000	0.287
C2	0.000	0.000	0.000	0.000	0.000	0.102	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.101	0.000	0.087
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D1	0.000	0.000	0.000	0.000	0.000	0.105	0.000	0.101
D2	0.000	0.000	0.000	0.000	0.000	0.760	0.000	0.647
D3	0.000	0.000	0.000	0.000	0.000	0.188	0.000	0.185
D4	0.000	0.000	0.000	0.000	0.000	0.293	0.000	0.254
D5	0.000	0.000	0.000	0.000	0.000	0.345	0.000	0.261
D6	0.000	0.000	0.000	0.000	0.000	0.211	0.000	0.160
D7	0.000	0.000	0.000	0.000	0.000	0.139	0.000	0.129
D8	0.000	0.000	0.000	0.000	0.000	0.150	0.000	0.132
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 4 (continued). Mass of Chlorophenols Removed from reactors (µg)

				2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	
E1	0.000	0.000	0.000	0.000	0.000	0.050	0.000	0.057
E2	0.000	0.000	0.000	0.000	0.000	0.510	0.000	0.434
E3	0.000	0.000	0.000	0.000	0.000	0.235	0.000	0.186
E4	0.000	0.000	0.000	0.000	0.000	0.174	0.000	0.151
E5	0.000	0.000	0.000	0.000	0.000	0.303	0.000	0.286
E6	0.000	0.000	0.000	0.000	0.000	0.161	0.000	0.152
E7	0.000	0.000	0.000	0.000	0.000	0.098	0.000	0.091
E8	0.000	0.000	0.000	0.000	0.000	0.080	0.000	0.078
E9	0.000	0.000	0.000	0.000	0.000	0.225	0.000	0.192

Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A2	0.000	0.000	0.000	0.000	0.000	0.172	0.000	0.267
A3	0.000	0.000	0.000	0.000	0.000	0.522	0.000	0.829
A4	0.000	0.000	0.000	0.000	0.000	0.195	0.000	0.189
A5	0.000	0.000	0.000	0.000	0.000	0.271	0.000	0.259
A6	0.000	0.000	0.000	0.000	0.000	0.101	0.000	0.121
A7	0.000	0.000	0.000	0.000	0.000	0.471	0.000	0.653
A8	0.000	0.000	0.000	0.000	0.000	0.054	0.000	0.061
A9	0.000	0.000	0.000	0.000	0.000	0.671	0.000	0.961
B2	0.000	0.000	0.000	0.000	0.000	0.153	0.000	0.107
B3	0.000	0.000	0.000	0.000	0.000	0.069	0.000	0.084
B4	0.000	0.000	0.000	0.000	0.000	0.136	0.000	0.138
B5	0.000	0.000	0.000	0.000	0.000	0.113	0.000	0.111
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.073
B7	0.000	0.000	0.000	0.000	0.000	0.381	0.000	0.376
B8	0.000	0.000	0.000	0.000	0.000	0.231	0.000	0.205
B9	0.000	0.000	0.000	0.000	0.000	0.518	0.000	0.603
C2	0.000	0.000	0.000	0.000	0.000	0.198	0.000	0.194
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.217	0.000	0.199
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.080
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.073
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.072
C8	0.000	0.000	0.000	0.000	0.000	0.165	0.000	0.183
C9	0.000	0.000	0.000	0.000	0.000	0.139	0.000	0.124
D2	0.000	0.000	0.000	0.000	0.000	0.411	0.000	0.690
D3	0.000	0.000	0.000	0.000	0.000	0.097	0.000	0.122

Table 4 (continued). Mass of Chlorophenols Removed from reactors (μg)

				2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	
D4	0.000	0.000	0.000	0.000	0.000	0.141	0.000	0.195
D5	0.000	0.000	0.000	0.000	0.000	0.219	0.000	0.281
D6	0.000	0.000	0.000	0.000	0.000	0.199	0.000	0.308
D7	0.000	0.000	0.000	0.000	0.000	0.404	0.000	0.468
D8	0.000	0.000	0.000	0.000	0.000	0.257	0.000	0.222
D9	0.000	0.000	0.000	0.000	0.000	0.096	0.000	0.108
E2	0.000	0.000	0.000	0.000	0.000	0.177	0.000	0.230
E3	0.000	0.000	0.000	0.000	0.000	0.109	0.000	0.142
E4	0.000	0.000	0.000	0.000	0.000	0.096	0.000	0.121
E5	0.000	0.000	0.000	0.000	0.000	0.358	0.945	0.534
E6	0.000	0.000	0.000	0.000	0.000	0.142	0.000	0.168
E7	0.000	0.000	0.000	0.000	0.000	0.447	0.000	0.725
E8	0.000	0.000	0.000	0.000	0.000	0.446	0.000	0.558
E9	0.000	0.000	0.000	0.000	0.000	0.785	0.000	1.229

Week 3	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.194
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.557	0.000	0.916
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	1.391	0.000	2.255
B6	0.000	0.000	0.000	0.000	0.000	0.608	0.000	1.232
B7	0.000	0.000	0.000	0.000	0.000	1.189	0.000	1.657
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.265
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.317
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 4 (continued). Mass of Chlorophenols Removed from reactors (μg)

				2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.173
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.175
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.121
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E3	0.000	0.000	0.000	0.000	0.000	1.114	0.731	1.195
E4	0.000	0.000	0.000	0.685	0.000	0.247	0.000	0.000
E5	0.000	0.000	0.000	0.166	0.000	0.713	0.553	1.043
E6	0.000	0.000	0.000	0.545	0.312	0.236	1.909	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.191	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.563	0.000	0.933
E9	0.000	0.000	0.000	0.000	0.000	0.667	0.207	1.169
Week 4	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.261	0.000	0.499
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.197
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.107
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 4 (continued). Mass of Chlorophenols Removed from reactors (μg)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E4	0.000	0.000	0.000	0.302	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.302	0.000	0.248	0.439	0.109
E6	0.000	0.000	0.000	0.308	0.152	0.000	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.341	0.000	0.527
E8	0.000	0.000	0.000	0.000	0.000	0.328	0.000	0.855
E9	0.000	0.000	0.000	0.000	0.000	0.319	0.158	0.755

Week 5	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.508	0.000	0.000	0.203	0.176
E6	0.000	0.000	0.000	0.001	0.000	0.323	0.000	0.358
E7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.153
E8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.067
E9	0.000	0.000	0.000	0.000	0.000	0.233	0.279	0.518

Table 4 (continued). Mass of Chlorophenols Removed from reactors (μg)

Week 6	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.566	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.753	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.481	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.572	0.000	0.000
E6	0.000	0.000	0.000	0.472	0.252	0.371	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.350	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.280	0.286	0.000
E9	0.000	0.000	0.000	0.000	0.000	0.183	0.349	0.000

Week 7	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.482	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.390
C8	0.000	0.000	0.000	0.000	0.000	0.638	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.187	0.235	0.416	0.511

Table 4 (continued). Mass of Chlorophenols Removed from reactors (µg)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
E8	0.000	0.000	0.000	0.000	0.000	0.874	0.365	0.537
E9	0.000	0.000	0.000	0.000	0.000	0.354	0.000	0.654
Week 8	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A8	0.000	0.000	0.000	0.000	0.000	1.656	0.000	0.386
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	4.140	0.000	0.600
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	1.963	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.634	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.457	0.000	0.681
E9	0.000	0.000	0.000	0.000	0.000	0.672	0.000	0.746
Week 9	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A9	0.000	0.000	0.000	0.000	0.000	0.128	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.382	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.464	0.000	0.000
E9	0.000	0.000	0.000	0.000	0.130	0.661	0.316	0.257

Table 5. Mass of Chlorophenols Added to Reactors as Treatment Mixtures (µg)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A1	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B1	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C1	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D1	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment
Mixtures (µg)

D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
E1	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.714
E2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment
Mixtures (µg)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.682
E2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
E9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	3.702
Week 3	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment Mixtures (µg)

C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
E3	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E4	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E5	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E6	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E7	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E8	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157
E9	0.000	0.000	0.000	0.000	0.000	0.889	0.000	4.157

Week 4	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment Mixtures (µg)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
E4	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
E5	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
E6	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
E7	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
E8	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
E9	0.000	0.000	0.000	0.000	0.000	0.529	0.000	3.166
Week 5	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.983
E6	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.983
E7	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.983
E8	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.983
E9	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.983

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment Mixtures (µg)

Week 6	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E6	0.000	0.000	0.000	0.000	0.000	1.040	2.142	2.264
E7	0.000	0.000	0.000	0.000	0.000	1.040	2.142	2.264
E8	0.000	0.000	0.000	0.000	0.000	1.040	2.142	2.264
E9	0.000	0.000	0.000	0.000	0.000	1.040	2.142	2.264

Week 7	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
E7	0.000	0.000	0.000	0.000	0.000	1.080	1.798	1.165

Table 5 (continued). Mass of Chlorophenols Added to Reactors as Treatment Mixtures (µg)

				2,3,5-	3,4,5-	2,3,5,6-	2,3,4,5-	
E8	0.000	0.000	0.000	0.000	0.000	1.080	1.798	1.165
E9	0.000	0.000	0.000	0.000	0.000	1.080	1.798	1.165
Week 8	3-CP	3,5-DCP	3,4-DCP	TCP	TCP	TeCP	TeCP	PCP
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	4.821	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	4.821	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	0.000	0.966	0.000
E9	0.000	0.000	0.000	0.000	0.000	0.000	0.966	0.000
				2,3,5-	3,4,5-	2,3,5,6-	2,3,4,5-	
Week 9	3-CP	3,5-DCP	3,4-DCP	TCP	TCP	TeCP	TeCP	PCP
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 6. Mass of Chlorophenols Remaining in Treatment Mixture after Draining, μg (negative numbers indicate a net loss in chlorophenol mass)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A1	0.000	0.000	0.000	0.000	0.000	-0.312	0.000	-0.234
A2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A3	0.000	0.000	0.000	0.000	0.000	-0.117	0.000	0.000
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	-0.105	0.000	-0.103
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	-0.127	0.000	-0.120
A8	0.000	0.000	0.000	0.000	0.000	-0.127	0.000	-0.109
A9	0.000	0.000	0.000	0.000	0.000	-0.147	0.000	-0.117
B1	0.000	0.000	0.000	0.000	0.000	-0.325	0.000	-0.200
B2	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	-0.700	0.000	-0.561
B5	0.000	0.000	0.000	0.000	0.000	-0.200	0.000	-0.201
B6	0.000	0.000	0.000	0.000	0.000	-0.241	0.000	-0.195
B7	0.000	0.000	0.000	0.000	0.000	-0.333	0.000	-0.232
B8	0.000	0.000	0.000	0.000	0.000	-0.224	0.000	-0.211
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C1	0.000	0.000	0.000	0.000	0.000	-0.321	0.000	-0.287
C2	0.000	0.000	0.000	0.000	0.000	-0.102	0.000	0.000
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	-0.101	0.000	-0.087
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D1	0.000	0.000	0.000	0.000	0.000	-0.105	0.000	0.598
D2	0.000	0.000	0.000	0.000	0.000	-0.760	0.000	0.052
D3	0.000	0.000	0.000	0.000	0.000	-0.188	0.000	0.515
D4	0.000	0.000	0.000	0.000	0.000	-0.293	0.000	0.446
D5	0.000	0.000	0.000	0.000	0.000	-0.345	0.000	0.438
D6	0.000	0.000	0.000	0.000	0.000	-0.211	0.000	0.539
D7	0.000	0.000	0.000	0.000	0.000	-0.139	0.000	0.571
D8	0.000	0.000	0.000	0.000	0.000	-0.150	0.000	0.568

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture after Draining (μg)

D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.699
E1	0.000	0.000	0.000	0.000	0.000	-0.050	0.000	3.657
E2	0.000	0.000	0.000	0.000	0.000	-0.510	0.000	-0.434
E3	0.000	0.000	0.000	0.000	0.000	-0.235	0.000	-0.186
E4	0.000	0.000	0.000	0.000	0.000	-0.174	0.000	-0.151
E5	0.000	0.000	0.000	0.000	0.000	-0.303	0.000	-0.286
E6	0.000	0.000	0.000	0.000	0.000	-0.161	0.000	-0.152
E7	0.000	0.000	0.000	0.000	0.000	-0.098	0.000	-0.091
E8	0.000	0.000	0.000	0.000	0.000	-0.080	0.000	-0.078
E9	0.000	0.000	0.000	0.000	0.000	-0.225	0.000	-0.192
Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A2	0.000	0.000	0.000	0.000	0.000	-0.172	0.000	-0.267
A3	0.000	0.000	0.000	0.000	0.000	-0.522	0.000	-0.829
A4	0.000	0.000	0.000	0.000	0.000	-0.195	0.000	-0.189
A5	0.000	0.000	0.000	0.000	0.000	-0.271	0.000	-0.259
A6	0.000	0.000	0.000	0.000	0.000	-0.101	0.000	-0.121
A7	0.000	0.000	0.000	0.000	0.000	-0.471	0.000	-0.653
A8	0.000	0.000	0.000	0.000	0.000	-0.054	0.000	-0.061
A9	0.000	0.000	0.000	0.000	0.000	-0.671	0.000	-0.961
B2	0.000	0.000	0.000	0.000	0.000	-0.153	0.000	-0.107
B3	0.000	0.000	0.000	0.000	0.000	-0.069	0.000	-0.084
B4	0.000	0.000	0.000	0.000	0.000	-0.136	0.000	-0.138
B5	0.000	0.000	0.000	0.000	0.000	-0.113	0.000	-0.111
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.073
B7	0.000	0.000	0.000	0.000	0.000	-0.381	0.000	-0.376
B8	0.000	0.000	0.000	0.000	0.000	-0.231	0.000	-0.205
B9	0.000	0.000	0.000	0.000	0.000	-0.518	0.000	-0.603
C2	0.000	0.000	0.000	0.000	0.000	-0.198	0.000	-0.194
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	-0.217	0.000	-0.199
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.080
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.073
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.072
C8	0.000	0.000	0.000	0.000	0.000	-0.165	0.000	-0.183
C9	0.000	0.000	0.000	0.000	0.000	-0.139	0.000	-0.124
D2	0.000	0.000	0.000	0.000	0.000	-0.411	0.000	-0.008

