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REM III PROGRAM

REMEDIAL PLANNING ACTIVITIES AT SELECTED UNCONTROLLED HAZARDOUS SUBSTANCE DISPOSAL SITES

FINAL DESIGN REPORT
REMEDIAL DESIGN
WIDE BEACH DEVELOPMENT SITE
WIDE BEACH, NEW YORK
TOWN OF BRANT
ERIE COUNTY, NEW YORK

FEBRUARY 1989
APPENDIX C
LABORATORY SCALE TESTING REPORT:
KPEG PROCESSING OF SOILS

EPA CONTRACT 68-01-7250

EBASCO SERVICES INCORPORATED

EPA WORK ASSIGNMENT NUMBER: 86-2L46
EPA CONTRACT NUMBER: 68-01-7250
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LABORATORY SCALE TESTING REPORT:

KPEG PROCESSING OF WIDE BEACH DEVELOPMENT SITE SOILS

September 30, 1988

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EXECUTIVE SUMMARY

Three types of soil from the Wide Beach Development site were tested under laboratory conditions to determine the feasibility of KPEG treatment for this site. The soil samples were a very high PCB, moderate organic soil (A),a low PCB, low organic soil(B) and a high PCB, high organic soil (C).

PCB levels in each type of soil were successfully reduced to below the 10 ppm objective set for this project. Analysis of the reagents and washes for PCB proves that the PCBs were destroyed and not merely extracted into the reagent and wash water.

Reaction times ranged from 4 hours for soil with an initial PCB concentration of 24 ppm at 140°C to 8 hours for soil with an initial PCB concentration of 690 ppm at 150°C. Both reaction times include the time required for heating the soil/reagent slurry from room temperature to the reaction temperature. The optimum reaction temperature for high PCB soils was found to be 150°C.

The treatment reagent was 1:1:2:2 PEG:TMH:DMSO:45%KOH(in water) t 100% loading. In other words, a 300 g batch of soil was treated with 50 g of PEG (polyethylene glycol with an average molecular weight of 400), 50 g of Dowanol TMH (triethylene glycol methyl ether and higher homologs), 100 g of DMSO (dimethyl sulfoxide) and 100 g of a 45 % (w/w) KOH (potassium hydroxide) in water. An early test in which the DMSO was replaced with sulfolane (tetrahydrothiophene-1,1-dioxide) showed that DMSO was the better sulfoxide because of its lower cost and enhanced recoverability.

Analysis of the reagent and wash water from the bench scale reactions indicated that 52-82% of the reagent components (on a dollar weighted basis) were recoverable for recycling in bench scale equipment. If the reagent recovery results are corrected for the relatively poor mass recovery of the reagent and wash water streams, the dollar weighted reagent recovery is 69-96%. These recoveries are roughly comparable to previous pilot study values.

Data from a series of reactions at 140°C indicate that higher organic content in the soil does not have a measurable effect the reaction rate, but does reduce reagent recovery and therefore increases treatment cost.

Settling with decantation was tested and found inadequate for separation of reagent and washes from soil. Pressure filtration produced better reagent recovery.

The samples of soil used for the lab study contained less than 1% of the soil weight as particles larger than 0.25 inches. The early suggestion that sorting the soil by particle size might reduce costs by avoiding the need to treat the larger rocks and pebbles was found impractical. The potential for dust evolution and human exposure to PCB dust would make sizing (below the 2" size required for the equipment) too hazardous in light of the small savings potential.

The treated soil was found non-mutagenic by the Ames test using two strains of Salmonella bacteria. The treated soil was found to be non toxic by oral administration to guinea pigs - the species most sensitive to PCBs. The LD50 of treated soil is over 5000 mg/kg.

The laboratory data indicate that KPEG treatment of Wide Beach soil will cost approximately $100 - $300 per ton of soil not including excavation.

Based on the results of the bench study, a pilot study using a 40 gallon reactor is recommended as the next step in scaling up to full scale soil processing.

TABLE OF CONTENTS

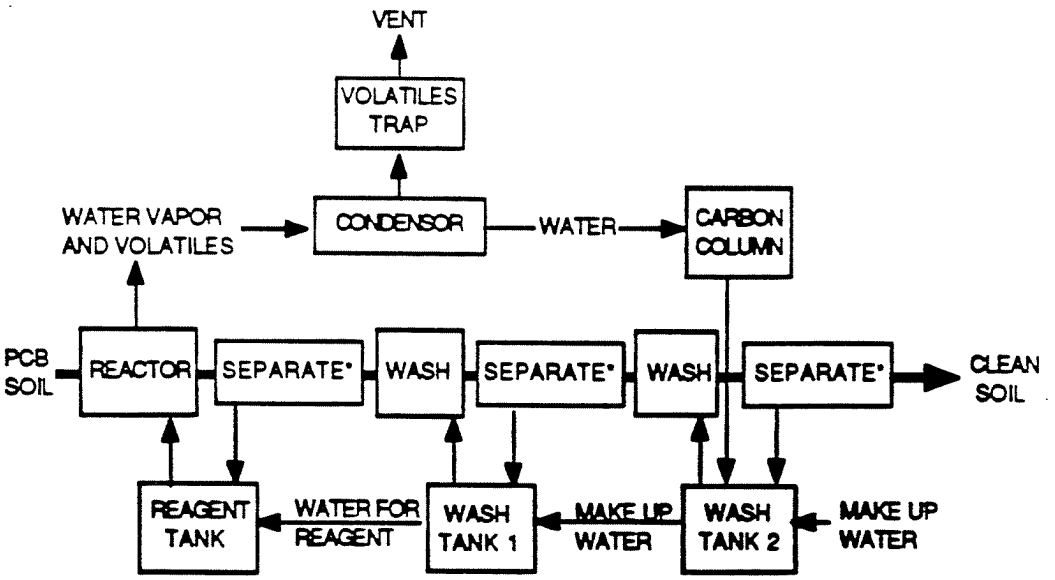
1. INTRODUCTION

The Wide Beach Development site is a residential development on the shores of Lake Erie. Waste oil applied to local roads as a dust suppressant contaminated the site with polychlorinated biphenyls (PCBs) in the late '60s/early '70s. An estimated 30,000 cubic yards of PCB contaminated material is present at the site, with contamination generally restricted to the top layer of soil.

This report contains the results of a laboratory study in which a KPEG treatment process developed by Galson Research Corporation (GRC) was tested for application to the Wide Beach Development site. The objectives of the laboratory study were to establish the reaction parameters for a larger scale pilot study, to evaluate the process exit fractions for disposal and/or re-use, and to make an initial estimate of the cost of full scale soil decontamination.

In KPEG soils processing, soil and reagent are mixed to form a slurry. The soil/reagent mixture is then heated to about 150 °C and held at that temperature with constant agitation until the PCB concentration is reduced to below the set clean level, in this case 10 ppm. At the end of the reaction, reagent is recovered by filtration and by washing the soil with three volumes of water. The decontaminated soil is then discharged, and the reagent and wash waters are recycled, as shown in the process diagram below.

PROCESS DIAGRAM FOR KPEG SOIL DECONTAMINATION



*SEPARATION BY PRESSURE FILTRATION, CENTRIFUGATION, OR SETTLING/DECANTATION

The reagent components include: a sulfoxide, e.g. sulfolane (SFLN) or dimethyl sulfoxide (DMSO); a glycol and/or capped glycol, e.g. polyethylene glycol 400 (PEG) and/or triethylene glycol methyl ether and higher homologs (TMH); solid or aqueous potassium hydroxide (KOH); and water. The glycol is reacted with KOH in the

presence of DMSO to form an alkoxide. The alkoxide reacts with one of the chlorine atoms on the biphenyl ring to produce a glycol-biphenyl ether and potassium chloride The sulfoxide acts as a cosolvent and catalyst , increasing the overall rate of reaction. The reactions involved are shown in Figure 2.

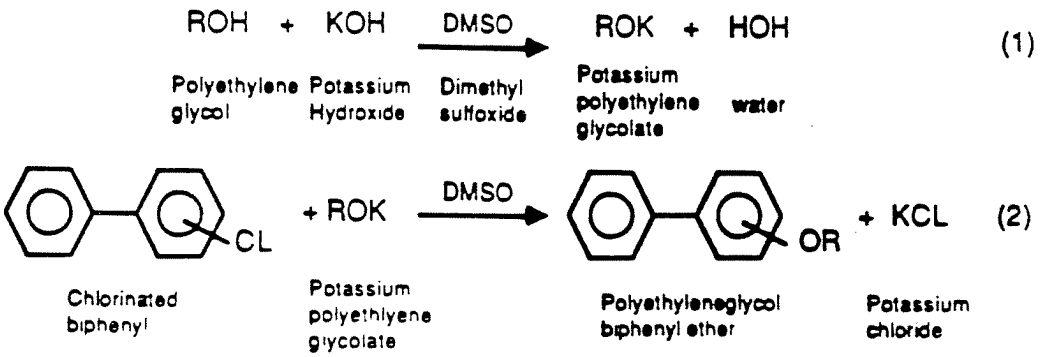


Figure 2. Reactions

Regardless of the processing scale, the reaction system is closed during the reaction to prevent release of materials to the environment. Water is distilled out of the reactor and collected in a condensate receiver. A trap is in line between the condensate receiver and the environment to collect any volatile compounds that are not condensed. The laboratory scale reaction apparatus is shown in Figure 3.

