PNNL-11998

0050598

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

K Basin Sludge Conditioning Process Testing

Fate of PCBs During K Basin Sludge Dissolution in Nitric Acid and With Hydrogen Peroxide Addition

G. M. Mong
A. J. Schmidt
E. W. Hoppe
K. H. Pool
K. L. Silvers
B. M. Thornton

APR 1999
RECEIVED
EDMC

July 1998

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights**. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161

This document was printed on recycled paper.
(9/97)

PNNL-11998

K Basin Sludge Conditioning Process Testing

Fate of PCBs During K Basin Sludge Dissolution in Nitric Acid and with Hydrogen Peroxide Addition

G. M. Mong
A. J. Schmidt
E. W. Hoppe
K. H. Pool
K. L. Silvers
B. M. Thornton

July 1998

Prepared for
Numatec Hanford Corporation

Work Supported by
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest National Laboratory
Richland, Washington 99352

THIS PAGE INTENTIONALLY
LEFT BLANK

Summary

The work described in this report is part of the studies being performed to address the fate of polychlorinated biphenyls (PCBs) in K Basin sludge before the sludge can be transferred to the Tank Waste Remediation System (TWRS) double shell tanks. One set of tests examined the effect of hydrogen peroxide on the disposition of PCBs in a simulated K Basin dissolver solution containing 0.5 M nitric acid/1 M $Fe(NO_3)_3$. A second series of tests examined the disposition of PCBs in a much stronger (~10 M) nitric acid solution, similar to that likely to be encountered in the dissolution of the sludge. The data strongly support the following conclusions:

The PCB congeners most representative of Aroclor 1254 (the predominant Aroclor found in the sludge) are not appreciably chemically altered by solutions of nitric acid or hydrogen peroxide at reflux temperatures. Mass balances of each PCB congener in the experimental tests were compared to control experiments in which only a single chemical variable (the primary reactant) was changed. The PCB recoveries from the actual tests were generally within a few percent of the control tests in these comparisons.

Reflux temperature nitric acid or water causes the volatilization of less chlorinated PCB congeners, to an extent, into the vapor phase. The fugacity (tendency to escape from solution) of these congeners from the solution is positively influenced by the acid strength of the solution; i.e., as acid concentrations increase, more PCBs are forced into the gas phase. With nitric acid media, there is an apparent route for the most volatile PCB congener tested (2,4 dichlorobiphenyl) to nitrate, even when 0.5 M nitric acid solution is employed. We hypothesize that gas phase nitration, utilizing NO_2 or NO vapors from the nitric acid, is the route for this reaction. This nitration reaction is relatively inefficient; however, substantial yields of nitrochlorobiphenyl and nitrodichlorobiphenyl species, presumably from 2,4 dichlorobiphenyl, were observed. The nitration effect is severely attenuated as PCB volatility or solubility is decreased, as only traces of nitrotetrachlorobiphenyl were noted in a few chromatograms. [Note: Aroclor 1254 contains essentially no dichlorobiphenyl; it is composed of 1% trichloro-, 15% tetrachloro-, 53% pentachloro-, 26% hexachloro-, and 4% heptachlorobiphenyl.]

The majority of PCB congeners that do not escape into the vapor phase remain associated with the solids in the reaction flask or with the surface of the reaction flask and associated components (condenser, thermometer), and are chemically unchanged.

Filtration (0.45-μm PVDF filter) removed 88% to 97% of the dissolved PCBs in spiked water. These results are consistent with similar filtration testing conducted as part of the K East Canister Sludge Characterization studies, in which a 0.45-μm glass fiber Tuffryn filter removed 94% of dissolved Aroclor 1254 in spiked water.

After neutralization and reprecipitation of the dissolver solution, 91% to 99% of the PCBs partitioned to the precipitate. These results indicate that the precipitation step is effective in partitioning PCBs associated with the dissolver solution into the solid stream. These results were obtained in tests conducted without filtration of the dissolver solution or the final supernatant.

In summary, PCB congeners were not significantly chemically altered or destroyed by hydrogen peroxide and/or nitric acid. However, this study further demonstrated the feasibility of using physical separation techniques to remove PCBs from streams generated from K Basin sludge processing. Most of the PCBs remain associated with the undissolved solids and surfaces on dissolver test system components. The small fraction of PCBs remaining in the dissolver solution is largely removed during filtration and by neutralization and reprecipitation (caustic adjustment).

Contents

Summary iii

1.0 Introduction 1

2.0 Description of the Experiments 3

2.1 Test System 3

2.2 PCB Spike Solution/Solid Substrate 3

2.3 PCB Extraction Approach 4

2.4 PCB Analysis via Gas Chromatograph (GC) with Electron Capture Detector (ECD) 4

2.5 Hydrogen Peroxide (Test 2) Description 5

2.6 Nitric Acid (Test 3) Description 6

3.0 Results 9

3.1 Results from Hydrogen Peroxide Tests (Test 2) 9

3.2 Results from Nitric Acid Tests (Test 3) 10

4.0 Conclusions 15

5.0 References 17

Appendix A Analytical Control Surrogate Recoveries (TCX and DCB) A.1

Appendix B Quantitative Results from Hydrogen Peroxide Addition Testing (Test 2 Experiments) B.1

Appendix C Quantitative Results from Nitric Acid Dissolution Testing (Test 3 Experiments) C.1

Tables

1 Conditions for Test 2 Experiments 6

2 Conditions for Test 3 Experiments 7

3 Mass Balances for Test 2 10

4 Mass Balances for Test 3 11

5 Recovery Results from Filtration of PCB-Spiked Water 13

1.0 Introduction

Two water-filled concrete pools (basins) in the 100K Area of the Hanford Site contain over 2100 metric tons of N Reactor fuel elements stored in aluminum or stainless canisters. During the time the fuel has been stored, approximately 52 m^3 of heterogeneous solid material have accumulated at the bottom of the K Basins. This sludge is a mixture of spent fuel element corrosion products, ion exchange materials (organic and inorganic), graphite-based gasket materials, iron and aluminum metal corrosion products, sand, and debris (Makenas et al. 1996, 1997). In addition, small amounts of polychlorinated biphenyls (PCBs) have been found, which are significant from a regulatory standpoint.