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture after Draining (µg)

D3	0.000	0.000	0.000	0.000	0.000	-0.097	0.000	0.560
D4	0.000	0.000	0.000	0.000	0.000	-0.141	0.000	0.487
D5	0.000	0.000	0.000	0.000	0.000	-0.219	0.000	0.401
D6	0.000	0.000	0.000	0.000	0.000	-0.199	0.000	0.374
D7	0.000	0.000	0.000	0.000	0.000	-0.404	0.000	0.214
D8	0.000	0.000	0.000	0.000	0.000	-0.257	0.000	0.460
D9	0.000	0.000	0.000	0.000	0.000	-0.096	0.000	0.574
E2	0.000	0.000	0.000	0.000	0.000	-0.177	0.000	3.472
E3	0.000	0.000	0.000	0.000	0.000	-0.109	0.000	3.559
E4	0.000	0.000	0.000	0.000	0.000	-0.096	0.000	3.581
E5	0.000	0.000	0.000	0.000	0.000	-0.358	-0.945	3.168
E6	0.000	0.000	0.000	0.000	0.000	-0.142	0.000	3.533
E7	0.000	0.000	0.000	0.000	0.000	-0.447	0.000	2.976
E8	0.000	0.000	0.000	0.000	0.000	-0.446	0.000	3.144
E9	0.000	0.000	0.000	0.000	0.000	-0.785	0.000	2.472

Week 3	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.194
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B3	0.000	0.000	0.000	0.000	0.000	-0.557	0.000	-0.916
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	-1.391	0.000	-2.255
B6	0.000	0.000	0.000	0.000	0.000	-0.608	0.000	-1.232
B7	0.000	0.000	0.000	0.000	0.000	-1.189	0.000	-1.657
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.265
C3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.317
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture after Draining (μg)

				2,3,5-	3,4,5-	2,3,5,6-	2,3,4,5-	
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.635
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.634
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.688
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.809
E3	0.000	0.000	0.000	0.000	0.000	-0.225	0.158	2.962
E4	0.000	0.000	0.000	-0.685	0.000	0.642	0.889	4.157
E5	0.000	0.000	0.000	-0.166	0.000	0.177	0.336	3.115
E6	0.000	0.000	0.000	-0.545	-0.312	0.653	-1.020	4.157
E7	0.000	0.000	0.000	0.000	0.000	0.698	0.889	4.157
E8	0.000	0.000	0.000	0.000	0.000	0.327	0.889	3.224
E9	0.000	0.000	0.000	0.000	0.000	0.223	0.682	2.989
Week 4	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	-0.261	0.000	-0.499
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.197
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.107
C4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D4	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture after Draining (µg)

				2,3,5-	3,4,5-	2,3,5,6-	2,3,4,5-	
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.528
E4	0.000	0.000	0.000	-0.302	0.000	0.529	0.000	3.166
E5	0.000	0.000	0.000	-0.302	0.000	0.281	-0.439	3.057
E6	0.000	0.000	0.000	-0.308	-0.152	0.529	0.000	3.166
E7	0.000	0.000	0.000	0.000	0.000	0.188	0.000	2.639
E8	0.000	0.000	0.000	0.000	0.000	0.201	0.000	2.311
E9	0.000	0.000	0.000	0.000	0.000	0.210	-0.158	2.411
Week 5	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D5	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
E5	0.000	0.000	0.000	-0.508	0.000	0.000	0.883	1.807
E6	0.000	0.000	0.000	-0.001	0.000	-0.323	1.086	1.625
E7	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.830
E8	0.000	0.000	0.000	0.000	0.000	0.000	1.086	1.916
E9	0.000	0.000	0.000	0.000	0.000	-0.233	0.807	1.465

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture after Draining (µg)

Week 6	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	-0.566	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	-0.753	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D6	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	-0.481	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	-0.572	0.000	0.000
E6	0.000	0.000	0.000	-0.472	-0.252	0.670	2.142	2.264
E7	0.000	0.000	0.000	0.000	0.000	0.690	2.142	2.264
E8	0.000	0.000	0.000	0.000	0.000	0.761	1.856	2.264
E9	0.000	0.000	0.000	0.000	0.000	0.857	1.793	2.264

Week 7	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
A9	0.000	0.000	0.000	0.000	0.000	-0.482	0.000	0.000
B7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C7	0.000	0.000	0.000	0.000	0.000	0.000	0.000	-0.390
C8	0.000	0.000	0.000	0.000	0.000	-0.638	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
D7	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
D8	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
D9	0.000	0.000	0.000	0.000	0.000	0.000	0.711	0.000
E7	0.000	0.000	0.000	0.000	-0.187	0.845	1.382	0.654

Table 6 (continued). Mass of Chlorophenols Remaining in Treatment Mixture Draining (μg)

	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
E8	0.000	0.000	0.000	0.000	0.000	0.205	1.433	0.628
E9	0.000	0.000	0.000	0.000	0.000	0.726	1.798	0.511
Week 8	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A8	0.000	0.000	0.000	0.000	0.000	-1.656	0.000	-0.386
A9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
B8	0.000	0.000	0.000	0.000	0.000	-4.140	0.000	-0.600
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C8	0.000	0.000	0.000	0.000	0.000	-1.963	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	-0.634	0.000	0.000
D8	0.000	0.000	0.000	0.000	0.000	4.821	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	4.821	0.000	0.000
E8	0.000	0.000	0.000	0.000	0.000	-0.457	0.966	-0.681
E9	0.000	0.000	0.000	0.000	0.000	-0.672	0.966	-0.746
Week 9	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A9	0.000	0.000	0.000	0.000	0.000	-0.128	0.000	0.000
B9	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C9	0.000	0.000	0.000	0.000	0.000	-0.382	0.000	0.000
D9	0.000	0.000	0.000	0.000	0.000	-0.464	0.000	0.000
E9	0.000	0.000	0.000	0.000	-0.130	-0.661	-0.316	-0.257

Table 7. Mass of Chlorophenols in Soil Slurry contained in Destructively
Sampled Reactors (μg)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A1	0.000	0.000	0.000	0.000	0.000	11.633	0.845	55.892
A2	0.000	0.000	0.000	0.000	0.000	10.560	1.437	61.079
A3	0.000	0.000	0.000	0.000	0.000	6.853	0.850	39.482
A4	0.000	0.000	0.000	0.985	0.000	7.874	1.284	50.161
A5	0.000	0.000	0.000	2.368	0.000	5.599	3.681	37.550
A6	0.000	0.000	0.000	0.000	0.000	3.684	2.014	33.133
A7	0.000	0.000	0.000	0.000	0.000	5.393	1.743	41.073
A8	0.000	0.000	0.000	2.602	0.000	7.009	5.636	45.254
A9	0.000	0.000	0.000	0.000	0.000	5.323	2.276	36.895
B1	0.000	0.000	0.000	0.000	0.000	20.014	1.705	93.225
B2	0.000	0.000	0.000	0.000	0.000	11.171	1.746	60.147
B3	0.000	0.000	0.000	1.897	0.000	6.930	3.773	36.899
B4	0.000	0.000	0.000	3.667	2.272	4.090	8.563	33.528
B5	0.000	0.000	0.000	4.921	3.939	3.212	11.773	23.852
B6	0.000	8.760	0.000	0.000	13.353	3.229	5.575	24.206
B7	0.000	3.165	0.000	5.197	4.731	7.090	13.119	32.329
B8	0.000	0.000	0.000	5.025	4.516	4.526	11.890	35.466
B9	0.000	0.000	0.000	5.850	10.041	6.356	15.883	39.291
C1	0.000	0.000	0.000	0.000	0.000	17.400	1.331	82.023
C2	0.000	0.000	0.000	0.000	0.000	8.564	2.013	59.774
C3	0.000	0.000	0.000	1.336	0.000	8.614	2.294	53.489
C4	0.000	0.000	0.000	0.871	0.000	4.823	1.940	38.820
C5	0.000	0.000	0.000	2.355	0.000	3.351	4.303	22.548
C6	0.000	0.000	0.000	2.143	4.916	2.884	6.920	19.067
C7	0.000	0.000	0.000	3.871	1.523	6.666	9.822	58.170
C8	0.000	0.000	0.000	2.970	4.177	3.214	6.571	26.634
C9	0.000	0.000	0.000	3.507	10.150	9.950	9.532	69.473
D1	0.000	0.000	0.000	1.227	0.000	20.043	1.711	94.163
D2	0.000	0.000	0.000	2.493	0.000	10.019	6.376	59.567
D3	0.000	3.367	0.000	5.063	3.112	7.761	13.849	52.762
D4	0.000	4.465	0.000	4.223	5.749	3.681	14.610	31.979
D5	0.000	3.645	0.000	5.524	5.400	4.204	16.592	34.085
D6	0.000	0.000	0.000	6.185	10.269	5.389	21.626	50.127
D7	0.000	3.012	0.000	4.586	5.827	3.519	15.272	42.715
D8	0.000	5.521	0.000	3.437	9.413	8.137	17.237	43.354
D9	0.000	0.000	0.000	4.846	6.400	4.583	19.186	38.347

Table 7 (continued). Mass of Chlorophenols in Soil Slurry contained in
Destructively Sampled Reactors (μg)

Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
E1	0.000	0.000	0.000	2.030	0.912	18.654	5.069	88.721
E2	0.000	0.000	0.000	4.278	1.861	13.519	13.376	74.453
E3	0.000	2.344	0.000	5.527	2.537	7.857	17.202	47.439
E4	0.000	3.187	0.000	7.006	2.966	4.685	18.223	43.290
E5	0.000	4.337	0.000	5.185	3.924	2.676	10.209	25.451
E6	0.000	0.000	0.000	2.885	9.744	1.593	10.110	15.185
E7	0.000	0.000	0.000	4.271	2.277	6.992	14.415	64.472
E8	0.000	0.000	0.000	1.932	2.092	6.689	9.154	45.299
E9	0.000	9.068	0.000	2.021	2.095	3.333	12.142	33.260

Table 8. Mass of Chlorophenols in Destructively Sampled Reactors adjusted
for washout (µg)

Week 1	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
A0	0.000	0.000	0.000	0.000	0.000	33.568	3.281	135.657
A1	0.000	0.000	0.000	0.000	0.000	11.945	0.845	56.126
A2	0.000	0.000	0.000	0.000	0.000	10.388	1.437	60.811
A3	0.000	0.000	0.000	0.000	0.000	6.215	0.850	38.460
A4	0.000	0.000	0.000	0.985	0.000	7.680	1.284	49.972
A5	0.000	0.000	0.000	2.368	0.000	5.223	3.681	37.188
A6	0.000	0.000	0.000	0.000	0.000	3.584	2.014	33.012
A7	0.000	0.000	0.000	0.000	0.000	4.795	1.743	40.300
A8	0.000	0.000	0.000	2.602	0.000	5.173	5.636	44.698
A9	0.000	0.000	0.000	0.000	0.000	3.895	2.276	35.817
B0	0.000	0.000	0.000	0.000	0.000	34.845	3.682	144.582
B1	0.000	0.000	0.000	0.000	0.000	20.338	1.705	93.424
B2	0.000	0.000	0.000	0.000	0.000	11.324	1.746	60.253
B3	0.000	0.000	0.000	1.897	0.000	7.556	3.773	37.899
B4	0.000	0.000	0.000	3.667	2.272	4.926	8.563	34.228
B5	0.000	0.000	0.000	4.921	3.939	4.916	11.773	26.420
B6	0.000	8.760	0.000	0.000	13.353	4.077	5.575	25.706
B7	0.000	3.165	0.000	5.197	4.731	9.821	13.119	35.093
B8	0.000	0.000	0.000	5.025	4.516	9.874	11.890	36.679
B9	0.000	0.000	0.000	5.850	10.041	6.874	15.883	40.265
C0	0.000	0.000	0.000	0.000	0.000	36.610	3.862	148.300
C1	0.000	0.000	0.000	0.000	0.000	17.720	1.331	82.310
C2	0.000	0.000	0.000	0.000	0.000	8.864	2.013	59.967
C3	0.000	0.000	0.000	1.336	0.000	8.614	2.294	53.489
C4	0.000	0.000	0.000	0.871	0.000	5.039	1.940	39.019
C5	0.000	0.000	0.000	2.355	0.000	3.351	4.303	22.629
C6	0.000	0.000	0.000	2.143	4.916	2.884	6.920	19.140
C7	0.000	0.000	0.000	3.871	1.523	6.666	9.822	58.949
C8	0.000	0.000	0.000	2.970	4.177	6.082	6.571	26.904
C9	0.000	0.000	0.000	3.507	10.150	11.105	9.532	69.596
D0	0.000	0.000	0.000	0.000	0.000	29.665	2.910	119.445
D1	0.000	0.000	0.000	1.227	0.000	20.148	1.711	93.565
D2	0.000	0.000	0.000	2.493	0.000	11.191	6.376	59.522
D3	0.000	3.367	0.000	5.063	3.112	8.046	13.849	50.879
D4	0.000	4.465	0.000	4.223	5.749	4.114	14.610	29.883
D5	0.000	3.645	0.000	5.524	5.400	4.767	16.592	31.909