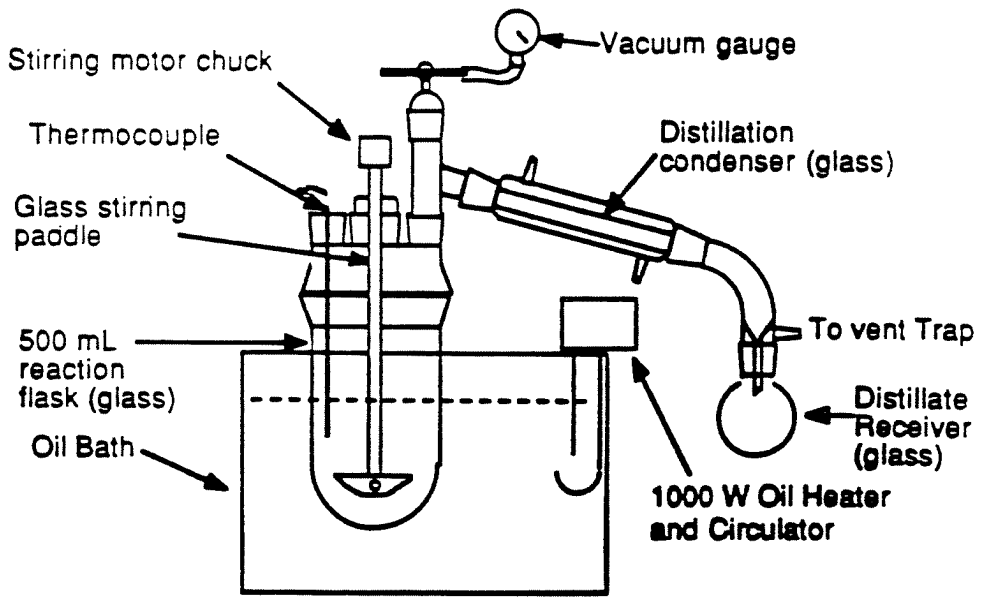


Figure 3. Lab Scale Soil Reactor

At the end of the reaction, reagent is recovered by decantation or filtration and the soil is washed with several volumes of water. In full scale processing, the decontaminated soil is discharged, and the reagent can be recycled. In a laboratory study, recycling is impractical because of the mass losses associated with the high surface to volume ratio of laboratory equipment. The reagents and washes are simply analyzed for reagent components so that the efficiency of reagent removal and the potential for reagent recycling on a larger scale can be evaluated.

This lab study proceeded according to the protocol of November 24, 1987 (see Appendix 1). The protocol called for three reactions, one with each soil type, using the same reagent formulation and reaction temperature to verify that the different soil types react similarly. Further experiments could then be done using only one of the three soils. A second reagent formulation (with sulfolane replacing DMSO) was to be tested to determine which formulation would be most cost effective. After selection of the best reagent, a second reaction temperature was to be tested: a lower temperature if all three soils went to <10 ppm PCB within 8 hours or a higher temperature if the PCB concentration in any of the soils was over 10 ppm after 8 hours. The final reaction was to be used to test settling and decantation as an alternative to pressure filtration as the method for separating reagent and wash water from the soil.

In addition to investigating reagent composition, reaction temperature, and separation methods, the protocol called for checking the PCB concentration of all process exit fractions, testing the toxicity and mutagenicity of the treated soil, and investigating the effect of processing on the organic content of the soil.

The protocol also called for investigation of factors that affect the cost of soil processing. These factors included particle size, recovery of reagent components, and the cost of waste disposal.

2. ESTABLISHMENT OF REACTION PARAMETERS

In order to proceed with a pilot study and full scale treatment of the soil at Wide Beach, the effectiveness of processing parameters such as temperature and reagent composition and the material handling methods must be established. The purpose of this portion of the laboratory study was to determine what reagent, temperature, and separation method produce the most promising results so that the minimum time would be used at the more expensive pilot and full scale stages. GRC's previous experience has shown that laboratory data accurately predict the effectiveness of reagents and reaction temperatures at pilot and full scale stages. Reagent recovery and separation techniques are more problematic in the lab because of the large surface to volume ratios of laboratory equipment and the associated mass losses. Lab data can be used to compare separation techniques and select the best one, but it cannot predict the absolute recovery of reagent and wash water in larger scale equipment.

2.1 Overview of Reactions: Heating and PCB Analysis

Three types of soil from the Wide Beach site were selected for testing. Soil A had a PCB concentration of 690 ppm although there was one "hot spot" at 1200 ppm. Soil B had a PCB concentration of 24 ppm. Soil C had a PCB concentration range of 490 to 620 ppm and the highest content of organic materials as determined by visual inspection. These three soils were passed through screens with 0.25 inch openings to remove any large rocks or sticks and were thoroughly mixed to make each batch as homogeneous as possible.

Six, 300 g batches of PCB contaminated soil have been processed in GRC's laboratory. The reagent formulation was 1:1:2:2 PEG:TMH:DMSO:45%KOH(in water) and the loading was 100%. In other words, a 300 g batch of soil was treated with 50 g of PEG (polyethylene glycol with an average molecular weight of 400), 50 g of Dowanol TMH (triethylene glycol methyl ether and higher homologs), 100 g of DMSO (dimethyl sulfoxide) and 100 g of a 45 % (w/w) KOH (potassium hydroxide) in water. For reaction #4, the DMSO was replaced with an equal weight of sulfolane (tetrahydrothiophene-1,1-dioxide). The initial four reactions were only heated to 140°C due to equipment problems. The fifth and sixth reactions were heated to 150°C and 160°C respectively. Table 1 contains temperature and PCB concentration data for the six reactions.

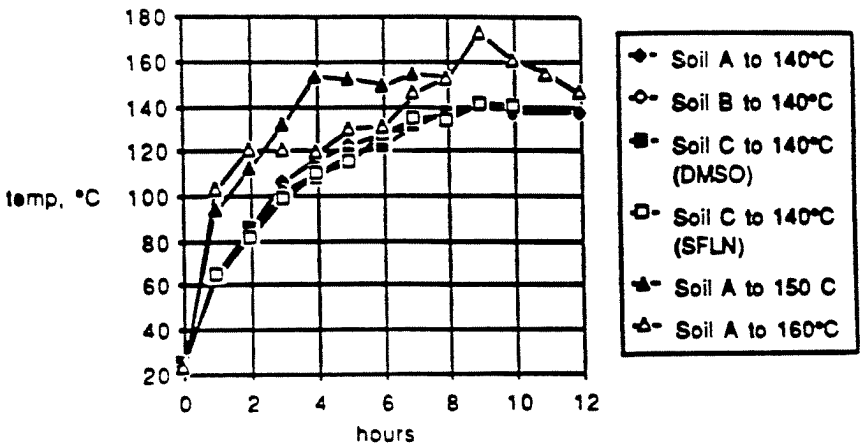
Table 1. Wide Beach Lab Reaction Results

Reaction #	1		2		3		4		5		6	
Soil used	Soil A		Soil B		Soil C		Soil C		Soil A		Soil A	
Max. Temp.	140°C		140°C		140°C		140°C		150°C		160°C	
Sulfoxide	DMSO		DMSO		DMSO		Sulfolane		DMSO		DMSO	
Time, hours	Temp	ppm	Temp	ppm	Temp	ppm	Temp	ppm	Temp	ppm	Temp	ppm
0	25	1200	25	24	25	620	25	490	25	690	23	640
1	66	620	63	16	64	550	65	380	94	740	103	690
2	86	590	82	21	86	670	82	1260	113	790	121	
3	106		102		99	430	99	760	132	156	120	
4	116	140	110	1.8	109	480	111	460	154	24	119	
5	123	92	119	0.99	118	35	115	59	152	9.8	130	
6	129	66			123	20	128	42	149	8.3	132	140
7	131	38			132	5.7	135	65	155	6.2	146	140
8	138	32			138	5.7	134	2.6	153	5.6	153	120
9	140				142	5.1	141	6.7			173	63
10	138	32			140	6.5	140	3.8			161	
11											155	49
12	138	24									146	71

Reactions #1 and #6 failed to reach the 10 ppm clean level. The reasons for failure of these reactions will be discussed later in this report. The only reaction of soil A that did reach the clean level was reaction #5. For that reason, the temperature and reagent formulation used for reaction #5 are recommended for the pilot test.

Figure 1 is a graph of temperature vs. time for the six reactions. This graph shows that the reactions that were heated to 140°C all required 5 hours to reach 120°C and 8 hours to reach the desired temperature. These reactions were done two at a time in a large oil bath heated with a 1000 W circulating heater (Hake model F4-M). Since the heating curves for the 140°C reactions are similar, differences in reaction rates for these reactions will be due to factors other than heating. The last two reactions were done in a smaller oil bath with the same 1000 W heater. The 150°C reaction required only 2.5 hours to reach 120°C and reached its target temperature in only four hours. The 160°C reaction reached 120°C in 2 hours. Violent boiling in the reactor made it necessary to temporarily stop heating that reaction. The differences in heating rates between reactions done two at a time and those done one at a time were due to differences in heating conditions, not differences in soils.