The predominant congener group of PCBs in the sludge has been identified as Aroclor 1254. The maximum concentration of PCBs in the sludge, which was found in the K East (KE) Weasel Pit, is 140 ppm (settled sludge basis) (Schmidt 1997). However, the distribution of the PCBs is non-uniform throughout the sludge (i.e., regions of high and low concentrations and places where no PCBs are present). Low concentrations of Aroclors 1016/1242, 1221, 1248, 1254, and 1260 have been identified and quantified in K West (KW) Canister sludge (Makenas et al. 1998). In some of these samples, the concentration of 1260 is higher than that of 1254.

Ultimately, it is planned to transfer the K Basin sludge to the Hanford double shell tanks. Before the sludge can be transferred, however, chemical pretreatment (i.e., K Basin Sludge Conditioning Process) is required to address criticality issues, and the destruction or removal of PCBs may be necessary.

Based on evaluations, engineering studies, and limited testing, Fluor Daniel Hanford recommended nitric acid dissolution of the K Basin sludge and, potentially, peroxide addition (FDH 1997). The peroxide addition, along with physical separations within the process, would be used to facilitate PCB removal and destruction.

The purpose of the work described in this report was to determine the fate of PCBs in the K Basin Sludge Conditioning Process. Specifically, PCB destruction/removal efficiency of hydrogen peroxide addition and nitric acid dissolution was examined. In one series of experiments (Test 2), the effects of adding hydrogen peroxide to a PCB-containing slurry that simulated acid-dissolved K Basin sludge were investigated. In the second series of experiments (Test 3), a PCB-spiked K Basin sludge simulant was acid dissolved, and the resulting dissolver solution was neutralized and precipitated (caustic adjustment). The resulting liquid, solid, and gas streams from both series of tests were extracted and analyzed to determine the fate of the PCBs.

The experimental approach, test apparatus, and analytical techniques are described in Section 2.0. The results are presented in Section 3.0, and Section 4.0 summarizes the conclusions. A follow-on study to this work, which focused on the partitioning of PCBs during neutralization and precipitation (caustic addition), was recently completed and is reported separately (Schmidt et al. 1998).

THIS PAGE INTENTIONALLY
LEFT BLANK

2.0 Description of the Experiments

2.1 Test System

The apparatus used for the tests was a closed system consisting of a 100-ml, three-neck Morton flask, equipped with a magnetic stir bar, thermometer, jacketed condenser, and addition funnel. The glass joints were assembled wetted (with water) to limit escape of vapors from the joints, and the condenser was attached to a recirculating 4°C water supply to fully condense refluxing materials. The top of the condenser was equipped with glass fiber plugged traps; each containing two separated, 2-cm plugs of fresh coconut charcoal obtained from Orbo-32 (Supelco) tubes.

2.2 PCB Spike Solution/Solid Substrate

A PCB-congener spiking solution for the tests was prepared by adding 22.89 mg 2,4 dichlorobiphenyl; 52.46 mg 2,2', 4,4' tetrachlorobiphenyl; 52.94 mg 2,2',4,4',6,6' hexachlorobiphenyl; and 3.87 mg 2,2',3,3',4,4',5,5' octachlorobiphenyl into a 25-ml class A volumetric flask and brought to volume with hexane. This resulted in a mixture that was 916 μg/ml, 2098 μg/ml, 2118 μg/ml, and 155 μg/ml for each of the respective congeners. The total PCB congener load for 1 ml of the spike solution was 5287 μg. This mixture of PCB congeners represented PCBs that bracket the chlorine content of Aroclor mixtures (e.g., Aroclor 1254, 1242), without the need to analyze the entire Aroclor PCB pattern in the data. The exceptionally clean chromatographic patterns provided simple quantification and ready identification of any major extra components appearing in the chromatograms. The relative amounts per congener chosen were meant to create a mix in which all analyses would fall within the dynamic range of the detector for each run, so that quantification could be done on a single dilution of each data point. The possibility of oxidation reactions does increase for less chlorinated PCB congeners; thus, 2,4 dichlorobiphenyl was chosen to represent the least chlorinated species, though these congeners are an insignificant (<1%) component in actual PCB mixtures that have been found in the K Basin sludge.

Silica/Zeolon 900H mixtures (2:1 weight ratio), less than 20-μm average particle size, were prepared to simulate the insoluble matrix purported to be present in K Basin sludge. The fine particle size chosen provides a surface area comparable to that expected to be found in the K Basin materials. Spikes of the master PCB spiking solution onto a 2:1 silica/Zeolon 900H mix yielded an average recovery of 87% dichlorobiphenyl, 85% tetrachlorobiphenyl, 86% hexachlorobiphenyl, and 96% octachlorobiphenyl. These data support the extractability of the desired analytes from the support media. Spiking the tests by amending the hexane master solution of PCBs onto a bed of silica/Zeolon directly into the reaction flask halted the transfer loss of PCBs during the experiments.

2.3 PCB Extraction Approach

The materials captured in each test were intended to be for total assay of PCB congener content in each fraction. To this end, the whole liquid/solid digest was collected along with a 3-ml rinse of water that was combined with the digested material. The reaction flask was rinsed first with acetone, then with hexane to afford 8 ml of mixed washes to determine how much PCB had dispersed onto the flask walls. Thermometer and addition funnel components were extracted with a similar wash and collected as a combined 10-ml wash. A 5-ml wash of the condenser with acetone and hexane was collected (along with a rinse of the glass joint). The granular activated carbon (GAC) traps consisted of a glass jointed tube connected to the condenser. The plugs (glass fiber) and two separate carbon traps were collected as completely separate portions without washing the glass tube used for collection. After Test 2 was conducted, the amount of dispersion of PCBs onto the thermometer and the addition funnel, and through the condenser, was found to be small in comparison to the PCB concentration associated with the undissolved solids; therefore, the extracts of the equipment were combined into one assay for experiments in Test 3.

Tetrachloroxylene and decachlorobiphenyl were added as surrogate compounds to all solid and supernatant samples just prior to the extraction process. Recovery of these compounds was used as a measure of efficiency of the extraction process. The surrogate recoveries represent the optimum recovery possible without the chemical treatment test variables included. The volatility of the surrogate compounds bracket the range represented by the PCBs chosen for these tests and for almost all of the possible PCB congeners. Recoveries ranging from ~90% to 100% spiked at 200 µg/sample were observed for the solid materials and ~60% to 100% spiked at 0.02 µg/sample for the liquid samples (see Appendix A).