Table 8 (continued). Mass of Chlorophenols in Destructively Sampled Reactors adjusted for washout (μg)

Week 2	3-CP	3,5-DCP	3,4-DCP	2,3,5-TCP	3,4,5-TCP	2,3,5,6-TeCP	2,3,4,5-TeCP	PCP
D6	0.000	0.000	0.000	6.185	10.269	5.800	21.626	47.877
D7	0.000	3.012	0.000	4.586	5.827	4.062	14.561	40.768
D8	0.000	5.521	0.000	3.437	9.413	4.205	16.526	41.111
D9	0.000	0.000	0.000	4.846	6.400	0.895	18.475	36.436
E0	0.000	0.000	0.000	0.000	0.000	29.665	2.910	119.445
E1	0.000	0.000	0.000	2.030	0.912	18.704	5.069	85.064
E2	0.000	0.000	0.000	4.278	1.861	14.206	13.376	71.415
E3	0.000	2.344	0.000	5.527	2.537	8.425	17.044	41.104
E4	0.000	3.187	0.000	7.994	2.966	3.785	17.334	32.537
E5	0.000	4.337	0.302	5.858	3.643	3.599	6.878	14.590
E6	0.000	0.000	0.000	4.211	10.459	0.368	7.902	0.591
E7	0.000	0.000	0.000	4.271	2.464	5.116	8.916	50.042
E8	0.000	0.000	0.000	1.932	2.092	6.177	2.924	32.571
E9	0.000	9.068	0.000	2.021	2.226	3.892	6.570	22.343

Table 9. Concentrations of Chlorophenols in Soil Pore Water (nmol/L)

Time	2,3,5-TCP			3,4,5-TCP			2,3,5,6-TeCP		
(days)	A	B	C	A	B	C	A	B	C
0	0.00	0.00	0.00	0.00	0.00	0.00	2.83	2.83	2.83
7	0.00	0.00	0.00	0.00	0.00	0.00	1.55	0.00	0.53
14	0.00	0.00	0.00	0.00	0.00	0.00	1.31	1.50	0.18
21	0.00	0.00	0.00	0.00	0.00	0.00	0.62	0.75	0.12
28	0.00	0.00	0.00	0.00	0.00	0.00	0.59	0.71	0.12
35	0.00	0.00	0.00	0.00	0.00	0.00	0.64	0.74	0.10
43	0.00	0.02	0.00	0.00	0.02	0.00	0.67	0.77	0.11
50	0.00	0.04	0.00	0.00	0.04	0.00	1.70	2.02	0.18
57	0.00	0.03	0.00	0.00	0.04	0.00	1.52	1.96	0.16
63	0.00	0.03	0.00	0.00	0.03	0.00	1.62	1.87	0.13
70	0.00	0.05	0.00	0.00	0.00	0.00	1.64	1.83	0.29
77	0.00	0.04	0.00	0.00	0.03	0.00	1.85	2.18	0.25
84	0.00	0.04	0.00	0.00	0.03	0.00	1.44	1.68	0.36
98	0.00	0.04	0.00	0.00	0.03	0.00	2.03	2.38	0.73
105	0.00	0.04	0.00	0.00	0.00	0.00	2.03	2.29	0.70
119	0.04	0.06	0.00	0.00	0.05	0.00	0.87	2.08	1.07
126	0.00	0.00	0.00	0.00	0.00	0.00	1.84	1.70	0.95
140	0.00	0.08	0.05	0.00	0.06	0.00	2.33	2.44	1.31
161	0.00	0.30	0.00	0.00	0.00	0.00	0.16	2.26	1.31

Table 9 (continued). Concentrations of Chlorophenols in Soil Pore Water (nmol/L)

Time	2,3,4,5-TeCP			PCP		
(days)	A	B	C	A	B	C
0	0.00	0.00	0.00	1.29	1.29	1.29
7	0.00	0.00	0.00	0.66	0.00	0.72
14	0.00	0.00	0.00	0.87	1.22	0.28
21	0.00	0.00	0.00	0.94	0.92	0.86
28	0.00	0.00	0.00	0.38	0.66	0.26
35	0.00	0.00	0.00	0.46	0.69	0.26
43	0.02	0.03	0.00	0.49	0.74	0.18
50	0.03	0.06	0.00	0.97	1.52	0.31
57	0.02	0.05	0.00	0.84	1.53	0.27
63	0.02	0.04	0.00	0.78	2.03	0.21
70	0.00	0.06	0.00	0.91	1.36	0.34
77	0.00	0.04	0.00	0.77	1.17	0.32
84	0.00	0.03	0.02	0.47	0.71	0.38
98	0.00	0.04	0.03	0.71	1.13	0.76
105	0.00	0.03	0.00	0.63	0.98	0.73
119	0.00	0.06	0.05	0.80	1.15	1.07
126	0.00	0.00	0.00	0.63	0.75	0.83
140	0.00	0.08	0.06	0.89	1.15	1.16
161	0.00	0.08	0.23	0.00	1.05	1.14

Table 10. Concentrations of Chlorophenols in Soil (μmol/kg)

Time	2,3,5-TCP			3,4,5-TCP			2,3,5,6-TeCP		
(days)	A	B	C	A	B	C	A	B	C
0	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.05	0.05
7	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.03
14	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.01	0.04
21	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00
28	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.02
35	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01
43	0.00	0.00	0.15	0.21	0.22	0.00	0.01	0.01	0.02
50	0.00	0.00	0.22	0.00	0.16	0.15	0.02	0.01	0.05
57	0.00	0.00	0.15	0.11	0.11	0.10	0.01	0.01	0.05
63	0.00	0.00	0.13	0.00	0.22	0.00	0.01	0.01	0.04
70	0.00	0.00	0.13	0.00	0.00	0.00	0.01	0.01	0.03
77	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.04
84	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.04
98	0.00	0.00	0.22	0.00	0.00	0.00	0.01	0.01	0.07
105	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.03
119	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.01	0.03
126	0.00	0.00	0.19	0.27	0.00	0.00	0.02	0.01	0.04
140	0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.01	0.04
161	0.00	0.00	0.28	0.10	0.17	0.19	0.02	0.01	0.05

Table 10 (continued). Concentrations of Chlorophenols in Soil (μmol/kg)

Time (days)	2,3,4,5-TeCP			PCP		
	A	B	C	A	B	C
0	0.00	0.00	0.00	26.59	26.59	26.59
7	0.00	0.00	0.00	2.29	0.65	5.62
14	0.00	0.00	0.00	2.27	0.77	6.42
21	0.00	0.00	0.00	1.10	0.40	0.26
28	0.00	0.00	0.00	0.88	0.30	3.19
35	0.00	0.00	0.00	1.10	0.73	1.39
43	0.10	0.14	0.13	1.57	0.77	5.04
50	0.11	0.13	0.25	2.31	0.66	9.66
57	0.07	0.08	0.13	0.57	0.44	7.75
63	0.00	0.14	0.11	1.17	0.80	5.51
70	0.00	0.00	0.09	1.19	0.35	4.98
77	0.00	0.00	0.00	0.40	0.30	4.01
84	0.00	0.00	0.00	0.18	0.34	5.81
98	0.00	0.00	0.16	0.54	0.24	7.44
105	0.00	0.00	0.13	0.54	0.16	4.29
119	0.00	0.11	0.00	1.30	0.26	.2.19
126	0.19	0.79	0.20	0.71	0.50	5.46
140	0.00	0.00	0.20	1.57	0.59	5.11
161	0.06	0.07	0.27	0.56	0.16	8.68

Table 11. Chlorophenols in Pilot-Scale Reactor Effluent Treatment Mixtures

Time (days)	Drain Water (µg/L)					
	3,5 DCP	2,3,5 TCP	3,4,5 TCP	2,3,5,6- TeCP	2,3,4,5 TeCP	PCP
0	0.00	0.00	0.00	0.00	0.00	0.00
7	0.00	0.00	0.00	0.00	0.00	0.00
14	0.00	0.00	0.00	0.00	0.00	0.00
21	0.00	0.00	0.00	0.59	0.00	47.76
28	0.00	0.00	0.00	0.26	0.00	7.87
35	0.00	0.00	0.00	0.27	0.00	32.17
43	0.00	0.00	0.00	0.27	0.00	14.81
50	0.00	0.00	0.00	0.09	0.00	12.73
57	297.62	15.99	30.04	0.00	0.00	0.00
63	179.16	0.00	18.09	0.00	0.00	0.00
70	0.00	0.00	0.00	0.00	0.00	0.00
77	0.00	0.00	0.00	0.00	0.00	0.00
84	0.00	0.00	0.00	0.00	0.00	0.00
98	0.00	0.00	0.00	0.00	0.00	0.00
105	0.00	0.00	0.00	0.00	0.00	0.00
119	0.00	0.00	0.00	0.00	0.00	0.00
126	0.00	0.00	0.00	0.26	0.00	0.00
140	0.00	0.00	0.00	1.94	0.00	0.00
161	0.00	0.00	0.00	1.39	0.00	31.28

Table 11 (continued). Chlorophenols in Pilot-Scale Reactor Effluent Treatment Mixtures