Figure 1. Wide Beach Soil Reaction Heatup



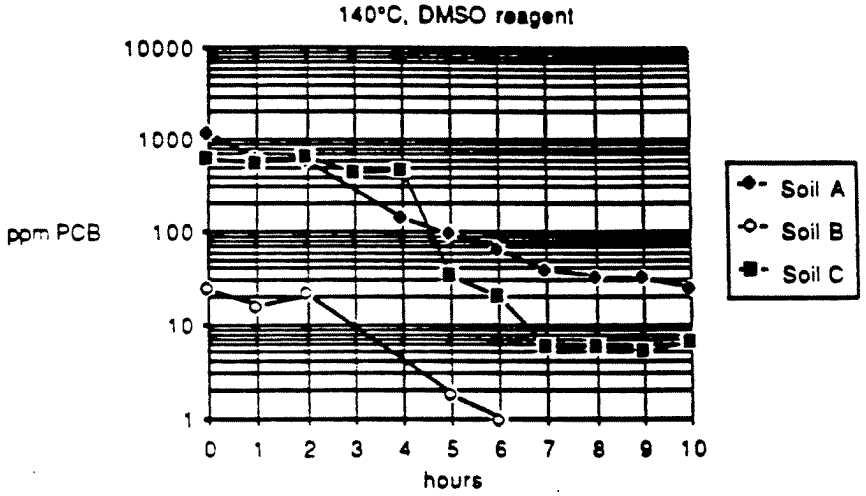
Distillation of water from the reactor takes place between 100°C and 120°C. In monitoring the PCB concentrations in the soil as the reactions progressed, it was noted that destruction of PCBs is very slow until the temperature exceeded 120°C. GRC's previous experience with transformer oil and other soils indicates that the reduced initial reaction rate is largely due to the quenching effect of water on the dechlorination reaction. The presence of water in the reaction mixture at the beginning of the reaction is unavoidable. Water is present in the untreated soil - about 24% of the soil weight is actually water. Pre-drying the soil would cause safety problems associated with generation of PCB contaminated dust. Water also serves to keep the KOH in a liquid state for easier handling and better recovery. When the PCB destruction reaction is complete, sufficient water must be returned to the reaction mixture to assure that the KOH will not solidify onto the soil or equipment during cooling. In lab reactions, this water is added when the reactor has cooled to between 100°C and 90°C. Cooing from 150°C reduces the thermal shock to the glassware and reduces the danger of steam burns to the person adding the water. In larger scale equipment, the water may be added while the reactor is still hot in order to speed up the cooling process.

Reactions of Wide Beach soil proceed more quickly when they are heated as rapidly as possible. Rapid heating allows the PCB dechlorination to get started before side reactions consume the KOH. (This factor will be discussed further in the reagent recovery section.) However, as noted in reaction #6, heating too rapidly between 100 and 120°C can cause violent boiling and associated process control problems. The limiting rate for heating depends on the design of the reaction equipment. In the lab scale equipment, the limiting heating rate for temperatures above 100°C is about 20°C/hour. During larger scale reactions, the heating process must be controlled so that safety is not compromised. The optimum heating conditions will vary with the size and design of the reaction equipment and must be checked at each stage of scale-up.

2.2 Similarity of Reaction Rates for Different Soils

Figures 2-4 are graphs of PCB concentration vs. time. Figure 2 shows that the three soils had similar reaction curve shapes when the maximum temperature and temperature ramp were the same. There was little reaction during the first two hours while the temperature was below 100°C. The slopes of the reaction curves were similar between two and six hours and the curves flattened out somewhat after 7 hours.

Figure 2. Wide Beach Initial Reactions



From these results, it is clear that although cycle time for decontamination may be dependent on initial PCB concentration, the different soil types are sufficiently similar that information obtained in lab tests using one batch of soil should be applicable to other batches of soil from the same site. Based on these results, all subsequent lab reactions were done using soil A - the "worst case" soil so that estimations of cycle time and other process parameters would be made conservatively and would provide adequate decontamination for the worst parts of the site. The PCB level of the A soil was roughly 10 times that of the average site soil. This set of reactions, at 140°C, failed to bring the PCB concentration below 10 ppm for the A soil, although the B soil was reduced to 1 ppm in 6 hours. Reaction at the temperature required to decontaminate soil A will also decontaminate soils B and C.

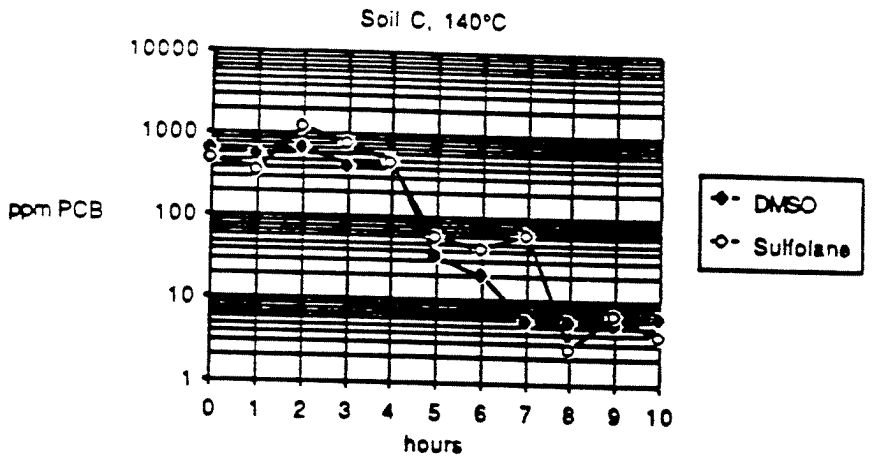
The fact that the additional vegetation and organic matter in soil C did not affect the reaction significantly is important for future decontamination plans. It means that the only pre-treatment the soil will require is breaking up clumps enough to prevent them from jamming the reactor stirring mechanism. Since organic matter does reduce reagent recovery, it would be desirable to remove as much vegetation from the soil as possible before processing. Procedures as simple as cutting the grass short and collecting the clippings before excavation can reduce overall processing costs.

2.3 Choice of Sulfoxide

The factor that differentiates the GRC/KPEG process from similar KPEG processes is the use of a sulfoxide as a catalyst and cosolvent. Sulfoxides greatly increase PCB dechlorination rate in comparison with KPEG alone. There are two sulfoxides that GRC has used in various lab and pilot studies; dimethyl sulfoxide (DMSO) and sulfolane (tetrahydrothiophene-1,1-dioxide). DMSO is cheaper and less viscous than sulfolane. Its slightly lower melting point makes it slightly easier to handle in cool weather, but its higher volatility makes it more prone to co-distill from the reactor with the water. Sulfolane is more thermally stable than DMSO. If reactions are to be done at temperatures in excess of 180°C, DMSO can not be used.

Figure 3 provides a comparison of DMSO with sulfolane as the sulfoxide in the reagent. The reaction curves were similar, although there was a little more scatter in the sulfolane reaction curve.

Figure 3. Wide Beach, DMSO vs. Sulfolane



The use of sulfolane did not significantly affect the reaction curve. However, the finished soil from the sulfolane reaction had a stronger odor than the soil from the DMSO reaction, although neither odor was extreme enough to cause problems. Heating of soils in the presence of polar solvents, including water, produces a change in soil odor, probably by extraction of amines and/or sulfur containing compounds from the organic fraction of the soil. Usually DMSO reactions have a stronger odor than sulfolane reactions, perhaps due to more efficient extraction. In this instance, the odor difference may simply be due to reduced recovery of the sulfolane reagent from the soil.

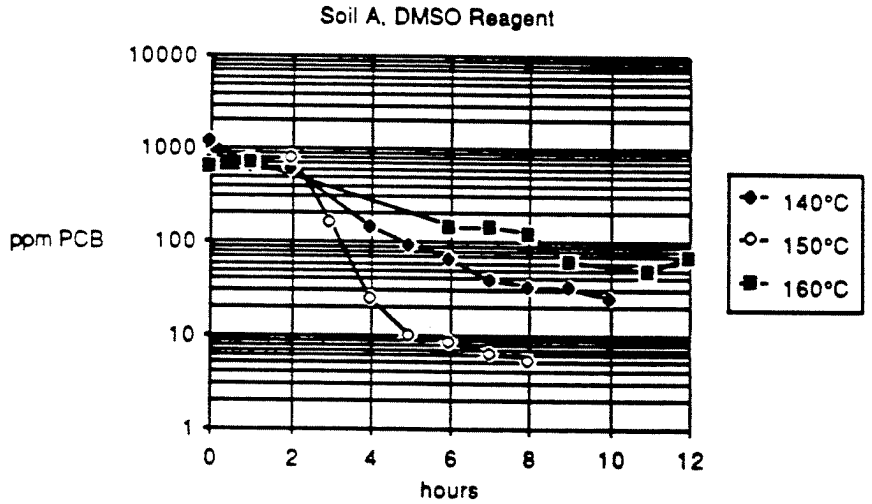
Reagent recovery data also indicate that DMSO is the more desirable sulfoxide. The sulfolane reagent was not recovered from the soil as well as the DMSO reagent. Since the use of sulfolane does not improve the results or odor of the reaction and is significantly more expensive than DMSO, DMSO was chosen as the sulfoxide for all subsequent work.