Following the addition of the surrogate compounds and sodium sulfate drying agent, the solid samples were extracted three times using high-intensity sonication and acetone/hexane (1:1). Liquid samples were processed using liquid-liquid extractions in separatory funnel with methylene chloride. Both liquid and solid sample extracts were then concentrated and exchanged into hexane.

2.4 PCB Analysis via Gas Chromatograph with Electron Capture Detector

After sample extraction and preparation, analysis was performed using a gas chromatograph (GC) with two electron capture detectors (ECD). The GC response factors for the ECDs were obtained on dilution of the PCB master solution. ECD allows detectability limits below the ng/ml range. GC conditions were a single 2-µl injection made on-column onto a 5 M fused silica retention gap split between two analytical columns: a 0.32 mm x 30 mm DB-17 (0.25-µm phase) and a 0.32 mm x 30 mm DB-1701 (0.25-µm phase). The oven-programmed temperature was 68°C for 0.5 min, then 20°C/min to 200°C, 3°C /min to 250°C, and 20°C/min to 280°C; held at 280°C for 15 min. The ECD temperature was 300°C with a purified nitrogen make-up gas.

2.5 Hydrogen Peroxide (Test 2) Description

Test 2 experiments are described in detail in Test Instruction TI-28510-2 (Silvers 1998a). Test 2 probed the effect of adding a 50-fold stoichiometric excess (versus PCB congener mixture) of 30% hydrogen peroxide to a mix of PCB congeners on a silica/Zeolon 900H support in 0.5 M nitric acid/1 M ferric nitrate solution (i.e., simulant of acid-dissolved K Basin sludge). Hydrogen peroxide was added dropwise when the reaction solution was at 60°C. A control experiment was also conducted using water in place of hydrogen peroxide to show how the mass balance of PCB congeners compared to an identical system set-up, without the hydrogen peroxide reactant. The experiment and control were done in duplicate, and were kept at 60°C for 1 hour after addition was complete. If the recovered mass of each congener was significantly different in the hydrogen peroxide test versus the control, the effectiveness of hydrogen peroxide in interacting with the PCBs would have been apparent. Inadvertent loss of the PCBs through fugacity from the media, into the vapor phase and onto the components of the reaction system, has to be considered in this type of test. Therefore, some unaccountable loss of PCB to the reaction vessel or to the charcoal traps was anticipated.

Test 2 was conducted by evenly amending a 2.5-g amount of silica/Zeolon (2:1 by weight) with 500 µl of the PCB master spiking solution so the total amount of added PCBs was 2642 µg. This created a solid matrix that was nominally 1000 ppm PCB (with respect to the solids) upon the start of the experiment. The rest of the solution was made up of a matrix containing 1 M ferric nitrate solution in 0.5 M nitric acid. The mixture was brought to 60°C temperature and 30% hydrogen peroxide (10 ml) added cautiously, dropwise, to the mix.

The expected oxidative reactions that might be envisioned in the mix could entail superoxide anions and hydroxyl radicals via the Fenton reaction; these reactions are thought to be optimized at around pH 1-4 with concentrations of ferric/ferrous ions similar to that used in this experiment. The Fenton reactions are specifically retarded in either strong acid (due to air oxidation of ferrous ion) or alkaline media (due to iron hydroxide precipitation). The hydroxyl radical is among the most potent oxidative chemical species available. The lifetime of hydroxyl radicals is exceedingly short (Haag and Yao 1992): the actual test needed only short reflux after the last addition of hydrogen peroxide to ensure the hydroxyl radicals potential participation in any reaction. The observed course from the addition of hydrogen peroxide indicated complex competing reaction chemistries were occurring, presumably involving the reduction of ferric species to ferrous species coupled with Fenton-like conditions (Laitinen and Harris 1975). The addition of hydrogen peroxide was accompanied with much evolution of gas, but, as the reaction proceeded, the medium changed from brown to violet. At this stage, the relative evolution of gas from hydrogen peroxide addition became extremely vigorous.

The evidence for competing kinetics is that this vigorous reaction of hydrogen peroxide was only present so long as the violet color persisted. If the addition of hydrogen peroxide was stopped, the violet color disappeared, and H_2O_2 had to be added to regenerate conditions where vigorous reaction was recurring.

Addition of H_2O_2 was conducted slowly so that the more vigorous evolution of gas could be controlled. The control experiment, used for PCB recovery comparison, was conducted identically, except a volume of water (10 ml) was used in place of hydrogen peroxide.

The individual runs and the actual experiments for Test 2 were conducted as shown in Table 1. All experiments were conducted in duplicate.

Table 1. Conditions for Test 2 Experiments

Experiment No.	Reactant	Conditions	PCB Concentration
3, 4	blank	$Fe(NO_3)_3$, HNO_3, 60°C	blank
5, 6	H_2O_2	$Fe(NO_3)_3$, HNO_3, 60°C	1000 ppm
7, 8	none	$Fe(NO_3)_3$, HNO_3, 60°C	1000 ppm

2.6 Nitric Acid (Test 3) Description

Test 3 experiments are described in detail in Test Instruction TI-28510-3 (Silvers 1998b). PCBs were amended to silica/Zeolon 900H media, and a sludge simulant (meant to mimic KE Basin Floor Weasel Pit Sludge) consisting primarily of iron oxides and aluminum oxide was added. In these experiments, nitric acid (16 M) was added so that the resulting solution was about 10 M nitric acid, and the mixture was refluxed for 6 hours. After the acid digestion, the insoluble solids were separated from the acid digest solution (dissolver solution). The dissolver solution was spilt into two samples of near equal volume. One spilt was filtered (0.45-µm PVDF filter) to remove any entrained particulates that could carry PCB. The other dissolver solution split remained unfiltered. Next, to mimic the K Basin Sludge Conditioning Process, both splits were neutralized and precipitated by adjusting the pHs of the dissolver solution splits to greater than 12 (via NaOH addition). The splits were then centrifuged to provide clarified supernatant. The supernatant from the split previously subjected to filtration was filtered again (0.45-µm PVDF filter) to remove any entrained particulates. The supernatant from the other neutralized split again remained unfiltered.