Time (days)	Chlorophenols Removed (mg)					
	3,5 DCP	2,3,5 TCP	3,4,5 TCP	2,3,5,6- TeCP	2,3,4,5 TeCP	PCP
0	0.00	0.00	0.00	0.00	0.00	0.00
7	0.00	0.00	0.00	0.00	0.00	0.00
14	0.00	0.00	0.00	0.00	0.00	0.00
21	0.00	0.00	0.00	0.17	0.00	13.85
28	0.00	0.00	0.00	0.08	0.00	2.36
35	0.00	0.00	0.00	0.08	0.00	9.65
43	0.00	0.00	0.00	0.06	0.00	3.55
50	0.00	0.00	0.00	0.02	0.00	2.93
57	65.48	3.52	6.61	0.00	0.00	0.00
63	39.40	0.00	3.98	0.00	0.00	0.00
70	0.00	0.00	0.00	0.00	0.00	0.00
77	0.00	0.00	0.00	0.00	0.00	0.00
84	0.00	0.00	0.00	0.00	0.00	0.00
98	0.00	0.00	0.00	0.00	0.00	0.00
105	0.00	0.00	0.00	0.00	0.00	0.00
119	0.00	0.00	0.00	0.00	0.00	0.00
126	0.00	0.00	0.00	0.00	0.00	0.00
140	0.00	0.00	0.00	0.00	0.00	0.00
161	0.00	0.00	0.00	0.00	0.00	0.00
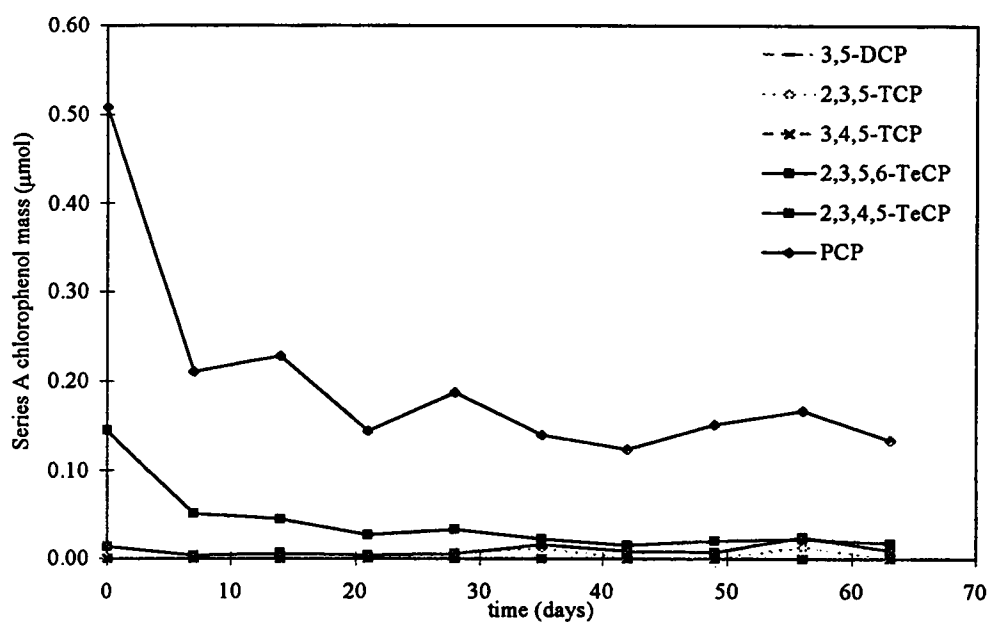
Figure 25. Mass of chlorophenols in bench-scale reactor series A
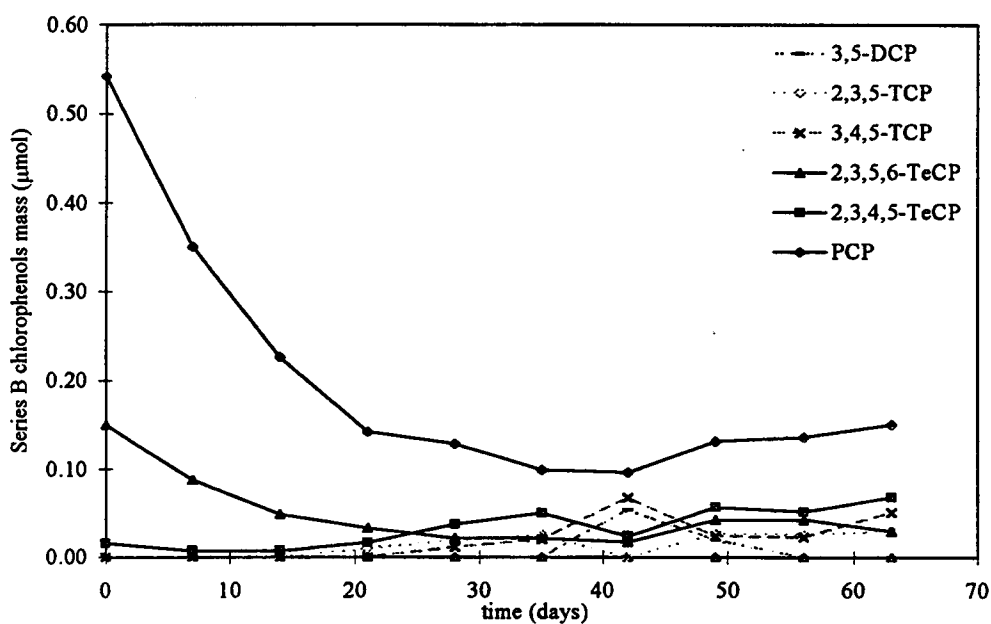
Figure 26. Mass of chlorophenols in bench-scale reactor series B
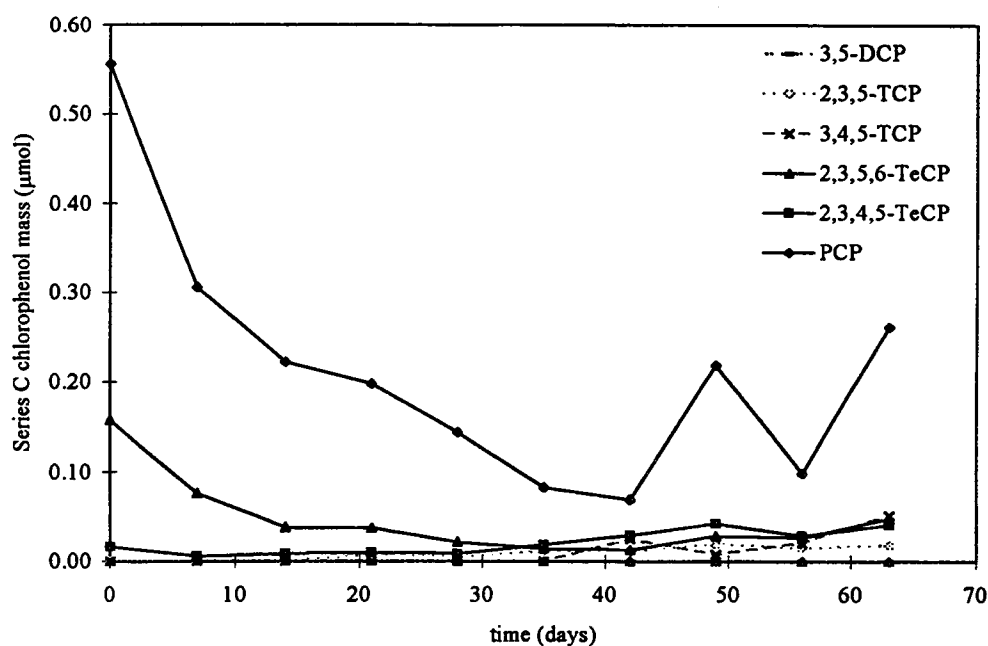
Figure 27. Mass of chlorophenols in bench-scale reactor series C
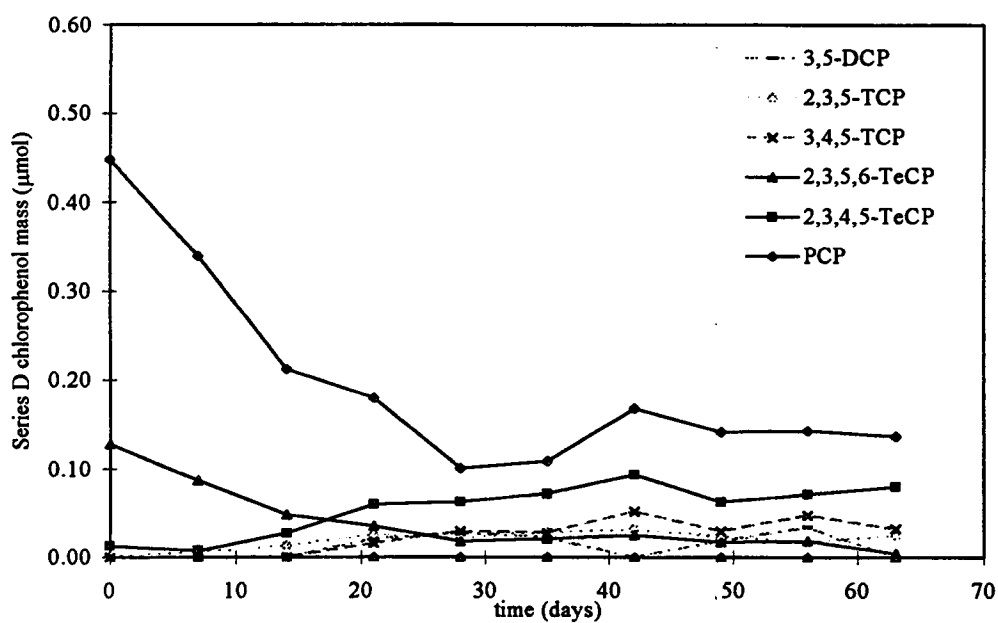
Figure 28. Mass of chlorophenols in bench-scale reactor series D
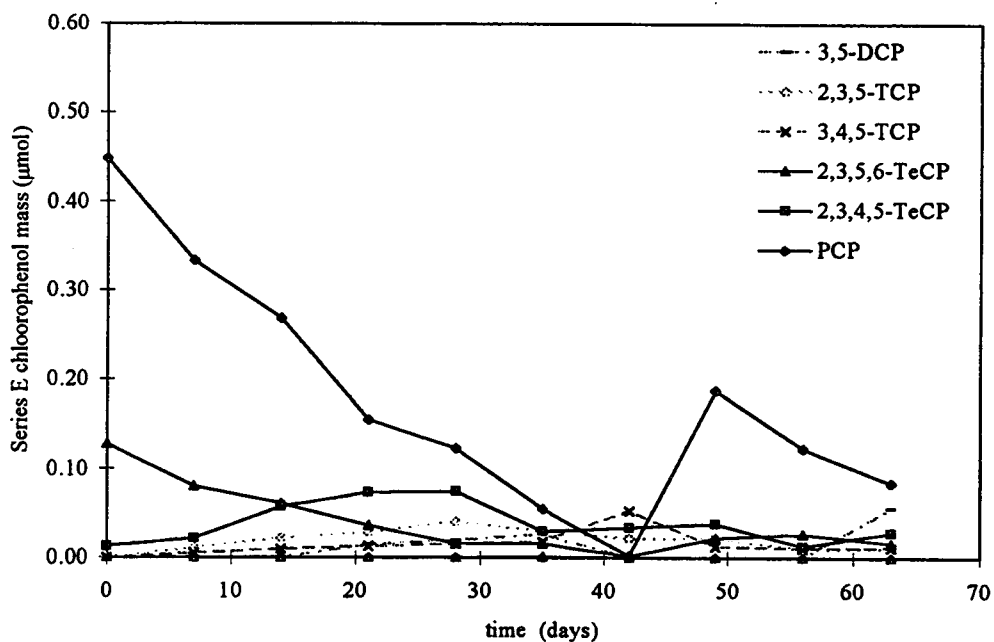
Figure 29. Mass of chlorophenols in bench-scale reactor series E
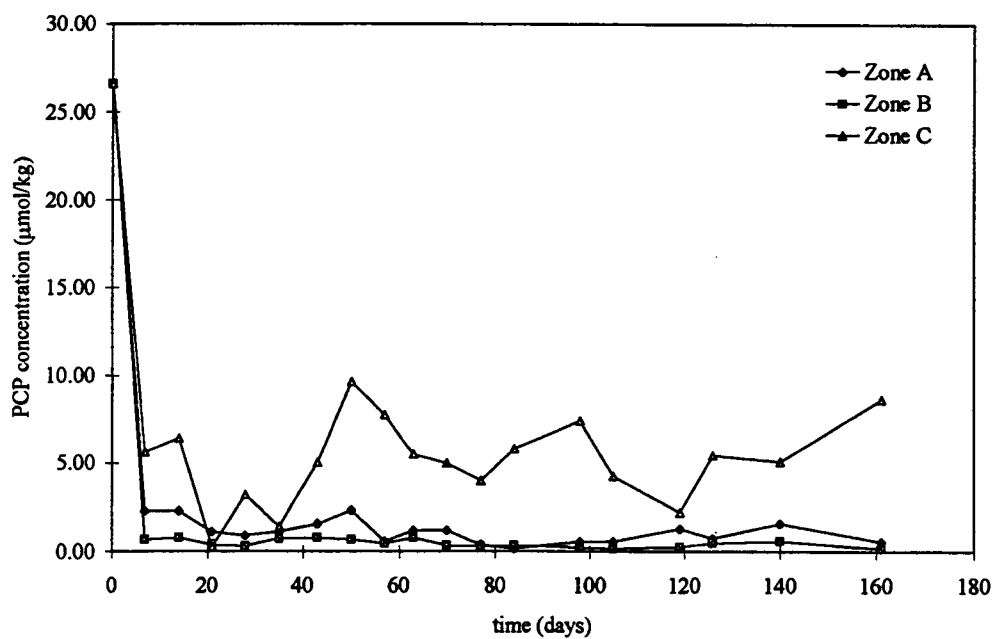