2.4 Effect of Temperature

The objective of studying the effect of temperature on the dechlorination reaction was to determine the temperature which provided the maximum reaction rate without significant reagent degradation.

Figure 4 shows the effect of 10° changes in reaction temperature. The 150°C reaction had a steeper slope and a lower final concentration than the 140°C reaction. Reaction at 160°C was less effective than reaction at 140°C, probably because the temperature halt made necessary by violent boiling in that reaction allowed KOH consumption to proceed faster than PCB dechlorination. This phenomenon will be explained in more detail in the discussion of reagent recovery.

Figure 4. Wide Beach, Effect of Temperature



The portion of soil A used for the 160° reaction appeared to contain more oil than the portion used for 140°C and 150°C reactions in spite of considerable effort to homogenize the soil before any reactions were done. The oil appeared in the distillate from the reaction. It is also possible that the soil contained the same amount of oil, but more of it distilled over because of the increased temperature.

Analysis of the reagents and washes for PEG (polyethylene glycol), TMH (triethylene glycol methyl ether and higher homologs), and DMSO (dimethyl sulfoxide) indicated no significant difference in reagent recovery between 150°C and 140°C reactions and slightly enhanced recovery for the 160°C reaction. Decantation was used as the separation technique for the 160°C reaction. The enhanced reagent recovery is the result of increased concentrations of reagent components found in the wash waters. In any case, degradation of PEG, TMH, or DMSO does not seem to be a factor in the slower reaction rate at 160°C. Based on the reaction rate results, 150°C is recommended as the reaction temperature for the pilot study.

2.5 Filtration vs. Settling

After a batch of soil is decontaminated, it is necessary to remove the reagent from the soil. Good reagent removal makes reagent available for recycling and leaves the soil ready to be returned to the environment. Two methods of reagent removal were tested in this study, pressure filtration and settling with decantation. Pressure filtration has a higher stage efficiency - that is it removes more liquid from the solids at each pass - but it requires some equipment. Settling with decantation requires no additional equipment but it does require that the soil be left in the reactor for a longer time for each soil wash and it has a lower stage efficiency - that is a lot of liquid is left with the soil for each pass and therefore more passes are required to get the same removal of reagent components from the soil.

Pressure filtration was used as the separation technique for the first five reactions. The sixth reaction, which was done using soil A at 160°C, was used to test settling and decantation as an alternative to pressure filtration for reagent recovery and soil washing. The reason for doing this test was to determine whether a cost savings could be realized by avoiding the use of filtration equipment.

Separation of reagent from the soil was less effective when decantation was used. Less reagent was recovered in the initial separation, and less soil was recovered after the final wash. Pressure filtration produced 155.5 g of finished dry soil from reaction 1 and 154.5 g of finished dry soil from reaction 5. Settling and decantation produced only 130.1 g of finished dry soil from reaction 6. All three reactions started with 300 g of moist soil. The lower soil recovery probably reflects loss of fine soil particles in decanting reagent. The soil/reagent interface was not visible, since the reagent became dark and opaque during the reaction.

The percentage of KOH in decanted reagent was markedly lower than the concentration of KOH in filtered reagent. The reagent formulation used in these reactions was a two phase mixture. The upper layer was richer in PEG, TMH, and DMSO while the lower layer was richer in KOH. When decantation was used to remove reagent, the KOH rich layer was left with the soil. In addition, the mass of reagent recovered by decantation was somewhat lower that the mass recovered from the same soil by filtration. Analysis of the reagents and washes for PEG, TMH and DMSO indicated reduced reagent recovery and soil washing efficiency with decantation. Based on these results, filtration provides a faster and more efficient means of separating liquids from the soil. The cost of the additional equipment will be offset by enhanced reagent recovery and faster soil processing.

3. ADDITIONAL EFFECTS OF PROCESSING

This part of the laboratory study had several objectives:

1. to verify that the PCBs from the soil were destroyed, not just moved to reagent, wash water or distillate.
2. to determine whether the wash water and distillate water could be recycled without further treatment.
3. to make certain that spent reagent would not have significant PCB concentrations.
4. to investigate what effects KPEG processing would have on the soil properties, particularly humic acid content and toxicity.

3.1 PCB Analysis of Exit Fractions

The exit fractions from this process are finished soil, condensate from the reaction, reagent, and wash waters. Analysis of these fractions for PCB is needed to evaluate the potential for recycling the reagent and wash waters, to evaluate potential use of condensate as a source of additional wash water, and to verify that the soil is adequately decontaminated. Exit fractions from all of the reactions were analyzed for PCB and the results are included in the complete list of PCB results for this project in Appendix 4. Table 2 is a summary of the results.

Table 2. PCB Analysis of Exit Fractions

Reaction #	1	2	3	4	5	6
Untreated soil	1200	24	620	490	690	640
Last soil sample during reaction	24	0.99	6.5	3.8	5.6	71
Final washed soil	NR	1.6	6.8	4.2	NR	NR
Condensate	NR	NR	NR	NR	NR	NR
Reagent	43	3.6	1.5	4.2	3.2	26
Wash 1	1.6	1.2	0.7	0.3	0.5	38
Wash 2	0.8	0.2	0.7	0.2	0.7	13
Wash 3	0.8	0.3	0.4	0.5	0.1	4
Wash 4					1.4	

PCB concentrations are in µg/g (ppm).
NR = Results not reported due to interference peaks

Three of the final soil samples and all of the condensates produced a distinctive peak pattern associated with interference from Teflon. Since many of the Teflon peaks co-elute with PCB peaks, they must be calculated as PCBs - thus causing inflated PCB concentrations to be reported. Since the results obtained from the analyses affected by the interference are not valid, they are not reported here. The most likely source of soil contamination was the lining of the pressure filter. The pressure filter was pre-leached before use by soaking it overnight in hot reagent, but scratches in the surface caused by soil particles could have exposed new surfaces which had not been adequately leached.

The contamination of the distillates was caused by applying lubricant to the teflon sleeves used to prevent sticking of the ground glass joints in the condenser system. We have not seen such contamination in other projects of this type because lubricant is not usually applied to ground glass joints where teflon sleeves are used. The lubricant enabled the teflon sleeves to release interfering compounds which would not usually be released. Since it was necessary to extract the entire distillate from each reaction, it was not possible to repeat these analyses. Condensate analysis will have to be repeated in the pilot test phase of this project when there will be enough sample to repeat an analysis if necessary and when no teflon sleeves will be needed.

Concentrations of PCBs in reagent were below 5 ppm except for reactions 1 and 6 when the soil PCB concentration did not reach the 10 ppm clean level. When the soil is below 10 ppm PCB, the reagent separated from it is below 5 ppm.

All of the washes were below 2 ppm PCB except for the reaction #6 where the PCB concentration was not adequately reduced in the soil. The reaction conditions used for reaction #6 will not be duplicated in pilot testing or full scale use of this process. Wash water can therefore be recycled without fear of cross contaminating batches of soil.

3.2 Effect of Processing on Total Organic Carbon in Soil

Because the reagent used in KPEG processing is extremely alkaline, there was concern that it would strip all of the humic acids from the soil and render it infertile. Analysis of soil for Total Organic Carbon (TOC) is an acceptable method of quantifying humic acids provided that there is no oil or other source of carbon on the soil.

Samples of treated and untreated soil were sent to Adirondac Labs for total organic carbon analysis by EPA-9060. The reports from Adirondac are included in Appendix 5. The results were as follows.

Sample #	Description	TOC
R70108711171212RP	Untreated soil A	1,140 µg/g
R70108711171130RP	Untreated soil B	700 µg/g
R70108711171145RP	Untreated soil C	1,600 µg/g
R70108712040303SRG	Treated soil A, Reaction 1	18,400 µg/g
R70108712040308SRG	Treated soil B, Reaction 2	13,700 µg/g
R70108712062315SRG	Treated soil C, Reaction 3	4,850 µg/g
R70108712062320SRG	Treated soil C, Reaction 4	29,000 µg/g
R70108712090530SRG	Treated soil A, Reaction 5	1,850 µg/g
R70108801201406SRG	Treated soil A, Reaction 6	17,600 µg/g

The results indicate that reagent components such as PEG are left in the soil after treatment, contributing to the Total Organic Carbon content of the soil. The wide variation in results indicates that washing efficiency was highly variable. The first two reactions were washed in the pressure filter without mixing the soil with the water before pressurizing. Subsequent reactions were mixed with each batch of water used to wash the soil.

The highest TOC concentration was found for reaction 4, in which sulfolane was used as the reagent instead of DMSO. Analysis for reagent components (see section 4.3) verified that sulfolane reagent is not washed off the soil as well as DMSO reagent. The TOC results from reactions 5 and 6 show clearly that pressure filtration removes reagent from soil better than settling and decantation.