Test 3 was conducted by evenly amending 1.25-g amounts of silica/Zeolon (2:1 by weight) with 1000 µl of the PCB mix (above) so the total amount of added PCBs was 5284 µg. This mix was allowed to equilibrate and outgas solvents before the sludge simulant was added. A 12.5-g mix of KE Basin Floor and Pit Sludge simulant was then added to the mix. The mix contained about 4.5 g of total solids; it was feasible to calculate the total PCB concentration in the solids (sludge and silica) as about 1170 ppm at the start of the experiment. The composition of this simulated sludge was as follows:

Fe_2O_3	117.8 g/L
FeO(OH)	131.4 g/L
$Al(OH)_3$	74.4 g/L
CaO	3.84 g/L
MgO	0.94 g/L
MnO_2	0.51 g/L
ZnO	0.71 g/L
Cr_2O_3	0.76 g/L
$CuSO_2 \cdot 5H_2O$	0.89 g/L
$CaCO_3$	9.5 g/L
NaOH	61 mmol
KOH	42 mmol
HNO_3	240 mmol

This composition results in a simulant medium that is approximately 1 L in volume and weighs 1260 g. This medium represented the inorganic media expected in the sludge, minus the uranium/lanthanide components and without the presence of organic resins, some of which (i.e., Duolite 359) have been shown, during PNNL studies, to irreversibly bind chlorinated organics.

The individual runs and the actual tests were conducted as shown in Table 2. All experiments were conducted in duplicate.

Table 2. Conditions for Test 3 Experiments

Experiment No.	Reactant	Conditions	PCB Concentration
33-1, 33-2	blank	equipment wash	blank
34, 35	boiling water	iron oxide, reflux	1170 ppm
36, 37	10 M HNO_3	iron oxide, reflux	1170 ppm

Test 3 included a control (Experiment No. 34 and 35) using the same concentration of PCBs in silica/Zeolon/iron oxide media, but with water as the liquid instead of 10 M HNO_3. The data obtained from the boiling water reaction were compared with the nitric acid media to ascertain the difference in PCB recovery between high nitric acid media and water in the same apparatus with substantially the same matrix conditions.

THIS PAGE INTENTIONALLY
LEFT BLANK

3.0 Results

The tables provided in Appendices B and C contain the analytical data from Tests 2 and 3, respectively. In both Tests 2 and 3, the data should be interpreted in conjunction with the control experiments associated with each experiment. It was found that the less volatile congeners have excellent total mass recovery from the reaction media; 70-80% of the hexachlorobiphenyl was recovered, while 90-100% of the octachlorobiphenyl was recovered in both tests. Comparing the recovery from control experiments demonstrates the extent of reaction (or lack of reaction) due to the chemical variable, hydrogen peroxide or nitric acid.

Most of the data are formatted (in Appendices B and C) to show the recovery of PCB congeners within the experimental apparatus. The recovery data are lower for less-chlorinated congeners and appear to be strongly influenced by the temperature regime experienced in each experiment; thus, a significant fraction of the less chlorinated PCB congeners appears to have been purged from the experimental apparatus due to volatilization. Control experiments conducted at substantially the same temperature as the test objective provide a baseline comparison for these observations.

3.1 Results from Hydrogen Peroxide Tests (Test 2)

Within Test 2 (summarized in Appendix B, per congener assayed in Test 2), the total amount of each PCB congener found in Experiments 5 and 6 (with peroxide) is similar to the amount found in Experiments 7 and 8 (control with no peroxide). That is, the addition of hydrogen peroxide does not appear to influence the net amount of PCBs in the system.

During the addition of hydrogen peroxide to the reaction mixture, an induction reaction was observed that consisted of general evolution of oxygen bubbles along with a color change from brown to violet in the solution. A vigorous evolution of oxygen and a slight elevation in temperature were also seen. While the reaction was conducted to maintain a slight purplish color to the solution during the addition, the vigorous purging of oxygen was not considered beneficial to solution reaction of PCBs. Consequently, this type of reaction was minimized by slow addition of hydrogen peroxide.

Since there was a net exhaust of gas from the reaction mixture, there was obvious aerosol created by the flask contents. The analytical result indicates significant dichlorobiphenyl, tetrachlorobiphenyl, and hexachlorobiphenyl have been carried onto the glass wool plugs and GAC beds. Spiking experiments determined that only 1% of dichlorobiphenyl could be directly extracted from GAC; much better results were found for tetrachloro (23% recovery by extraction) and hexachloro (62% recovery). Octachlorobiphenyl also appears to suffer from a low recovery from this medium (6%). Results for GAC recovery in the tables (Appendices B and C) have been normalized by these recovery factors. Direct extraction by solvents is apparently insufficient in partitioning PCBs from the carbon surface.

Better PCB congener recoveries from GAC may have been possible if exhaustive soxhlet extraction had been used; however, a significantly greater level of effort would be required to employ this technique. We used direct solvent extraction with ultrasonic agitation as a convenient means to determine whether the GAC was capturing any PCB in the offgas. The appearance of 2,4 dichorobiphenyl in the GAC and in the end plugs of the GAC beds strongly indicates that the GAC is being exhausted by the presence of moisture or acid fumes, or that the more volatile PCB components are not strongly retained by the GAC configuration used. Furthermore, it is speculated that the PCBs unaccounted for in other portions of the experiment have most likely been lost to volatilization during the test or during analytical steps.

The qualities of PCB congeners recovered from the experimental components were not significantly affected by the addition of peroxide versus the control test where water was added. The generally lower recovery of PCBs from the peroxide test was countered by the much larger appearance of PCB components in the offgas, captured on the GAC. The best interpretation is that volatilization/aerosol production occurred in the addition of peroxide, which allowed the more volatile PCBs to leave the reaction flask. Of the PCBs recovered, the vast majority of each congener was concentrated in the reaction glassware or the solids in the flask where they were originally deposited.

Mass balances for the Test 2 experiments are provided in Table 3.