Figure 30. Concentration of PCP in pilot-scale reactor soil treatment zones
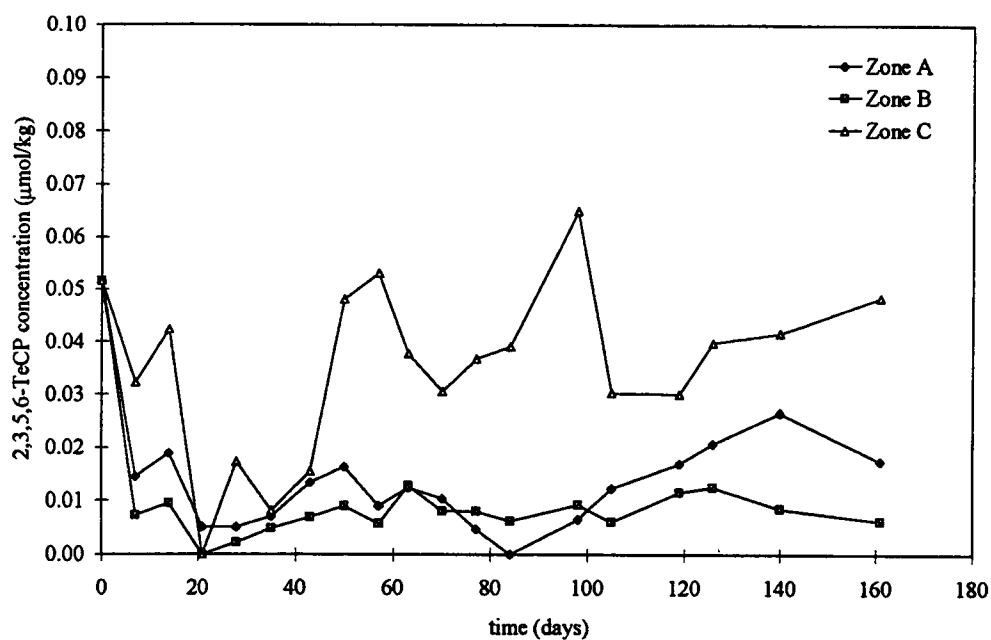
Figure 31. Concentration of 2,3,5,6-TeCP in pilot-scale reactor soil treatment zones
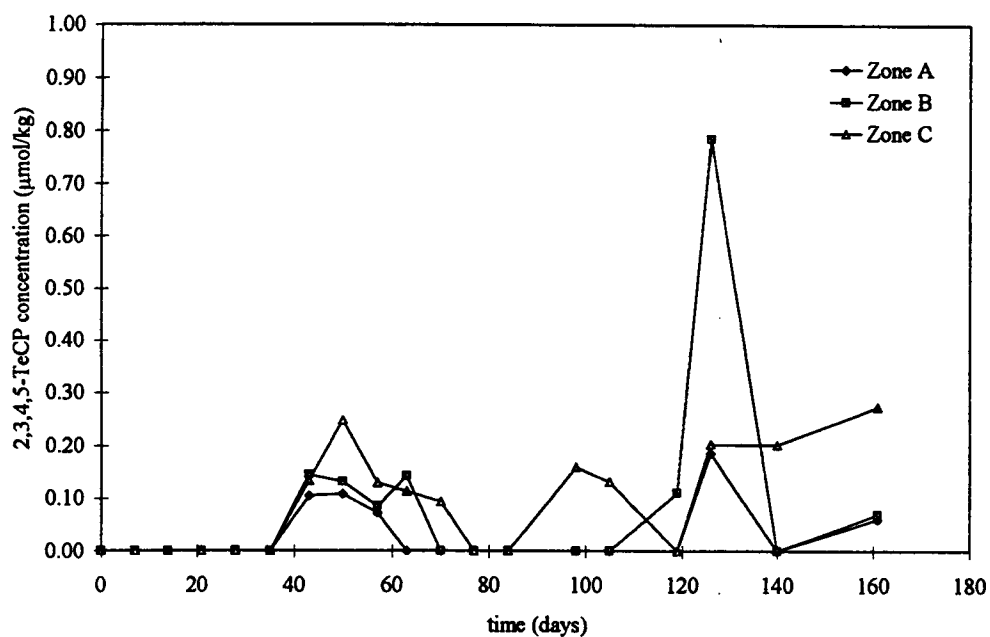
Figure 32. Concentration of 2,3,4,5-TeCP in pilot-scale reactor soil treatment zones
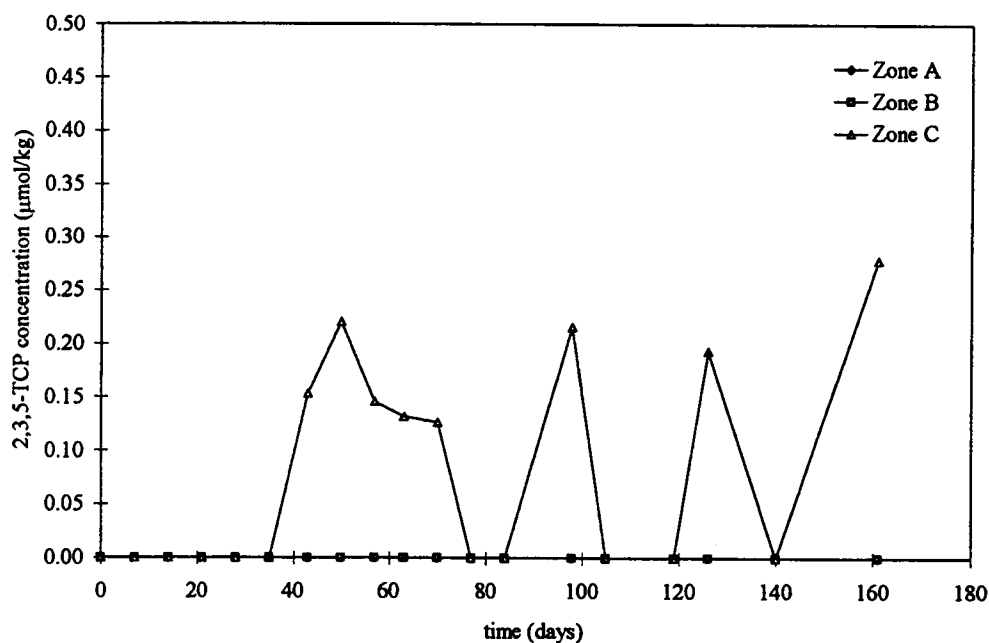
Figure 33. Concentration of 2,3,5-TCP in pilot-scale reactor soil treatment zones
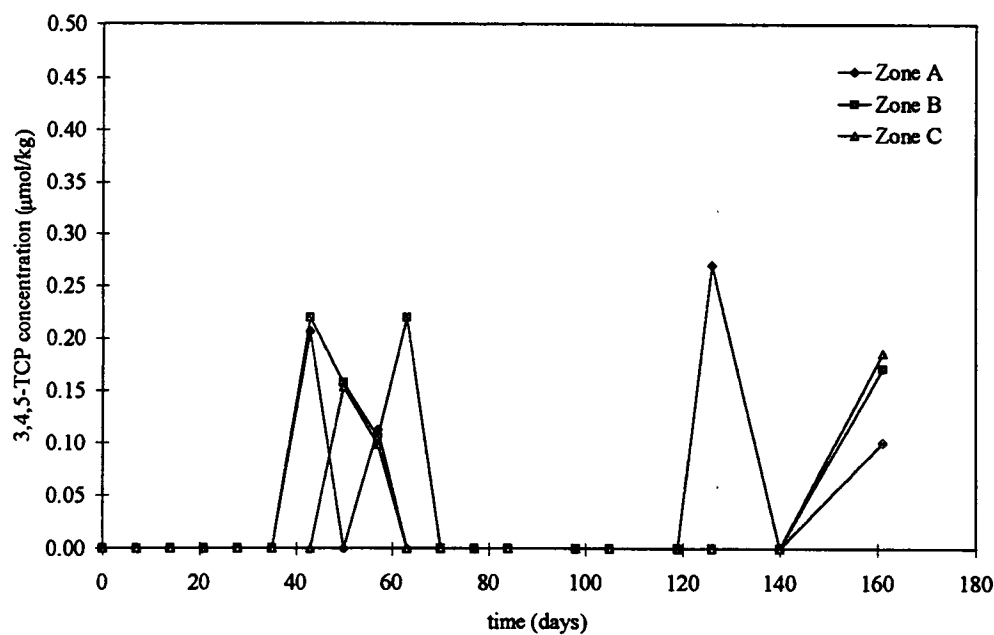
Figure 34. Concentration of 3,4,5-TCP in pilot-scale reactor soil treatment zones
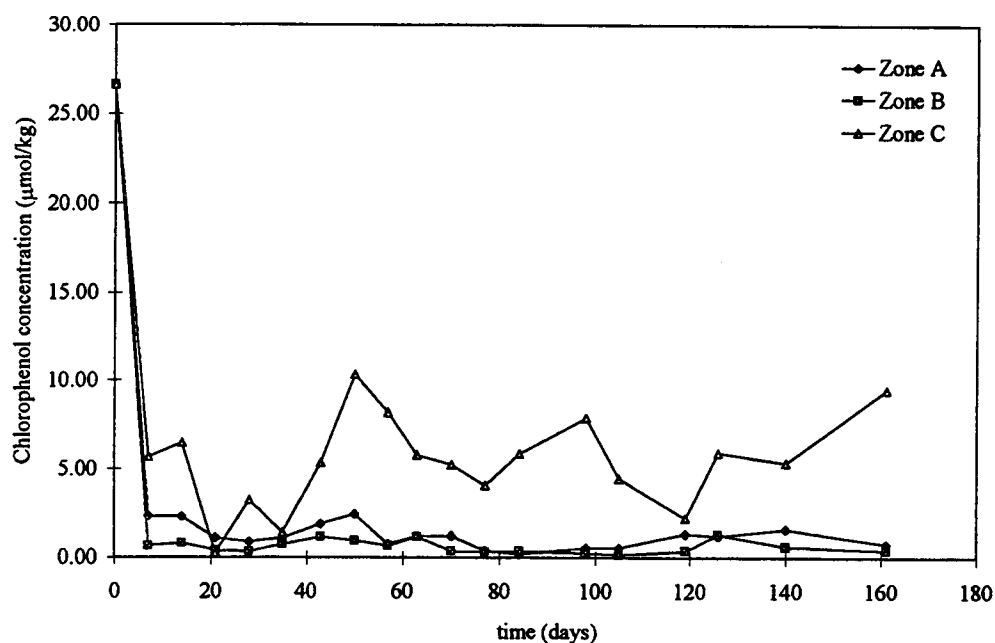
Figure 35. Mass balance on chlorophenols in pilot-scale reactor soil treatment zones
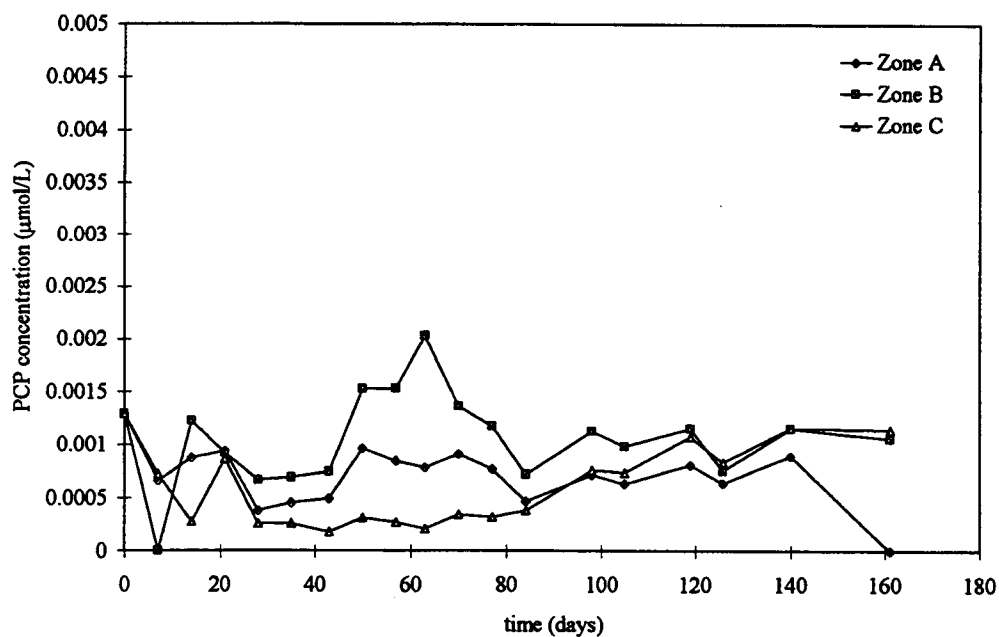
Figure 36. Concentration of PCP in pilot-scale reactor pore water
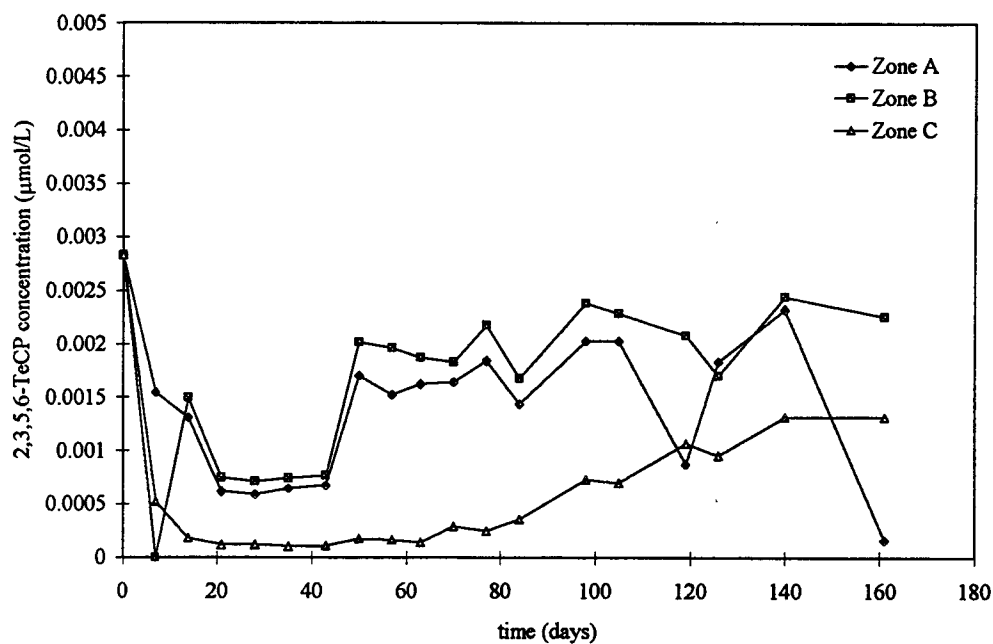
Figure 37. Concentration of 2,3,5,6-TeCP n pilot-scale reactor pore water
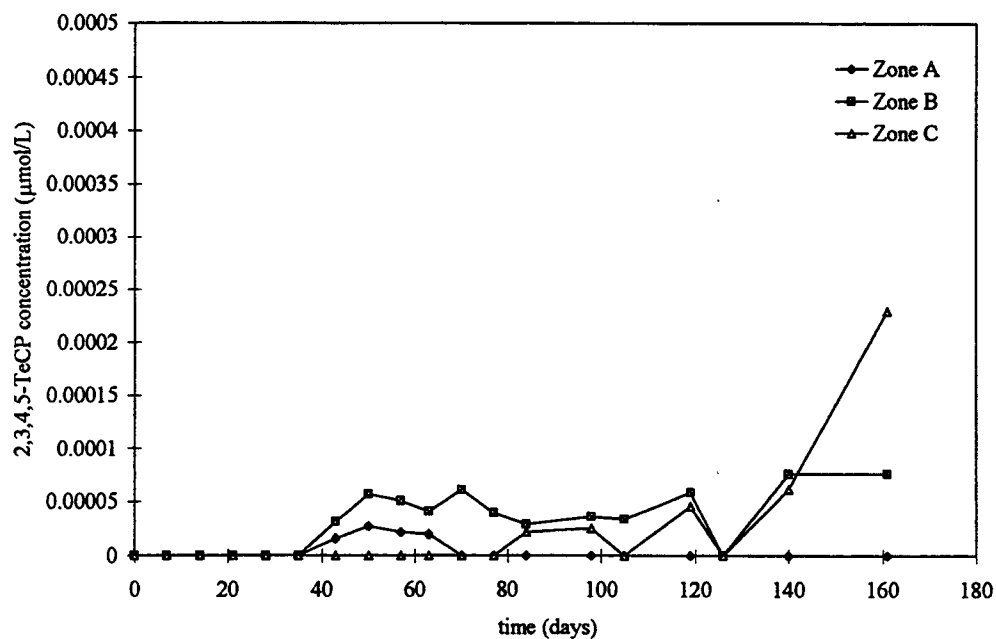
Figure 38. Concentration of 2,3,4,5-TeCP in pilot-scale reactor pore water
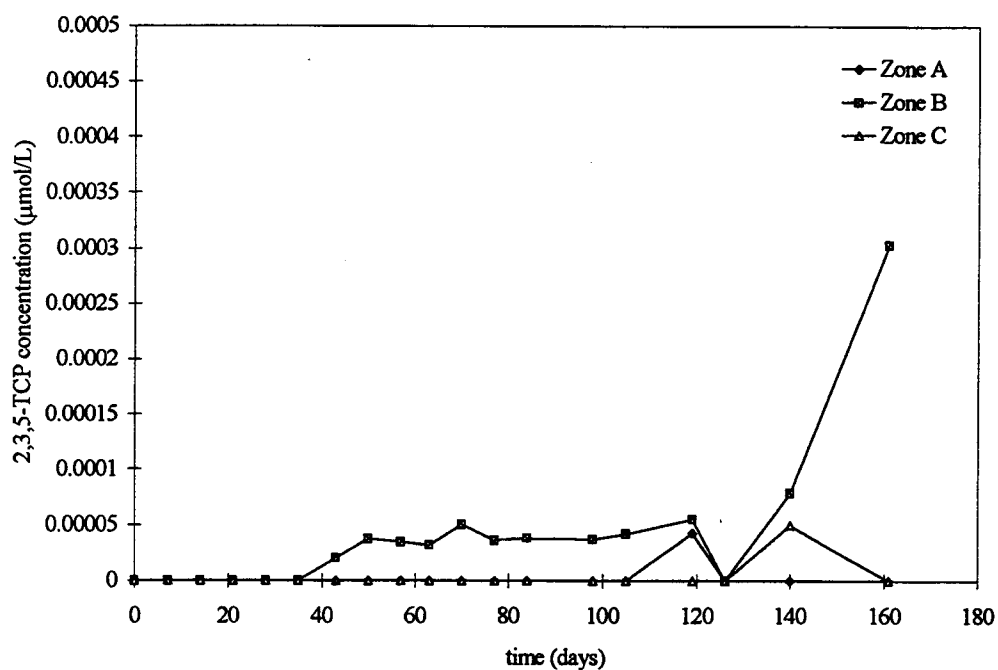
Figure 39. Concentration of 2,3,5-TCP in pilot-scale reactor pore water
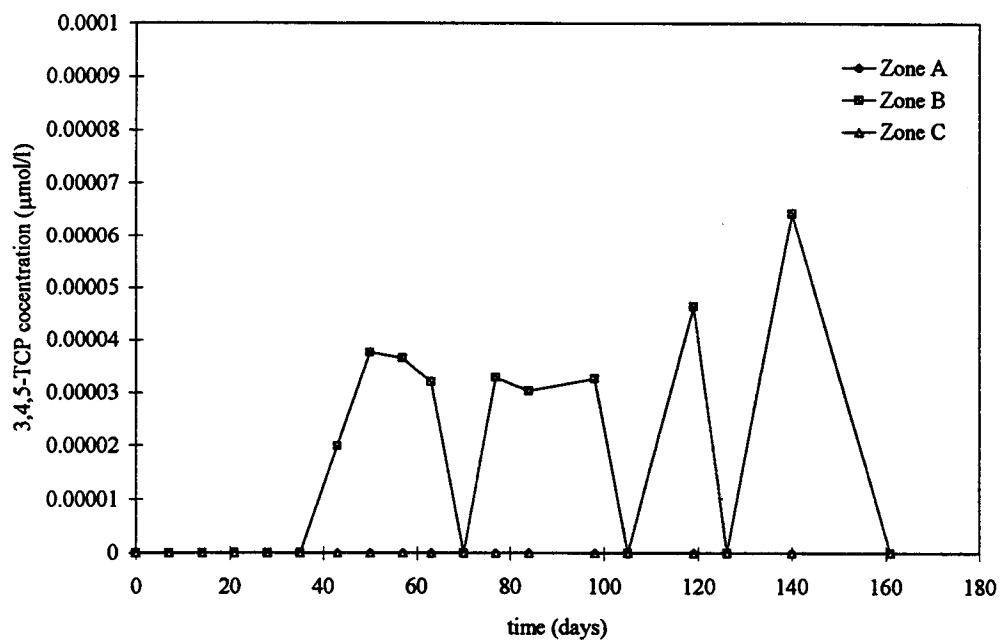
Figure 40. Concentration of 3,4,5-TCP in pilot-scale reactor pore water
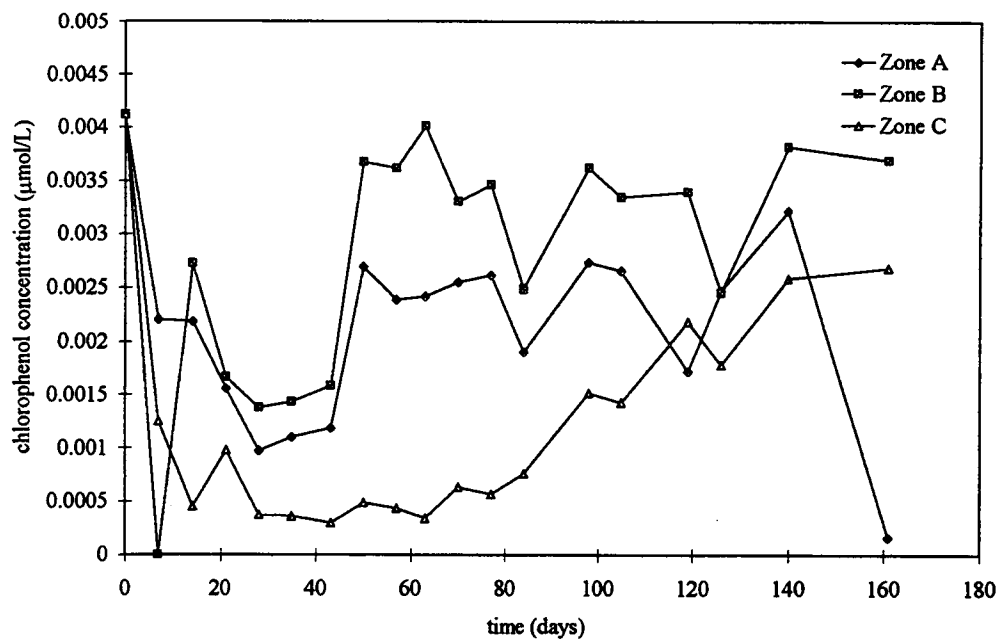