There are no regulations for PEG, TMH or DMSO content of soils, since these are non-toxic compounds. As a part of the pilot study, a sample of treated soil could be extracted and analyzed for PEG, TMH, DMSO and KOH to determine the final soil content of these compounds. The effect the reagent components have on soil fertility is unknown at this time. A more productive approach to testing soil fertility might be to neutralize the residual KOH in the soil (the pH of treated soil is 10) with a dilute acid and fertilizer solution during the final wash, then plant some grass seed in it. This could be done with a batch of soil from the pilot study.

Since the TOC results showed only that reagent was not removed completely from the soil, they could not provide any information on the humic acid content of treated soil.

3.3 Soil Toxicity Testing

Although previous work done by the EPA has shown that the PCB/KPEG reaction products are non-toxic, there was no information that would prove that KPEG treatment of Wide Beach soil would not generate toxic by-products. It is possible that soil components other than PCBs could react with KPEG to produce toxic substances. As a precaution, toxicity testing was requested for treated soil.

It was important to neutralize residual alkalinity before toxicity testing so that any toxic effects would be due to compounds other than KOH, which is already known to be harmful by virtue of its causticity. Neutralization prior to toxicity testing is a valid procedure, since soil will not be returned to the environment until its pH is between 5 and 9, proving that any residual KOH has been neutralized or rinsed away. A portion of the final treated soil from the 150°C reaction of soil A (reaction #5) was neutralized by slurrying with water and adding hydrochloric acid. The neutralized soil was dried and sent to Illinois Institute of Technology Research Institute (IITRI) for toxicity testing. Two types of tests were done, an oral test with guinea pigs for acute toxicity and an Ames test with two strains of salmonella bacteria for mutagenicity.

The oral toxicity test was conducted by administering 5000 mg of the processed soil per kg of body weight to 5 male and 5 female guinea pigs. The animals were observed for 14 days and then killed and subjected to a limited gross necropsy. None of the guinea pigs died during the 14 day observation period. Consequently, the LD50 for guinea pigs was estimated to be greater than 5000 mg/kg. During the necropsy, all of the animals appeared to be clinically normal.

For Ames testing, the processed soil was suspended and diluted in dimethyl sulfoxide (DMSO) and filtered through a 0.45 micron filter. The DMSO was assumed to have extracted all the compounds of interest from the soil. The DMSO solution was added to the media in which the bacteria were growing. The dose levels tested were equivalent to 0.005, 0.05, 0.5, 1.0, and 5.0 mg soil per plate. Some of the plates were activated by addition of an extract (S9) from rat liver. After 2 days of incubation, the plates were examined to count the number of bacterial colonies that had undergone mutation. Doses ranging from 0.05 mg to 5.0 mg/plate were toxic to one of the tester strains (TA98). Therefore mutagenicity cannot be evaluated for that strain of bacteria except at 0.005 mg/plate, the lowest dosage level used. At that low dosage, the soil was non-mutagenic. The soil was non-toxic as well as non-mutagenic to the other tester strain (TA100) with and without activation at doses up to 5.0 mg/plate. The results of the Ames test indicate that processed soil is not mutagenic. A complete report is in Appendix 6.

4. FACTORS AFFECTING THE COST OF PROCESSING

The major cost factors for KPEG processing are the amount of soil to be treated, equipment cost, cycle time, and reagent cost. Reagent cost is strongly dependent on how much reagent can be recovered from each batch of soil, what chemicals must be replaced in order to recycle it, and how many times it can be recycled before it is no longer effective or requires reprocessing. The objectives of this section of the lab study were to determine whether certain size fractions of the soil would not require treatment, what mass recovery was obtainable for reagent and wash water in lab equipment, how much of each reagent component could be accounted for and what would have to be added to the reagent prior to recycling. Because of large (up to 30%) relative mass losses associated with lab size equipment, doing a series of reactions with reagent recycling in the lab is not expected to give useful data. The objective was to quantify the mass losses in lab equipment and examine the reagent quality after one use so that reagent costs for larger scale operations could be estimated.

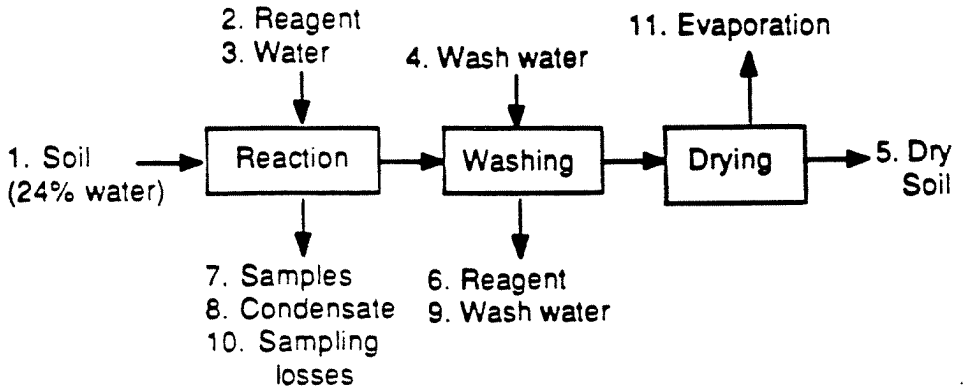
4.1 Particle Size Analysis

When PCBs are released onto soil, they become distributed in such a way that the concentration of PCB is greater in fine soil than it is on gravel or pebbles. The purpose of particle size analysis was to determine whether we could sieve out a significant portion of the site soil that would not require treatment because it's PCB content was already low. Large rocks have to be removed from the soil or broken up before treatment anyway. If we could avoid breaking up and treating large rocks, a cost savings could be achieved for the remediation of the whole site.

Results of the particle size analysis of the Wide Beach soil are provided in Appendix 7. The results were based on hydrometer analysis rather than dry sieving. Hydrometer analysis showed significant amounts of silt and clay in the soil. The soil testing lab (Empire Soils Investigations) decided that it would be unsafe to dry and sieve contaminated soil at their facility because of the dust that would be evolved and the high PCB concentration in the soil. In deciding whether to pursue analysis of sieve fractions, it was important to note that when the soil was passed through 1/4" screen before reactions were done, there was very little (<1% of the soil weight) retained by the screen. Sieving soil with screens under 1 inch without grinding in full scale operations would be difficult. In addition, there would be major problems with dust evolution. For these reasons, PCB analysis of sieve fractions has been abandoned.

4.2 Gravimetric Mass Balance

Gravimetric mass balances were calculated to quantify the mass losses for laboratory equipment. A block diagram of the laboratory process is shown below:



Gravimetric mass balances were calculated for each treatment test. Detailed results are in Appendix 8. A summary of the results is given in Table 3.

Table 3. Wide Beach Gravimetric Mass Balance Summary
(all weights in grams)

Reaction #	1	2	3	4	5	6
Inputs						
1.Soil (as received)	300.5	300.4	300	300	300.8	300
2.Reagent	300.6	300	300	301	300	300
3.Water*	53.7	33.4	121	47.1	233.6	181.7
4.Washes	720	600	720.1	720	960	910.5
Total Inputs	1374.8	1233.8	1441.1	1368.1	1794.4	1692.2
Outputs						
5.Soil (dry)	155.5	225.8	141.5	161.5	154.1	130.1
6.Reagent	162.9	194.3	155.3	65.7	228.9	147.8
7.Samples	64.5	27.3	64.3	50.9	38.9	59.5
8.Condensate	53.2	33.4	121	47.1	183.6	149.8
9.Washes	631.6	542.3	587.4	647.4	1007.3	889.6
Total Outputs	1067.7	1023.1	1069.5	972.6	1612.8	1376.8
Overall %Recovery	77.7	82.9	74.2	71.1	89.9	81.4
% Recovery of Reagent and Wash Water**	77.8	81.8	72.8	69.8	98.1	85.7
10.Sampling losses	85.8	34.7	100.4	110.1	42.2	101.1
11. Evaporation	221.3	176	271.2	285.4	139.4	214.3

*used to replace condensate and rinse apparatus during reaction
**100 x mass of reagent + wash recovered (output)/mass of reagent + wash (input)

The data in Table 3 are valuable in predicting reagent cost and water usage during larger scale processing. The mass recovery of reagents and washes is combined with the analytical recovery (Section 4.3, Table 4) to calculate reagent usage in the lab scale and to predict what the cost of reagent will be in full scale treatment.

Significant material losses were confined to two steps of the process: sampling and drying the soil after treatment. Sampling losses included the material that remained on the inside of the sampling pipettes and whatever water and other volatiles left the reactor while the samples were being taken. These losses were calculated by comparing the weight of the loaded reactor before reaction to the sum of the weights of the loaded reactor and all of the samples after reaction. Sampling losses will represent a smaller percentage of the total mass as the process is scaled up.

The evaporation loss represents the difference between dry soil and soil saturated with water. The soil that will come out of the reactor in full scale treatment will be saturated. The difference in water content between saturated soil and the initial water content of the soil will be the water usage for processing.

4.3 Recovery of Reagent Components

By analyzing the reagent and washes for reagent components, we can determine how much of the various components of the reagent have been degraded or left on the soil and how much has been transferred to the washes. We can then calculate what we need to add to the reagent to restore its original concentrations of the various components. From these calculations, we can estimate the cost of reagent for site remediation with recycling of the reagent.