Table 3. Mass Balances for Test 2

Test ID	Undissolved Solids (g)	Unfiltered Supernatant (ml)
5	2.53	20
6	2.63	20
7	2.77	20
8	2.80	20

3.2 Results from Nitric Acid Tests (Test 3)

Test 3 probed the dispersion of PCBs in the glass reactor system in boiling nitric acid media versus the control experiment in boiling water media. In Experiments 36 and 37 a 13-ml portion of 16 M nitric acid was added to the spiked solids/12.5-g iron oxide sludge simulant. This gave an initial concentration of about 10 M nitric acid. After reflux for 6 hours, the mix was cooled and the solutions/solids and rinses were collected in the same fashion as in Test 2 activities. In this case, we collected the supernatant liquids (18 ml prior to a 5-ml water rinse of the flask), and this solution was divided in half. Half of this liquid was filtered through a 0.45-µm PVDF filter (Acrodisc), and a 5-ml gastight syringe was used to immediately filter the liquid (12-13 ml at this point). The filtered and unfiltered supernatants were treated with 4 ml of 10 M NaOH, and the pH checked with indicator paper to ensure that the precipitate and supernatant were greater than pH 12. The vials and syringe were also rinsed, contributing an additional 3-5 ml of volume to the total. The precipitated solids from this process were very voluminous (15-20 ml volume) at this point, and obviously contained much occluded water. Drying and analytical extraction of

these solids was very difficult due to the gelatinous nature of the precipitate. Mass balances for the Test 3 experiments are given in Table 4.

Table 4. Mass Balances for Test 3

Test ID	Undissolved Solids (g)	Unfiltered Supernatant (ml)	Filtered Supernatant (ml)	Unfiltered Neutralized		Filtered Neutralized	
				Sludge Volume (ml)	Supernatant Volume (ml)	Sludge Volume (ml)	Supernatant Volume (ml)
34	3.79	9	9	--	--	--	--
35	4.04	9	9	--	--	--	--
36	3.89	9	9	16	5	16	5
37	3.95	9	9	13	8	13	8

When analytical tests were complete, a weight was obtained on the washed solids remaining in the reactor digest. In both Experiments 36 and 37, a weight of 3.8 g was obtained. The sludge from Experiments 34 and 35 was washed and found to contain 4.6 g of solids. This indicated that only about 1 g of solids dissolved in 10 M nitric acid, or that (since 1.25 g of insoluble silica is present in the solids) only 22% of the solids dissolved in the processing of Experiments 36 and 37. The boiling water experiments (34 and 35) were treated in a similar manner: dividing the liquids and neutralization. No precipitates formed upon addition of NaOH in these two experiments.

PCB analysis of Test 3 fractions mimicked the results found in Test 2 to a large extent. Of the recovered PCB totals (per congener), 90% to 100% of each of the recovered PCB load was associated with the undissolved solids and the glassware. Overall material balances for each congener were 100% octachlorobiphenyl recovered; 85% hexachlorobiphenyl recovered; 75% tetrachlorobiphenyl recovered; and between 10% and 47% dichlorobiphenyl recovered. The lower recovery for the more volatile components is indicative of loss through outgassing of the volatile component; particularly in light of the demonstrated inefficiency of the GAC entrapment scheme and the lack of any observable tri-, penta-, or heptachlorinated intermediate species, which would be obvious dechlorination products from the PCBs used.

The appearance of nitrochlorobiphenyl and nitrodichlorobiphenyl in the undissolved solids and liquids in Experiment 36 (i.e., nitric acid test) indicates that some of the 2,4 dichlorobiphenyl is participating in undefined reactions with nitric acid or NO_2/NO components in the system. The trace amounts of (apparently) nitrotetrachlorobiphenyl found in Experiments 36 and 37 is a strong indication that the mechanism must involve gas phase reactions between the NO_2/NO in the system and volatilized PCBs, as tetrachlorobiphenyl has a vapor pressure approximately 20 times less than 2,4 dichlorobiphenyl (Erickson 1992). There is also the possibility that higher chlorinated congeners are less kinetically able to enter into the nitration reaction because of the extra chlorine's inductive draw on the biphenyl ring. In any event, there does appear to be an oxidation mechanism for 2,4-dichlorobiphenyl in refluxing nitric acid medium, which leads to both dechlorination/nitration and simple nitration of the ring. The nitration was evidenced even in Test 2, where the nitric acid concentration was less than 0.5 M. Since aromatic nitration reactions

in solution require concentrated nitric acid and dehydrating conditions (e.g., sulfuric acid), it is unlikely that these observed products have an origin in the liquid phase, supporting the hypothesis that these materials arise from gas phase reactions.

In acid media, there is a marked dispersion of the PCBs from the original solids onto the surfaces of the reaction flask/condenser. Indeed (see the spreadsheets in Appendix C), the ratio of distribution of tetrachloro- and hexachlorobiphenyl to the container in boiling water (approximately 1:2) is approximately reversed when the medium is changed to boiling 10 M nitric acid (hexachlorobiphenyl averages 2:1 container: solids ratio; tetrachlorobiphenyl is about 4:1 container: solids ratio). Dichlorobiphenyl is subject to volatilization, and markedly less is recovered, especially from acidic media. Octachlorobiphenyl is not as soluble as the other congeners, but still demonstrates some of this effect.

Since the bulk of PCB Aroclor mixtures is concentrated in the tetrachloro to heptachloro range, Test 3 gives convincing evidence that the primary materials (as Aroclor 1254), which will be present in the K Basin sludge, will be dispersed in the solids and on the reaction vessel, with the lesser concentration in the liquid phase being controlled by the solubility limit of each PCB congener in the media. Comparison of the recovery of tetrachoro-, hexachloro-, and octachlorobiphenyls between the boiling water tests and the boiling acid tests does not support any important chemical reactions of PCBs in acid media.

Filtering the liquid supernatant in Experiments 34 through 37 showed that up to 85-98% of the PCB congeners in the liquid phase are removed by forcing the liquid through a 0.45-μm PVDF membrane. A control experiment was conducted to determine whether the filter removed PCB-contaminated particles or whether the filter medium itself removed PCB components dissolved in the liquid. PCBs are well known to have affinity for glass and (especially) organic surfaces.