Figure 41. Mass balance on chlorophenols in pilot-scale reactor pore water
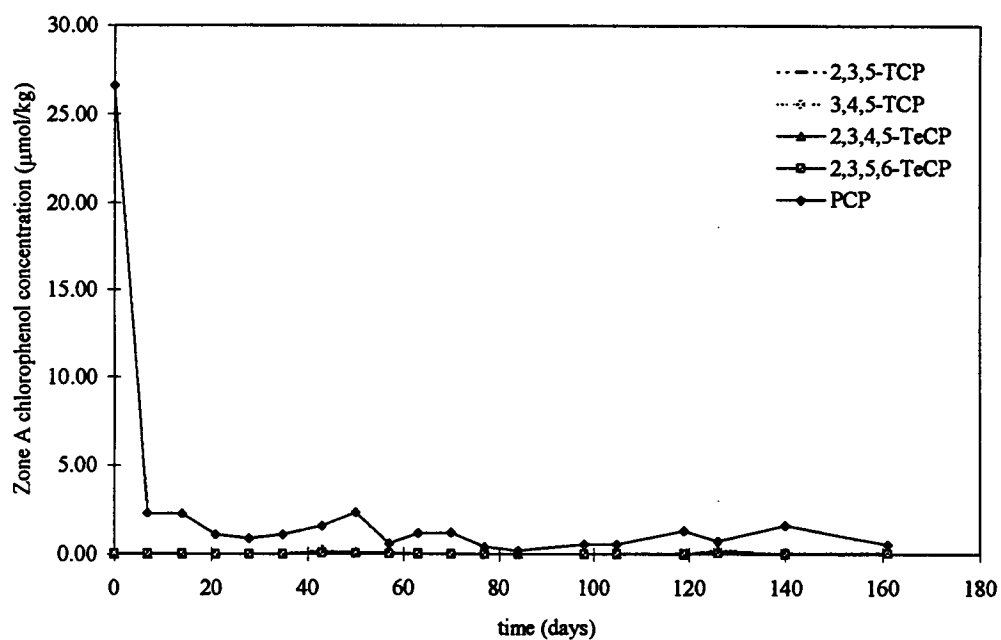
Figure 42. Concentration of chlorophenols in pilot-scale soil treatment zone A
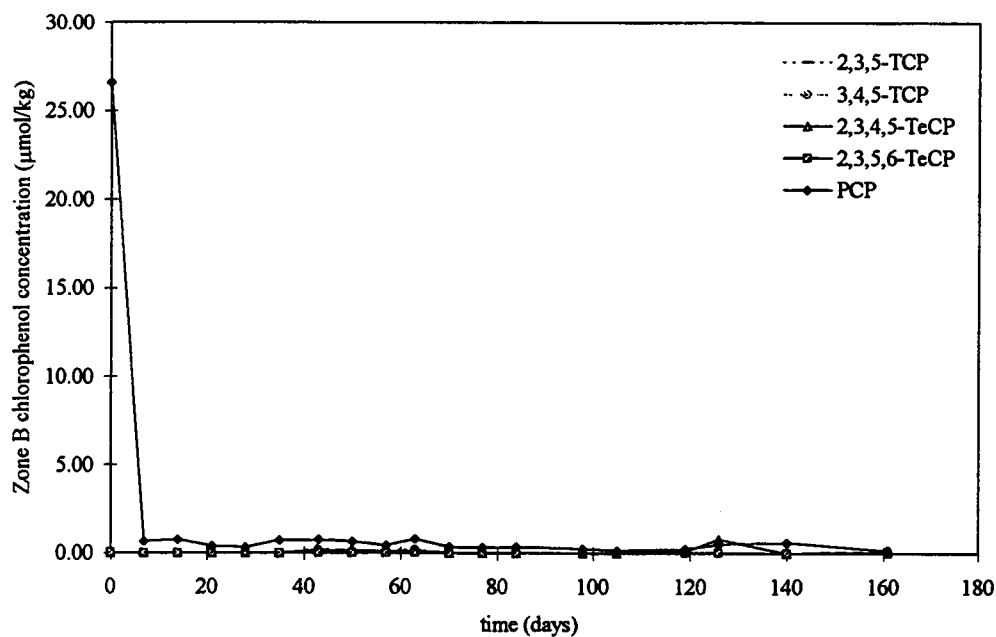
Figure 43. Concentration of chlorophenols in pilot-scale soil treatment zone B
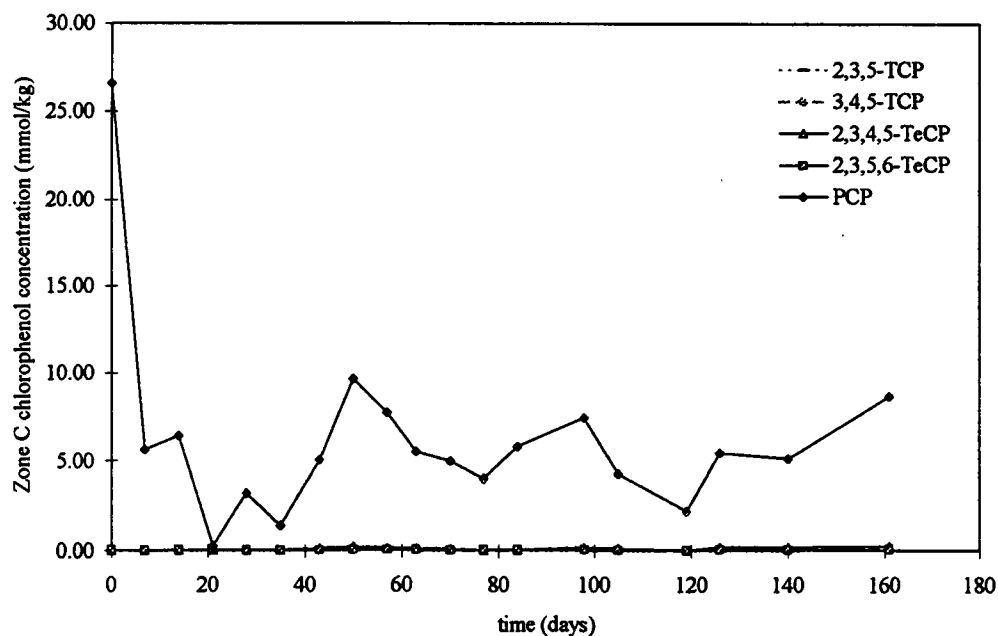
Figure 44. Concentration of chlorophenols in pilot-scale soil treatment zone C
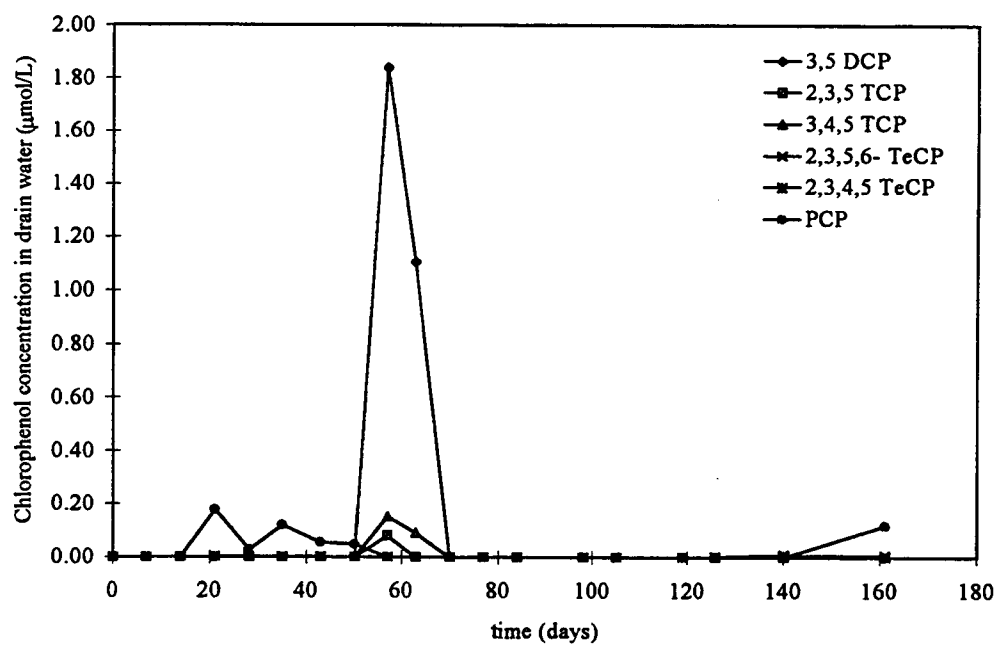
Figure 45. Concentration of chlorophenols in pilot-scale reactor drain water
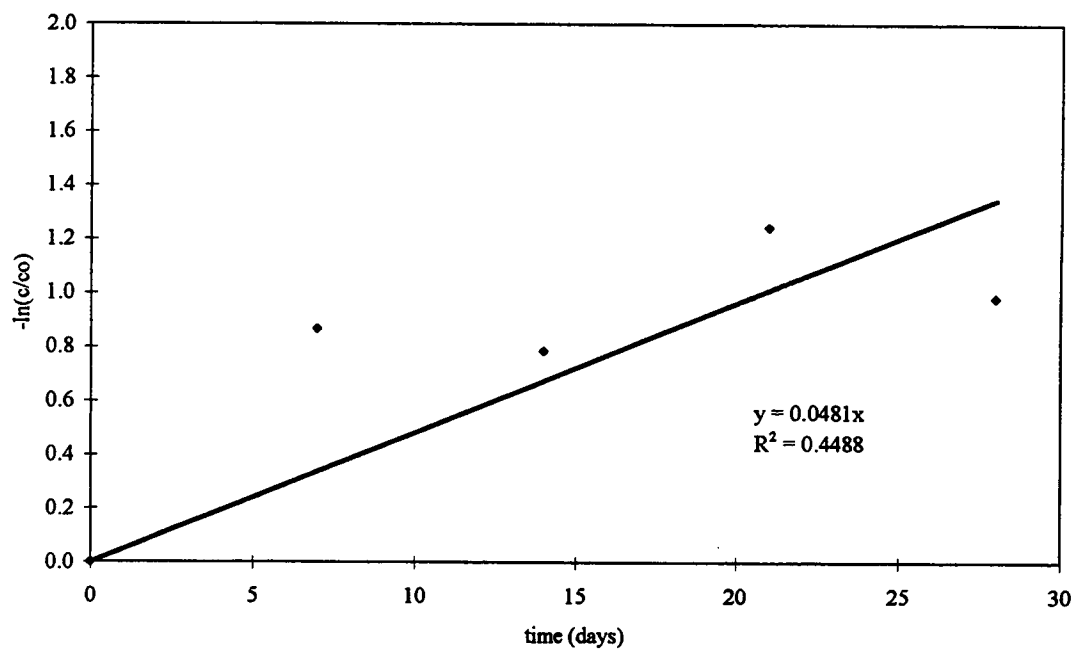
Figure 46. First order reaction kinetics model for PCP removal in Series A
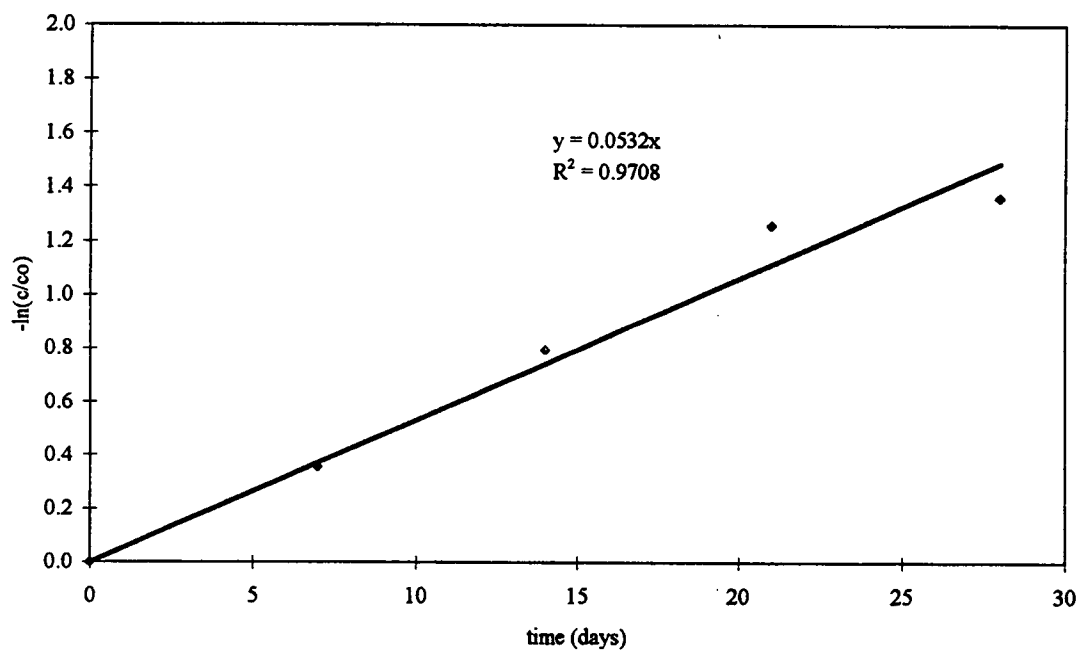
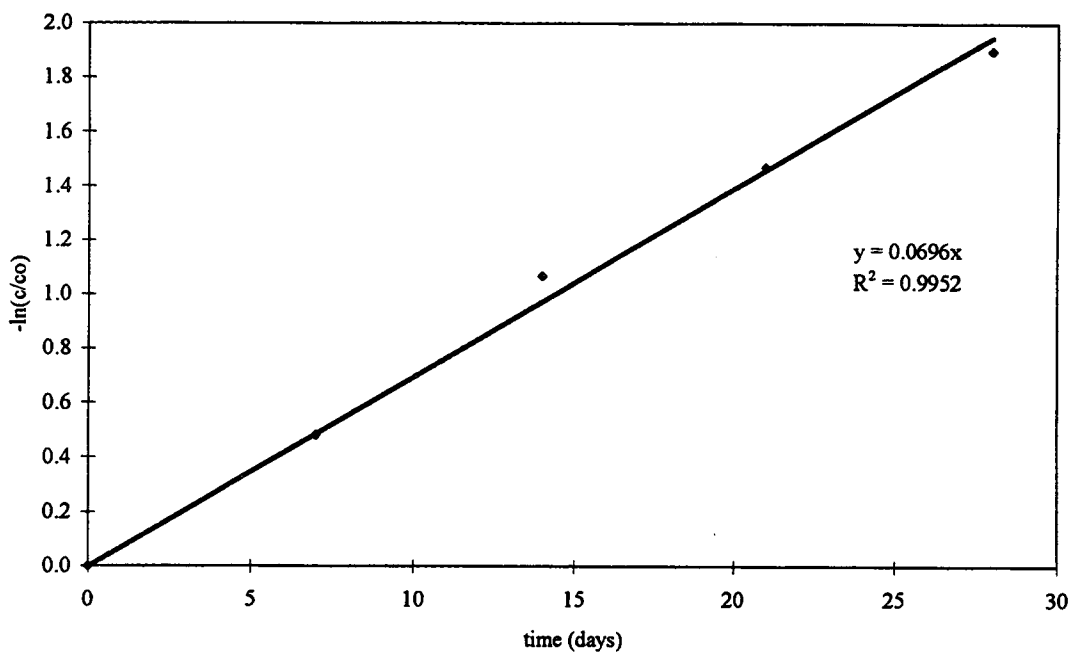Figure 47. First order reaction kinetics model for PCP removal in Series B
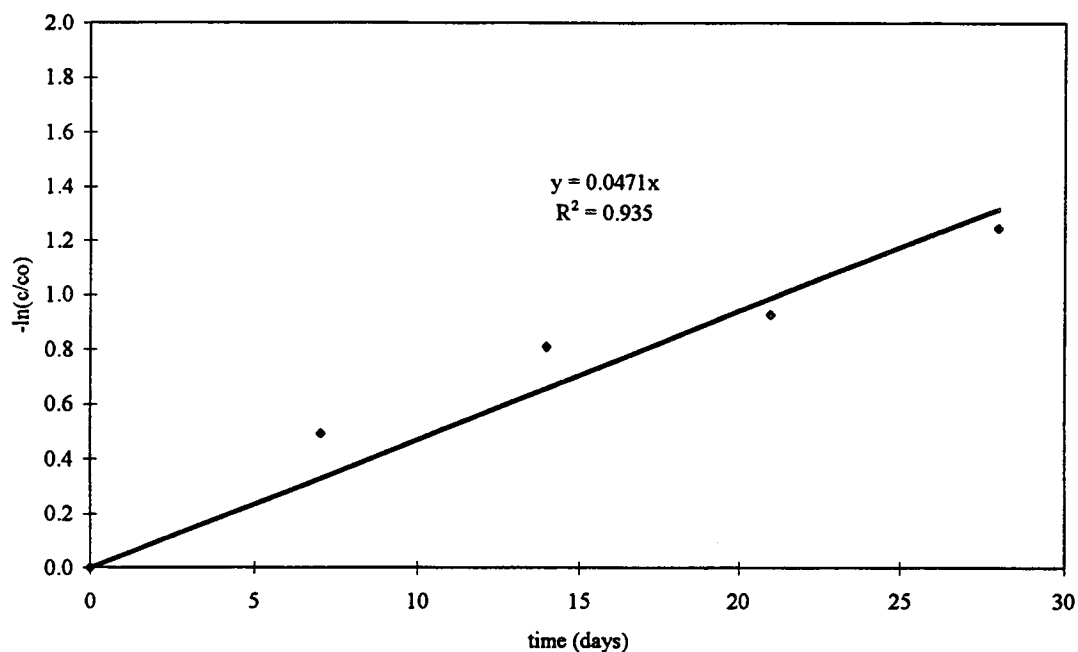
Figure 48. First order reaction kinetics model for PCP removal in Series C
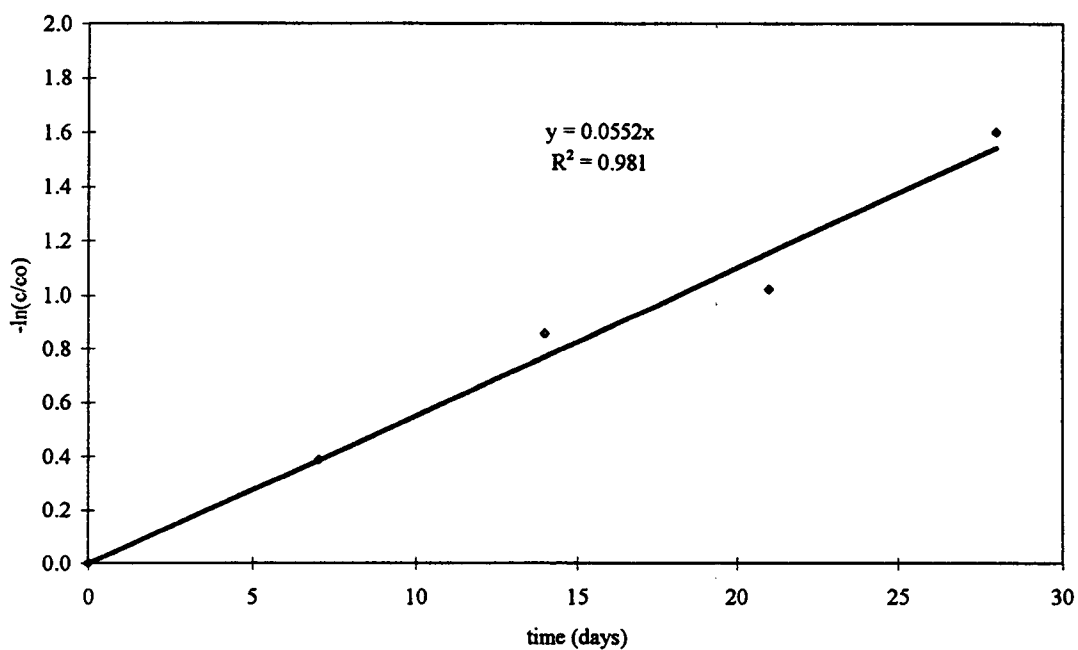
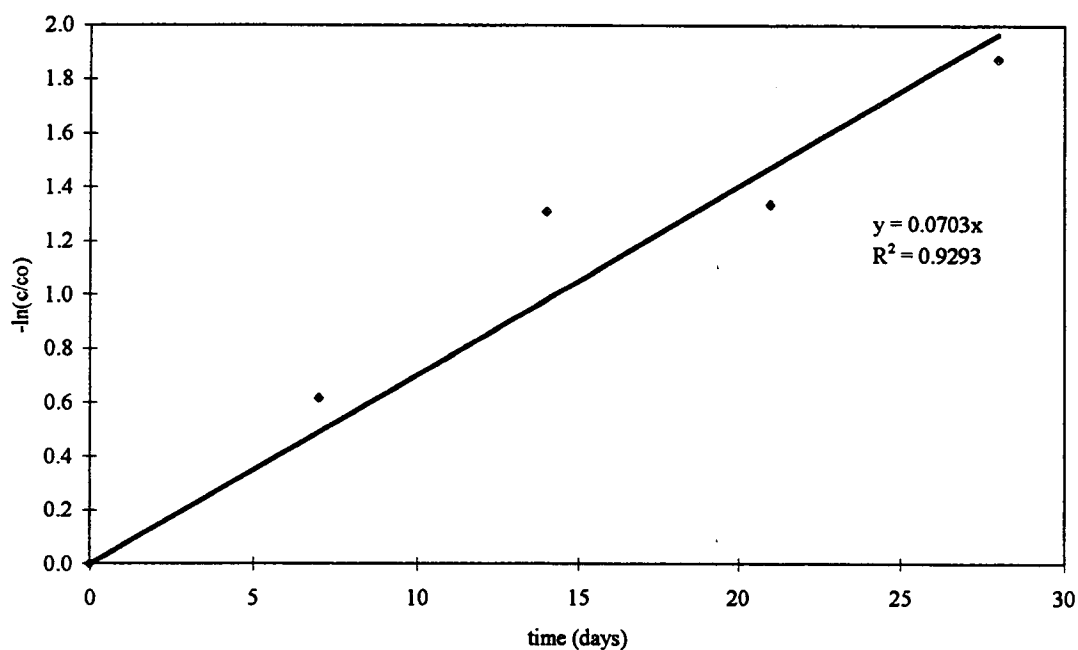Figure 49. First order reaction kinetics model for PCP removal in Series D
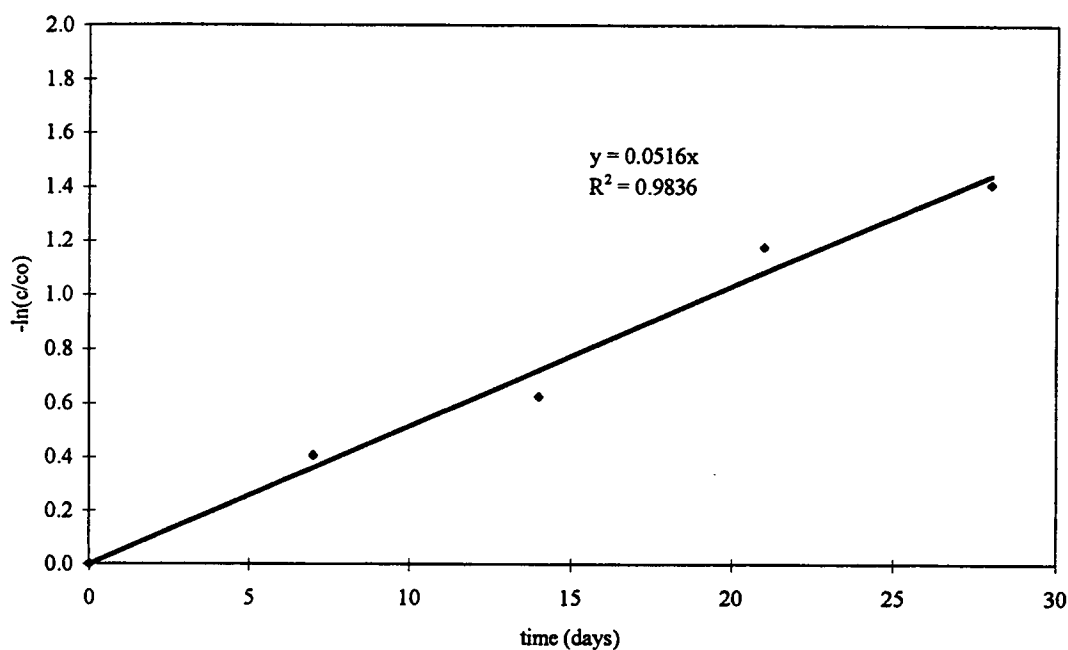
Figure 50. First order reaction kinetics model for PCP removal in Series E
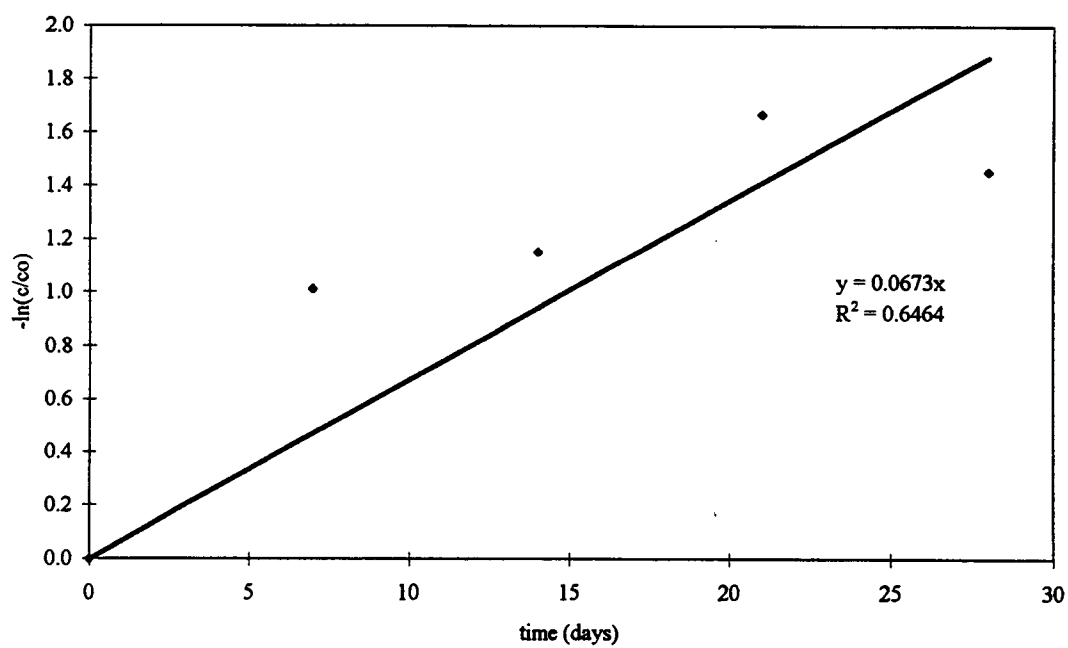
Figure 51. First order reaction kinetics model for 2,3,5,6-TeCP removal in Series A