All of the reagents and washes were analyzed by high pressure liquid chromatography (HPLC) for PEG, TMH, and DMSO and analyzed by titration for KOH. The results were used to calculate mass balances for each reagent component. Detailed results are in the mass balance spreadsheets in Appendix 8. The results are summarized in the Table 4.

Table 4. Analytical Recovery of Reagent Components as Percentage of Mass Used

Reaction #	1	2	3	4	5	6
Soil	A	B	C	C	A	A
Temperature	140	140	140	140	150	160
Sulfoxide	DMSO	DMSO	DMSO	SFLN	DMSO	DMSO
PEG						
Reagent	63	88	88	50	110	65
Wash 1	3	3	7	42		35
Wash 2				17		12
TOTAL	66	91	95	109	110	112
TMH						
Reagent	56	65	66	24	54	43
Wash 1	6	7	17	39	5	21
Wash 2	8	5	1	4	1	13
Wash 3	2			1		4
TOTAL	72	77	84	68	60	81
Sulfoxide						
Reagent	51	60	21	27	59	45
Wash 1	6	7	7	43	7	24
Wash 2	9	6	1	5	1	13
Wash 3	3			2		4
TOTAL	69	73	29	77	67	86
KOH						
Reagent	3	24	10	1	5	2
Wash 1	13	23	20	32	13	15
Wash 2	31	17	2	5	6	6
Wash 3	9	5	2	3	2	5
Wash 4					2	
TOTAL	56	69	34	41	28	28

The values in Table 4 are percentages of the mass originally used in the reaction that were found in the fraction. These percentages are calculated by multiplying the mass of the fraction recovered by the concentration of the compound in the fraction. For example, 162.9 g of reagent were recovered from reaction 1. The concentration of PEG in that reagent was 193 mg/g, so that there were (162.9 x 193 =) 31,400 mg or 31.4 g of PEG in the reagent. That accounts for 63% of the 50.1 g of PEG originally used in reaction #1. Likewise 3% of the PEG from reaction 1 was found in the first wash for a total of 66% of the PEG accounted for.

Analytical recovery refers to the ability to account for the whereabouts of the compounds, not to their immediate availability for re-use. Under actual processing conditions, after each reaction the reagent will be analyzed for the reagent components and fortified to return it to the original concentrations. Then part of the first wash will have to be added to the reagent to replace water lost in distillation. Part of the second wash will be added to the first wash to bring it up to the correct weight. Part of the third wash will be added to the second wash and the distilled water from the previous reaction (and perhaps additional fresh water) will be added to the third wash. The wash series will represent a counter current extraction series in terms of the

reagent components. It is assumed that during a long series of reactions, the concentrations of reagent components in the washes will reach a "steady state" and not change significantly from one reaction to the next. After the first few reactions, the reagent components in the washes will presumably have been replaced through fortification of the reagent. Therefore, analytical recovery of reagent components in the whole system after a single reaction roughly approximates actual recovery under steady state conditions.

Reactions 4 and 6 are the only reactions that had a detectable amount of PEG in the second wash. Reaction 4 was done with sulfolane as the sulfoxide instead of DMSO. Apparently, the sulfolane reagent does not wash off the soil as efficiently as the DMSO formulation. The difference is probably due to the higher viscosity of sulfolane. Reaction 6 used decantation as the separation technique instead of pressure filtration. It is obvious that improved washing efficiency is achieved with pressure filtration. PEG was not detectable in the third wash for any of the reactions.

Recoveries of TMH and sulfoxide agreed with each other and remained between 60 and 90% in all reactions except for #3, where the DMSO recovery was abnormally low. The reasons for low DMSO recovery in reaction #3 are not understood at this point in time.

It is interesting to note that KOH was not recovered in the reagent as well as it was in the washes. This is to be expected. The solubility of KOH in the glycol - sulfoxide mixture at room temperature (25°C) is only about 5%. GRC's experience has shown that KOH levels several times the solubility increase the reaction rate. The reagent formulation (1:1:2:2 PEG 400:TMH:DMSO:45% KOH) used throughout this project contained 15% KOH on a dry weight basis. After the PCB destruction reaction is complete, a small amount of water is added to the reactor during cooling in an attempt to prevent KOH from solidifying onto the soil and the reactor surfaces. It may be that there was not enough time allowed for dissolution and mixing before the initial filtration, leaving the KOH on the soil until the washes removed it. In larger scale processing, it will be possible to remove the reagent from the soil at a higher temperature, so that the KOH will be less likely to solidify onto the soil.

In comparing the raw data from the three reactions of soil A, there was a marked decrease in concentration of KOH in the reagent when decantation was used (5.6 mg/g for reaction 6) instead of pressure filtration (8.4 mg/g for reaction 1 and 10.1 mg/g for reaction 5), although the total recovery from all reagents and washes did not change significantly. The reagent formulation used in these reactions was a two phase mixture. The upper layer was richer in PEG, TMH, and DMSO while the lower layer was richer in KOH. When decantation was used to remove reagent, the KOH rich layer was left with the soil. Wash waters did not develop two phases, and the first wash of reaction 6 contained more KOH than the first wash of reaction 5.

The difference in KOH recovery between 140°C and 150°C was unexpected. The KOH recovery from reaction 5, at 150°C, agrees well with the KOH recovery from reaction 6, at 160°C. KOH recovery from reactions 5 and 6 is half that of reaction 1, done with the same soil at 140°C. Reaction #6 had a delay in its heat-up and failed to remove the PCBs from the soil (see sections 2.1 and 2.4) in spite of the increased reaction temperature.

The best explanation for all of these observations is that the soil slowly consumes KOH and that the loss of KOH prevented PCB destruction in reaction 6. To check that theory, a small sample of soil A was slurried with deionized water and titrated with dilute KOH. The pH was repeatedly brought up to 8.2 (slightly alkaline) by the addition of KOH, and slowly drifted down again as the KOH was consumed by the soil. Since the KOH recovery decreased between the 140°C reaction and the 150°C reaction it is reasonable to conclude that the consumption of KOH is accelerated at increased temperatures. Because of the heating delay in the 160°C reaction (#6), the KOH concentration in the reactor was probably greatly reduced before the temperature was high enough for the PCB dechlorination to proceed. With less KOH available to drive the PCB destruction reaction, the PCBs were not adequately reduced. The 150°C reaction (#5) probably had PCB destruction and KOH consumption going on at the same time. Most of the PCBs were probably removed before the KOH concentration was reduced enough to affect the reaction rate - i.e. before the fourth or fifth hour.

4.4 Economic Evaluation of Reagent Recovery

Reagent replacement is a major cost item for full scale processing. Other tests have shown that reagent recycle efficiency is strongly affected by process size. Larger scale processing provides greater recycle efficiency. This is demonstrated in the table below, which compares the results of Wide Beach laboratory studies to the results of Bengart & Memel (B&M) pilot (100 lb samples) test data. Mass recovery of the reagent and wash streams for the B&M pilot testing was 94-95%, vs an average 83% mass recovery for the Wide Beach lab runs. This is reflected in the estimated reagent costs for the two sizes of operation, with an average estimated cost of $44/ton soil based on pilot data vs $315/ton soil based on lab data.

Table 5. Reagent Recovery and Cost

	Wide Beach lab reactions using DMSO reagent						B&M Pilot data		Bulk
Compound	run 1	run 2	run 3	run 5	run 6	Average	run 1	run 2	$/lb.
PEG 400 ± 30%	66%	91%	95%	110%	112%	95%	100%	100%	0.61
TMH ± 10%	72%	77%	84%	60%	81%	75%	100%	94%	0.57
DMSO ± 10%	69%	73%	29%	67%	86%	65%	100%	100%	0.82
KOH ± 10%	56%	69%	34%	28%	28%	43%	82%	50%	0.17
mass recovery*	78%	82%	73%	98%	86%	83%	94%	95%	
reagent, $/ton soil†	$342	$245	$504	$318	$170	$315	$20	$68	
corrected cost**	$141	$67	$300	$303	$26	$169			

*100 x total mass of reagent and wash recovered (output)/total mass of reagent and wash used (input)
†Calculated replacement cost of reagent components lost during processing.
**corrected to 100 % mass recovery, assuming ratios of recovered compound do not change.

A great deal of the difference in estimated reagent costs between pilot and laboratory data is due to the low mass recovery of the laboratory case. If the calculated reagent cost is corrected mathematically to a 100% mass recovery, the estimated average reagent cost drops from $315 to $169/ton soil, as shown in the table above. Bulk reagent cost data are included to allow checking of calculations.

The average scaled estimated costs for each run, seen in the preceding table, are not randomly distributed. The two lowest cost runs had calculated reagent costs averaging $48/ton, very similar to the Bengart & Memel data, while the three highest cost runs had calculated reagent costs averaging $248/ton soil. The reason for this odd distribution is unknown. (note: Since we are interested only in recovery of reagent formulated with DMSO, data from reaction #4, in which the DMSO was replaced with sulfolane, are not used here.)