A level of PCB spike was constructed to mimic the results found on the filtered liquids in Experiments 34 through 37 with the PCBs originally introduced into a initial water solution. Two results were immediately obvious: 88-97% of the dissolved PCBs were removed from the solution by the filter, and only 40-70% of the PCBs introduced into the solution were found upon analysis of the unfiltered water solution (Table 5). The easiest explanation of this latter phenomenon is that the container walls contribute to binding some of the PCB components during the short contact time between spiking the water and extraction of the solution. It is important to note that the initial containers were NOT rinsed with solvents prior to analysis for just this purpose. The direct analysis of the liquid provides a measure of how much PCB is lost to the container, the syringe, and other surfaces before it reaches the filter medium. The single 0.45-μm PVDF filter effectively removes dissolved PCB components, and may provide the best means of relieving a dissolver solution of entrained PCB materials.

After neutralization and reprecipitation of the unfiltered dissolver solution, 91% to 99% of the PCBs partitioned to the precipitate. These results indicate that the precipitation step is effective in partitioning PCBs associated with the dissolver solution into the solid (sludge) stream.

Table 5. Recovery Results from Filtration of PCB-Spiked Water

PCB Congener	Filter Removal Efficiency (%, N=2)	Spiking Concentration (ng/ml)
2,4 Dichlorobiphenyl	88.58	1.83
2,2',4,4' Tetrachlorobiphenyl	96.68	4.20
2,2',4,4',6,6' Hexachlorobiphenyl	96.50	4.23
2,2',3,3',4,4',5,5' Octachlorobiphenyl	94.81	0.31
Analyses were performed on 10-ml aqueous solutions spiked with PCB congeners before and after filtration using a 0.45-μm PVDF cartridge connected to a 10-ml gas-tight syringe.		

For the filtered dissolver solution, 59% to 96% of the recovered PCBs partitioned to the precipitate after neutralization and reprecipitation.

The total quantity of PCBs recovered in the precipitated solids in which the dissolver stream was not filtered, was six to nine times greater than in the precipitated solids generated after filtration of the dissolver solution. This result provides additional evidence that significant quantities of PCBs can be removed from the dissolver solution by filtration.

THIS PAGE INTENTIONALLY
LEFT BLANK

4.0 Conclusions

Hydrogen peroxide and nitric acid served to disperse PCBs within the reaction vessel in our tests; however, the majority of the highly insoluble tetrachlorobiphenyl, hexachlorobiphenyl, and octachlorobiphenyl remained bound to the respective original solid surfaces. Careful control experiments used as comparison/normalization tests were very helpful in understanding the inevitable loss of trace components from the system. The more volatile components (such as 2,4 dichlorobiphenyl) entrained in the vapor phase may have either strongly bound to our capturing GAC, may have offgassed through the GAC already rendered partially ineffective by exposure to moisture or NO_2 vapors, or may have participated to a degree in nitration reactions.

The tests conducted here provided a basis for understanding the potential extent of dispersal of the PCBs through the system. The higher chlorinated congener PCBs showed a marked preference to remain on solid surfaces within the reaction vessel at reflux conditions. The amount of PCBs appearing in the liquid phase was governed primarily by their solubility in that medium, and they appeared to be stripped from the aqueous media by organic-based filter membranes. Losses of PCBs in solution can also be attributed to attraction to the (relatively) non-polar surfaces of the walls of vessels that transport the fluid. A series of filter surfaces may serve to polish virtually all PCBs from solution phase. The Aroclor 1254 formulation, with the majority of congeners in the tetrachloro- to heptachlorobiphenyl range, should behave in similar fashion to the PCBs chosen for this model study. Volatilization of PCBs appears to attenuate at about the tetrachorobiphenyl substitution range. Most of the PCB components will be governed by their solubility restrictions and attractions to solid surfaces, such as the reaction vessel walls and acid insoluble solids, and the precipitate that forms upon neutralization of the dissolver solution.

THIS PAGE INTENTIONALLY
LEFT BLANK

5.0 References

Erickson, M. D. 1992. *Analytical Chemistry of PCBs*, Lewis Publishers, Ann Arbor, Michigan.

Fluor Daniel Hanford, Inc. (FDH), 1997. "Recommended Strategy for Disposal of K Basin Sludge." Letter Report to J.D. Wagoner, U.S. Department of Energy, June 30, 1997. FDH-9755700, Richland, Washington.

Haag, W. R. and C.C.D. Yao. 1992. "Rate Constants for Reaction of Hydroxyl Radicals with Several Drinking Water Contaminants." *Environ. Sci. Technol.* 26(5):1005-1013.

Laitinen, H. A. and W. E. Harris. 1975. *Chemical Analysis, an Advanced Text and Reference,* 2nd edition, pp. 298-303. McGraw-Hill, New York.

Makenas, B. J., T. L. Welsh, R. B. Baker, D. R. Hansen, and G. R. Golcar. 1996. "Analysis of Sludge from Hanford K East Basin Floor and Weasel Pit." WHC-SP-1182, Westinghouse Hanford Company, Richland, Washington.

Makenas, B. J., T. L. Welsh, R. B. Baker, E. W. Hoppe, A. J. Schmidt, J. Abrefah, J. M. Tingey, P. R. Bredt, and G. R. Golcar. 1997. "Analysis of Sludge from Hanford K East Basin Canisters." HNF-SP-1201, DE&S Hanford, Inc., Richland, Washington.

Schmidt, A. J., G. M. Mong, E. W. Hoppe, S. O. Slate, K. L. Silvers, and B. M. Thornton, 1998. "Partitioning of PCBs in Dissolver Solution After Neutralization/Precipitation (Caustic Adjustment)." PNNL-11997, Pacific Northwest National Laboratory, Richland, Washington.

Schmidt, A. J. 1997. "Evaluation of Polychlorinated Biphenyls (PCB) Data from K East Basin Sludge Samples." Letter to R. P. Omberg (DESH) on January 7, 1997, and appendix to HNF-2367, DE&S Hanford, Richland, Washington.

Silvers, K. L. 1998a. Letter to F. W. Moore (DESH) from K. L. Silvers (PNNL) dated March 16, 1998. Letter Number 28510-09. Transmittal of Test Instruction TI-28510-2 Rev. 0. "Test #2 Hydrogen Peroxide Treatment of Simulated Dissolver Stream."