Figure 52. First order reaction kinetics model for 2,3,5,6-TeCP removal in Series B

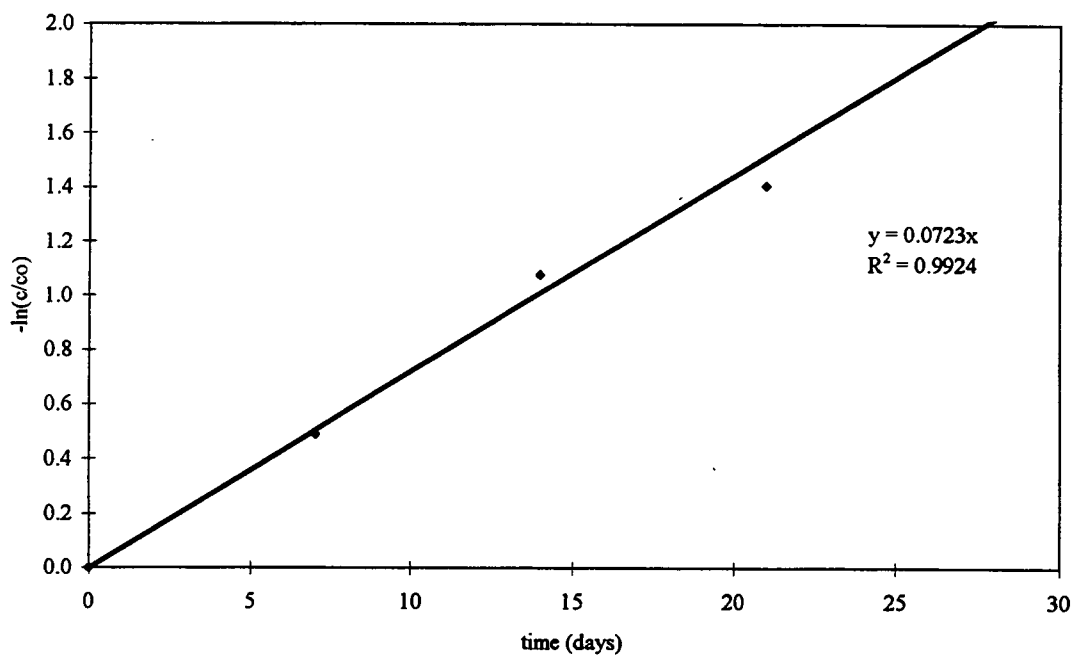Figure 53. First order reaction kinetics model for 2,3,5,6-TeCP removal in Series C