The poor mass recovery achieved in the laboratory study is probably a function of the high surface: volume ratio in laboratory equipment. Since this type of study had not previously been done using lab equipment, the exact relationship between mass recovery to equipment size is uncertain. The mass recovery should be improved by the use of pilot scale equipment, which will reduce the surface/volume ratio by roughly a factor of twenty.

4.5 Cost of Waste Disposal

The KPEG process has no normal discharges, except at the end of the project when the leftover reagent and wash water must be disposed of. For a unit such as that envisioned for Wide Beach, this will total some 6,000 gallons of reagent and 12,000-18,000 gallons of wash water. The wash water can be distilled by running it through the reactor units, leaving behind a residue (about 500 gallons total) of formerly dissolved reagent. The exact amount of residue from the water wash will be determined during the pilot step, when wash water from a number of recycle runs will be available. Thus the total amount of reagent and wash water still bottoms to be disposed of is on the order of 65,000 #.

The most functional method of disposing of the leftover reagent and distilled wash water is to take it to another site for use. If this is not feasible, disposal of the reagent material in a permitted PCB incinerator would cost about $20,000 including transportation. This estimate is based on 1988 telephone conversations with vendors and is subject to change without notice. In general, incineration would be the preferable method of treating the material.

4.6 Overall Cost of Processing

A summary cost estimate for one year's operation of a three reactor system is shown in the table below;

COST ANALYSIS FOR TRIPLE RIG OPERATIONS WITH PCB SOILS AT WIDE BEACH SITE

ASSUMPTIONS		
Nominal processing rate, tons/batch	90	(3 UNITS @ 20 YDS EACH @ 3000#/YD)
estimated cycle time, hours	12	
processing hours/day	24	
estimated % processing time	70%	
Average processing rate, tons/hour	5.25	tons/year=45990
depreciation, %/year	20%	
maintenance costs, % of capital/year	30%	
capital cost, $	$2,300,000	(ROUGH ESTIMATE - INCLUDING DESIGN COSTS)
average soil moisture level	24%	(based on soil A test results)
fuel oil costs, $/million btu	$10	
office overhead factor	2.5	
field overhead factor	1.5	
salaries, $/hour		
field manager	$22.0	(on all shifts)
field operator	$15.0	(on all shifts)
chemist	$17.0	(12 hour day shift only)
senior scientist	$29.0	

Costs for processing (no profit)	# units	cost/unit	total	$/ton	% of total $
1. Per diem	1898	$75	$142350	$3.10	1.9%
2. personal protective equipment	1898	$40	$75920	$1.65	1.0%
3. reagent	45990	$50	$2299500	$50	30.1%
4. waste disposal	65000	$0.3	$19500	$0.42	0.2%
5. field labor					
field manager	8760	$33	$289080	$6.29	3.8%
chemist	4380	$26	$111690	$2.43	1.5%
field operator	8760	$23	$197100	$4.29	2.6%
6. office support					
senior scientist	876	$73	$63510	$1.38	0.8%
7. fuel costs	45990	$4.80	$220752	$4.80	2.9%
8. depreciation			$460000	$10.00	6.0%
9. maintenance			$690000	$15.00	9.0%
10. travel, Syracuse to/from WB site	270	$100	$27040	$0.59	0.4%
subtotal			$4591242		
contingency (25% of total cost)			$1912252	$42	25%
subtotal			$6503494		
Profit (15% of total cost)			$1147867	$25	15%
TOTAL COST, $/YEAR OF OPERATION			$7656561	$166	100%

The cost breakdown for full scale processing is approximately 35% raw materials (reagent, waste and fuel), 25% capital and labor, and 40% contingency and profit. This can be used to set a rough calculation formula for estimation of costs as a function of reagent cost and cycle time.

$/ton soil	= (($/year fixed cost)/(tons/year)) + ($/ton variable cost)) *(1+1/(contingency+profit))
fixed cost	= per diem + PPE + waste disposal (assumes annual clean out of reactor and disposal of all used reagent and washes) +labor + depreciation + maintenance. + travel
fixed cost	=2070990 $/year
tons/year	= (90 tons/batch/cycle time, hours)*8760 hours/year*fraction operating time
	=551,880/cycle time, hours
variable cost	= fuel cost+reagent cost
variable cost	= $4.8 + reagent cost, $/ton
$/ton soil	=((2070990*cycle time/ 551,880)+4.8+reagent cost)*1.66
	=((3.75*cycle time, hours)+$4.8 fuel cost +reagent cost)*1.66
	=$7.97 +6.22*cycle time+1.66* reagent cost

This calculation can be used to help prioritize changes in operating conditions and equipment for achievement of minimum processing cost.

5. RECOMMENDATIONS FOR PILOT STUDY

The objective of the pilot study is to provide information required for design of a full scale treatment unit and to refine the estimates of reagent usage and treatment costs. There are some operations, such as removing reagent from the soil slurry while it is still hot, that cannot be done in lab scale equipment which have a significant impact on process efficiency and cost. The pilot stage is where these parameters are investigated more effectively. GRC recommends that two batches of soil be used to check the reaction performance with low PCB soil and high PCB soil. The remaining eight reactions should be done with average soil and used to investigate reagent recovery and recycling.

5.1. Soil Preparation Requirements

In order to proceed with the pilot study as efficiently as possible, Ebasco should provide GRC with 10 batches of soil, 25 gallons each, sieved through 0.5 inch screening, in 55 gallon drums. The batches should be as follows: one batch of high PCB soil, one batch of low PCB soil, and eight batches of average PCB soil. The eight batches of average PCB soil should be from a single, well mixed, composite so that the batches will be as similar to each other as possible. No pre-treatment other than sieving and homogenizing is required or recommended.

5.2 Reactor Operation

For each batch of soil, the reactor should be loaded with 25 gallons of soil and 25 gallons of reagent (1:1:2:2 PEG:TMH:DMSO:45%KOH). The reactor should be operated with a maximum temperature of 150°C and samples should be taken hourly an analyzed for PCB by GRC's usual rapid method. The reaction should be stopped when the PCB concentration is less than 10 ppm for 2 consecutive samples, when the reaction has ceased to reduce the PCB concentration in three consecutive samples, or after 8 hours at 150°C, whichever comes first.

5.3 Separation of Soil and Reagent

The reagent should be removed from the soil in the reactor by pressure filtration through a filter body built into the reactor. The filtration should be done while the soil/reagent mixture is still hot (over 100°C) so that reagent viscosity will be minimized and filtration efficiency will be maximized. (Hot filtration was not possible in lab equipment. The difference could be significant for full scale operations). If the filter body in the reactor plugs up or otherwise fails to function properly, the soil/reagent slurry should be dumped into a pressure filtration can salvaged from a pilot study done by GRC last year.

5.4 Reagent Recovery

The reagent should be analyzed for its components (PEG, TMH, DMSO, and KOH) after each reaction. The missing components should be replaced so that the reagent is fortified to its original strength. The reagent should then be brought up to the correct volume using water from the first soil wash. The first soil wash should be brought up to the correct volume with water from the second wash. The second wash should be brought up to the correct volume with water from the third wash. The third wash should be brought up to the correct volume with fresh water.

5.5 Washing of Soil

The soil should be washed in the reactor and the wash water should be removed from the soil through the same filter body used to remove the reagent. The pH of the soil slurried with the 3rd wash should be checked before final separation. If the pH is greater than 9, hydrochloric acid should be added to the reactor to bring the slurry to the pH range between 5 and 7. The slurry should then be agitated in the reactor for one hour and the pH checked to make sure it remains below 9. Records of the amount of acid added and the final pH of the slurry should be maintained carefully so that the cost of neutralization for full scale treatment can be accurately estimated.

5.6 Additional Investigations

PCB analysis of the distillates from the lab reactions was not successful (see section 3.1) and there was not enough sample to repeat the analysis. For that reason, PCB analysis of process distillates is particularly critical. An additional study should also be conducted to test procedures for recycling the distilled water if it is PCB contaminated. These tests would involve checking the effectiveness of a charcoal column in removing the PCBs from the water and establishing the volume of water that can be treated before PCBs break through the charcoal column. Such information is needed for designing the full scale treatment unit.

QA/QC activity during the lab study was minimal due to the small volumes of the various samples available. In many cases there simply was not enough sample in existence to do duplicates and spikes. For that reason the data from the lab study are of unknown quality. The Pilot study will generate sample volumes large enough that any analysis can be repeated several times if necessary. The pilot study can therefore be used to check the QA/QC procedures intended for the full scale remediation and to establish the quality of data generated by GRC's analytical procedures.

There is some concern whether the treated soil will be suitable for the front yards of Wide Beach residents. GRC suggests testing the ability of the soil to support grass. One of the early batches of treated soil could be placed in an open container, treated with a fertilizer solution and planted with grass seed. The container of soil could be watered occasionally and observed during the remaining pilot study.

Appendix 1.

Lab Testing Protocol

Wide Beach Bench Test Protocol

The protocol for testing the Galson APEG process on Wide Beach soil will consist of the following tasks.

1. As soon as soil samples are collected from the site, Galson will do a rapid screening of the samples to make sure that there is enough PCB in the soil to adequately represent conditions on the site and produce useful information on the dechlorination process kinetics.