Silvers, K. L. 1998b. Letter to F. W. Moore (DESH) from K.L. Silvers (PNNL) dated April 27, 1998. Letter Number 28510-11. Transmittal of Test Instructions TI-28510-3 Rev. 0. "Test #3, Fate of PCBs During Nitric Acid Digestion/Dissolution Without Peroxide."

THIS PAGE INTENTIONALLY
LEFT BLANK

Appendix A

Analytical Control Surrogate Recoveries (TCX and DCB)

THIS PAGE INTENTIONALLY
LEFT BLANK

TEST 2 **Tetrachloroxylene** 200 ug added to solids

0.02 ug added to liquids

Test	Liquids ug found	% recovered
Blank	0.014	68.300
1	na	na
2	na	na
3	0.012	61.900
4	0.019	93.200
5	0.019	95.100
6	0.013	67.300
7	0.015	73.700
8	0.016	80.300

Solids ug found	Solids Back Ext	Total ug found	% recovered
198.060		198.060	99.03
207.680		207.680	103.84
191.280		191.280	95.64
195.390		195.390	97.70
188.660		188.660	94.33
199.790	0.103	199.893	99.95
198.910	0.142	199.052	99.53
201.480	0.062	201.542	100.77
195.680	0.033	195.713	97.86

TEST 2 **Decachlorobiphenyl** 200 ug added to solids

0.02 ug added to liquids

Test	Liquids ug found	% recovered
Blank	0.012	61.600
1	na	na
2	na	na
3	0.014	69.200
4	0.016	79.700
5	0.018	88.200
6	0.016	81.600
7	0.017	86.000
8	0.021	107.300

Solids ug found	Solids Back Ext	Total ug found	% recovered
186.410		186.410	93.21
194.190		194.190	97.10
181.070		181.070	90.54
180.700		180.700	90.35
170.330	0.398	170.728	85.36
185.910	0.157	186.067	93.03
187.400	0.196	187.596	93.80
191.190	0.078	191.268	95.63
183.590	0.095	183.685	91.84

THIS PAGE INTENTIONALLY
LEFT BLANK

Appendix B

Quantitative Results from Hydrogen Peroxide Addition Testing (Test 2 Experiments)

THIS PAGE INTENTIONALLY
LEFT BLANK

TEST 2

2,4 Dichlorobiphenyl **457.8 ug added**

Test ID	Liquids (ug)	Flask/Thermometer (ug)	Condenser (ug)	Container (ug)	Solids (ug)	Solids Back Ext (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
Blank	0.000	na	na	na	0.000	na	na	0.000	na
1	0.000	na	na	na	417.131	na	na	417.131	91.12
2	0.000	na	na	na	376.800	na	na	376.800	82.31
3	0.005	0.021	0.004	0.004	1.954	----	----	1.988	na
4	0.000	0.000	0.000	0.080	0.991	0.000	----	1.071	na
5	1.343	4.788	36.852	11.617	4.651	0.000	13.790	73.040	15.95
6	1.224	5.422	8.488	8.883	2.898	0.000	23.810	50.726	11.08
7	1.247	1.884	0.305	47.468	2.671	0.644	1.164	55.383	12.10
8	3.143	3.554	4.977	31.689	96.111	0.485	1.428	141.386	30.88

TEST 2

2, 2', 4, 4' Tetrachlorobiphenyl **1049.2 ug added**

Test ID	Liquids (ug)	Flask/Thermometer (ug)	Condenser (ug)	Container (ug)	Solids (ug)	Solids Back Ext (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
Blank	0.000	na	na	na	0.887	na	na	0.887	na
1	0.000	na	na	na	938.517	na	na	938.517	89.45
2	0.000	na	na	na	852.902	na	na	852.902	81.29
3	0.010	0.016	0.008	0.001	2.837	----	----	2.872	na
4	0.002	0.002	0.005	0.001	0.689	0.242	----	0.941	na
5	3.940	23.907	43.877	109.094	227.267	0.222	14.957	423.264	40.34
6	3.271	38.507	26.549	78.573	194.479	0.192	23.743	365.313	34.82
7	1.986	7.024	1.101	133.784	474.187	0.332	2.428	620.841	59.17
8	5.735	21.855	7.144	81.906	527.282	0.578	0.792	645.293	61.50

TEST 2

2, 2', 4, 4', 6, 6' Hexachlorobiphenyl **1058.8 ug added**

Test ID	Liquids (ug)	Flask/Thermometer (ug)	Condenser (ug)	Container (ug)	Solids (ug)	Solids Back Ext (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
Blank	0.000	na	na	na	0.940	na	na	0.940	na
1	0.000	na	na	na	961.189	na	na	961.189	90.78
2	0.000	na	na	na	870.393	na	na	870.393	82.21
3	0.012	0.017	0.010	0.001	3.313	----	----	3.353	na
4	0.001	0.004	0.008	0.003	0.842	0.171	----	1.029	na
5	4.140	22.994	31.857	136.220	539.944	0.533	11.214	746.902	70.54
6	3.197	36.362	18.097	95.777	573.576	0.454	17.463	744.926	70.36
7	1.814	6.803	0.951	145.437	653.969	0.615	2.478	812.067	76.70
8	5.458	19.889	5.555	85.146	657.778	0.563	0.541	774.930	73.19

TEST 2

2, 2', 3, 3', 4, 4', 5, 5' Octachlorobiphenyl **77.4 ug added**

Test ID	Liquids (ug)	Flask/Thermometer (ug)	Condenser (ug)	Container (ug)	Solids (ug)	Solids Back Ext (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
Blank	0.000	na	na	na	0.381	na	na	0.381	na
1	0.000	na	na	na	78.032	na	na	78.032	100.82
2	0.000	na	na	na	71.653	na	na	71.653	92.57
3	0.001	0.001	0.003	0.000	0.311	----	----	0.317	na
4	0.000	0.001	0.003	0.001	0.141	0.000	----	0.145	na
5	0.432	1.030	0.582	11.004	55.101	0.063	0.354	68.566	88.59
6	0.370	1.544	0.511	8.361	59.795	0.080	1.087	71.748	92.70
7	0.166	0.487	0.052	11.701	61.238	0.055	1.003	74.703	96.52
8	0.555	1.847	0.382	5.626	64.349	0.075	0.213	73.047	94.38