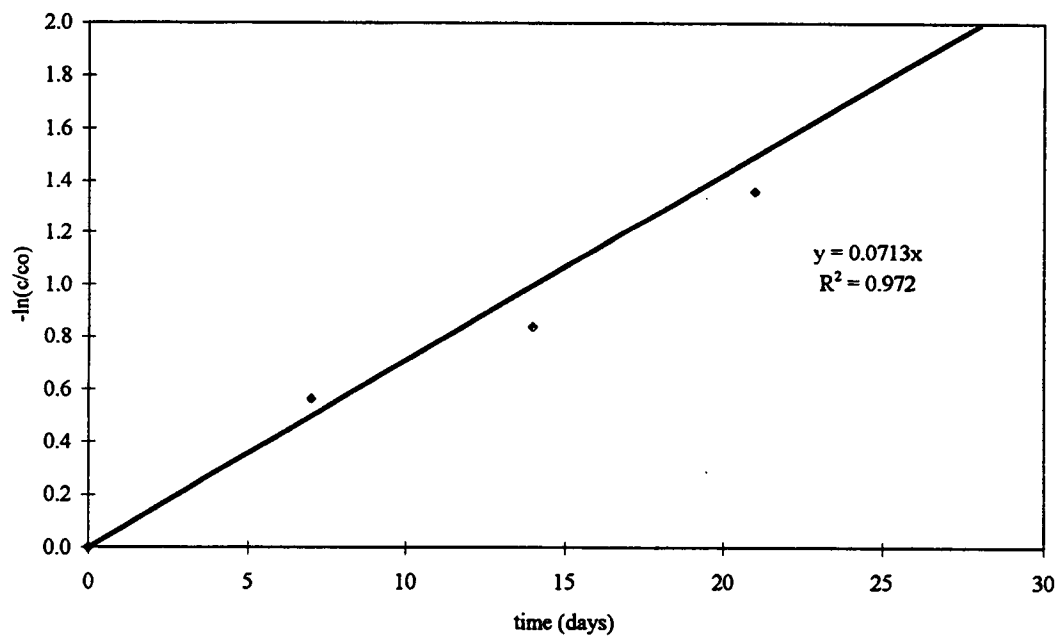Figure 54. First order reaction kinetics model for 2,3,5,6-TeCP removal in Series D

Figure 55. First order reaction kinetics model for 2,3,5,6-TeCP removal in Series E