TEST 2 GAC (Granular Activated Charcoal) Detail

2,4 Dichlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
5	5690.00	7588.65	279.50	212.77	18.60
6	5350.00	17446.81	187.80	737.59	88.20
7	68.00	921.99	35.20	134.75	4.50
8	10.00	638.30	67.00	709.22	3.20

TEST 2 GAC (Granular Activated Charcoal) Detail

2, 2', 4, 4' Tetrachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
5	9897.00	3327.51	1377.00	250.66	104.60
6	14180.00	7379.91	900.90	798.69	483.10
7	570.00	1751.09	76.30	22.27	7.90
8	43.00	506.55	154.20	82.53	6.20

TEST 2 GAC (Granular Activated Charcoal) Detail

2, 2', 4, 4', 6, 6' Hexachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
5	7745.00	2056.45	1054.00	246.45	111.90
6	11280.00	4458.06	669.10	661.77	393.60
7	978.00	1414.52	66.60	12.10	6.40
8	50.00	356.45	103.30	26.29	5.10

TEST 2 GAC (Granular Activated Charcoal) Detail

2, 2', 3, 3', 4, 4', 5, 5' Octachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
5	104.00	238.10	1.70	9.52	0.60
6	215.00	857.14	4.90	6.35	3.30
7	63.00	936.51	0.50	3.17	0.30
8	0.00	206.35	1.50	4.76	0.30

THIS PAGE INTENTIONALLY
LEFT BLANK

Appendix C

Quantitative Results from Nitric Acid Dissolution Testing (Test 3 Experiment)

THIS PAGE INTENTIONALLY
LEFT BLANK

TEST 3

2,4 Dichlorobiphenyl **915.6 ug added**

Test ID	Liquids (ug)	Container Rinses (ug)	Solids (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
34	0.546	279.600	132.860	2.325	415.331	45.36
35	0.484	305.800	123.700	1.622	431.606	47.14
36	0.102	257.000	2.960	0.597	260.659	28.47
37	0.356	91.400	3.370	0.929	96.055	10.49

TEST 3

2, 2', 4, 4' Tetrachlorobiphenyl **2098.4 ug added**

Test ID	Liquids (ug)	Container Rinses (ug)	Solids (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
34	0.699	642.700	905.190	0.347	1548.937	73.82
35	0.844	699.230	928.420	0.133	1628.627	77.61
36	0.201	1224.000	280.130	0.120	1504.451	71.70
37	0.780	1311.000	299.810	0.208	1611.799	76.81

TEST 3

2, 2', 4, 4', 6, 6' Hexachlorobiphenyl **2117.6 ug added**

Test ID	Liquids (ug)	Container Rinses (ug)	Solids (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
34	0.503	547.400	1156.440	0.213	1704.557	80.49
35	0.620	545.200	1293.600	0.134	1839.554	86.87
36	0.178	1016.000	786.120	0.071	1802.369	85.11
37	1.081	1326.000	481.260	0.100	1808.441	85.40

TEST 3

2, 2', 3, 3', 4, 4', 5, 5' Octachlorobiphenyl **154.8 ug added**

Test ID	Liquids (ug)	Container Rinses (ug)	Solids (ug)	GAC Sum (ug)	Total (ug)	Percent recovered
34	0.022	25.930	114.850	0.043	140.844	90.98
35	0.027	23.240	132.020	0.010	155.297	100.32
36	0.047	51.200	98.440	0.003	149.690	96.70
37	0.185	78.990	77.090	0.003	156.267	100.95

TEST 3 Supernate results

ng of PCB congener found

Sample Identifier	34 F	34 U	35 F	35 U	36 F	36 U	36 F (ppt)	36 U (ppt)	37 F	37 U	37 F (ppt)	37 U (ppt)
2,4 DiCB	20.1	1344.0	10.2	1201.0	10.2	0.9	40.0	76.4	1.8	1.3	381.0	60.3
2,2',4,4' TetraCB	10.2	1738.0	12.1	2098.0	17.2	18.1	10.6	205.0	2.0	11.6	17.0	945.0
2,2',4,4',6,6' HexaCB	2.7	1255.0	10.0	1541.0	12.1	5.8	6.0	199.0	1.6	4.3	7.2	1338.0
2,2',3,3',4,4',5,5' OctaCB	0.5	54.1	0.5	66.6	0.4	0.8	1.0	56.0	0.2	0.5	22.1	208.0

F = Supernate filtered using a 0.45 u PVDF filter

U = Unfiltered supernate

ppt = precipitate after pH adjustment

TEST 3 GAC (Granular Activated Charcoal) Detail

2,4 Dichlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
34	0.00	1205.67	0.90	794.33	324.20
35	462.00	709.22	6.90	432.62	10.80
36	9.00	354.61	17.70	212.77	2.80
37	140.00	638.30	7.90	0.00	143.30

TEST 3 GAC (Granular Activated Charcoal) Detail

2, 2', 4, 4' Tetrachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
34	35.00	257.64	3.30	17.47	34.00
35	32.00	74.24	4.60	10.92	11.20
36	18.00	87.34	6.70	6.55	1.80
37	62.00	117.90	4.50	0.00	23.70

TEST 3 GAC (Granular Activated Charcoal) Detail

2, 2', 4, 4', 6, 6' Hexachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
34	59.00	111.29	2.30	16.45	24.40
35	24.00	40.32	6.10	19.68	43.80
36	23.00	38.71	3.60	4.03	1.30
37	44.00	46.77	3.90	0.00	5.00

TEST 3 GAC (Granular Activated Charcoal) Detail

2, 2', 3, 3', 4, 4', 5, 5' Octachlorobiphenyl

Test ID	Plug 1 (ng)	Trap 1 (ng)	Plug 2 (ng)	Trap 2 (ng)	Plug 3 (ng)
34	3.00	31.75	0.80	3.17	3.80
35	0.00	0.00	0.90	3.17	6.20
36	1.00	0.00	0.30	1.59	0.20
37	2.00	0.00	0.40	0.00	0.30