2. Prepare 3 batches of soil for laboratory work. Batch A will have PCB concentration in excess of 1000 ppm and relatively low levels of humic and vegetative matter. Batch B will contain less than less than 500 ppm PCB and relatively low levels of humic and vegetative matter. Batch C will have a relatively high concentration of PCB and relatively high levels of humic and vegetative matter. Each batch of soil will be passed through a screen (1/4 " size) to remove large rocks, and will be blended to produce a homogeneous sample.

3. Grain size analysis of Soil A will be done by Empire Soil Investigations using ASTM methods D422 and D4318 (which replaced D423). The quality control procedures specified in the ASTM methods will be followed. The various size fractions will be returned to Galson Research. When the size distribution is known, Galson and Ebasco will confer and select size fractions for PCB analysis to determine the relationship between particle size and PCB concentration at this site. These PCB analyses will be done by the method described in Galson's original proposal.

4. Reaction curves (ppm PCB vs. time) for the three batches of soil will be generated using a reagent formulation of 1:1:2:1:1 PEG 400: TMH: DMSO: KOH: water at 150°C. Hourly samples will be taken from the reactors and analyzed for PCB. These reactions will continue until the PCB concentration is less that 10 ppm or for a maximum of 8 hours. If the reaction proceeds at similar rates regardless of initial PCB concentration and vegetative matter content, batch C will be used for all subsequent work. If the three reactions are not similar, Galson and Ebasco will have to confer and revise the subsequent reaction series and schedule. At the end of the reactions, soil and reagent will be separated by pressure filtration and the soil will be washed with water in the pressure filter. The various exit fractions will be weighed and analyzed for reagent components so that reagent use can be estimated. Reactions, PCB analysis and analysis for reagent components will be conducted according to the procedures described in the appendix of the original Galson proposal.

5. A reaction rate curve for soil C will be generated using 1:1:2:1:1 PEG 400: TMH: Sulfolane: KOH: water at 150°C. The exit fractions will be analyzed for reagent components and the results will be compared with the results of the initial reaction between soil C and the reagent containing DMSO. The two reagents will be compared for effectiveness, reaction rate, reagent stability and total processing cost. At this point, Galson and Ebasco will confer and select the best reagent formulation.

6. If the initial reaction using the selected formulation was successful in reducing PCB concentration to less than 10 ppm in less than 8 hours at 150°C, the reaction will be repeated at 120°C. [If the initial reaction required more than 8 hours to reduce PCB concentrations to less than 10 ppm. the reaction will be repeated at 180°C.] The reaction curve will be evaluated in terms of processing cost. At this point, Galson and Ebasco will confer and decide if additional temperature tests are desirable. This reaction will also be used to test settling and decantation as an alternative separation method for processing. The exit fractions (soil, reagent and washes) will be analyzed for reagent components and the results will be compared with the results of the initial reactions in which pressure filtration was used as the separation technique. Galson and Ebasco will confer and select the separation method for subsequent work.

7. Samples of the treated soil from the reaction using the selected temperature, reagent, and separation method will be sent to IITRI for toxicity testing. A two strain Ames test will be used to determine mutagenicity and an oral (gavage) test on guinea pigs will be done to determine acute toxicity.

8. The treated and untreated soil from all of the reactions will be analyzed for total organic chlorine. These analyses will determine whether the levels of chlorinated organic compounds in the treated soil are acceptable. Soil C will be analyzed for humic material before and after treatment. Humic material will be determined by subtracting oil and grease from the total organic carbon (TOC) content of the soil. Oil and grease will be determined by GTS and TOC will be determined by Adirondac Labs Inc. These analyses will provide information on the effect of processing on humic content of the soil and will provide information on the amount of humic matter that will be transferred to reagent and wash phases during processing.

9. The cost of disposal of anticipated waste streams (spent reagent, wash water, and process distillate) will be evaluated along with the total cost of various treatment methods that could be used for these waste streams. If cost savings could be achieved by treating the waste streams on site, the treatment methods will be tested in the laboratory and the results will be evaluated in terms of disposal costs.

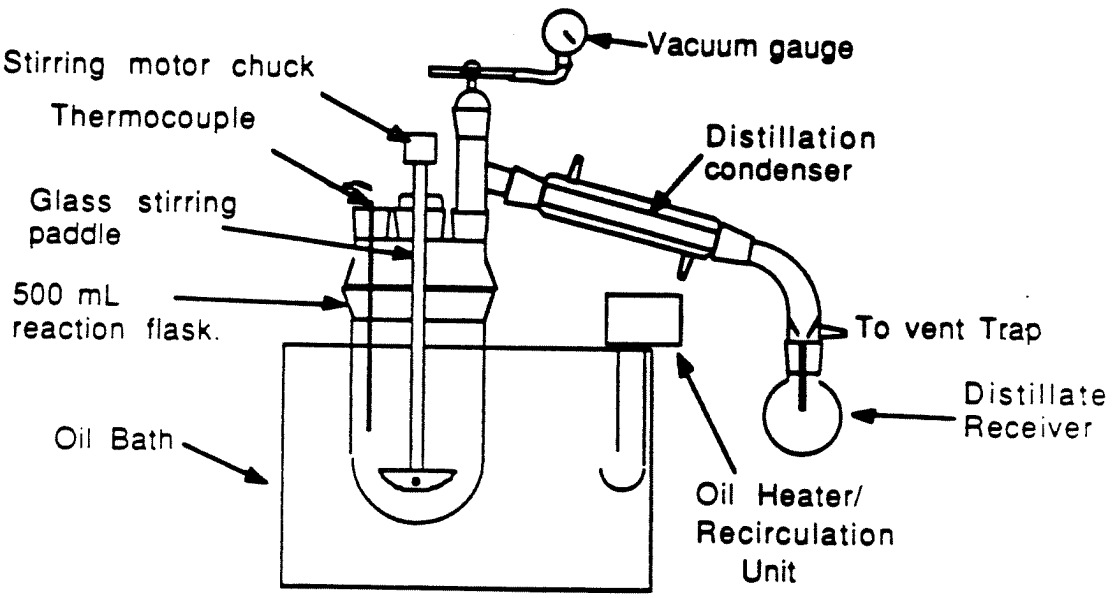
Appendix 2.

Reaction Procedures

Protocol for PCB Soil Reactions

The GRC standard soil reactor is illustrated below.

SOILS REACTOR



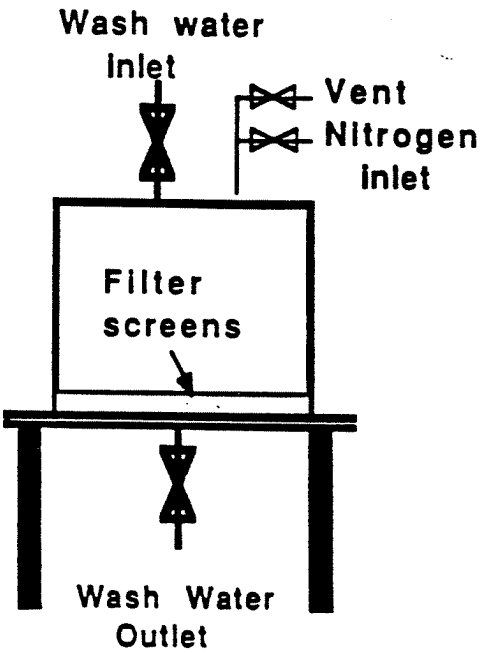
The bottom of the reactor is positioned in a thermostatically controlled oil bath as shown. The oil bath with circulating heater and associated ring stands will be set up in a fume hood. The distillate receiver and jars for reagent, soil, and washes will be tared before starting the reaction.

Soil, usually 300 g, and reagents (for example 200 g of 1:1:2 PEG:TMH:DMSO plus100 g of saturated KOH in water) are weighed into the reactor bottom. The reactor is clamped together and set up with all the accessories as shown above.

Reactions are timed from the start of heating. During heating, slight vacuum (1-2" Hg) is maintained so that water is distilled off. Water distills between 110 and 130°C. When the temperature approaches 140, the vacuum is turned off. Samples are taken throughout the reaction, about one per hour, and analyzed according to GRC's method for PCBs in soil.

After the PCB concentration in the soil has reached the desired "clean" level the reactor is cooled to about 100°C and water is added to liquify the KOH and restore the reagent to its original water content. The reactor is cooled to room temperature and emptied into the pressure filtration unit shown on the following page.

Pressure Filter Assembly



Nitrogen or air pressure is applied so that the reagent is filtered out and collected in its tared jar. Pressure is released and the filtration assembly is opened at the top. Washes are used to rinse the reactor and are then poured into the filtration assembly. Pressure is applied to force the washes through the soil. Each wash is filtered out and collected in its tared jar. The washing process is repeated for the desired number of washes, usually 3.

When all the liquids are in their tared jars, the jars (and the distillate receiver) are re-weighed and mass recoveries are calculated. The reagent and washes may be analyzed for the various reagent components so that mass balances for each component can be calculated.

Appendix 3.

Analytical Methods

GRC Analytical Method for PCB in Soil

Soil samples generated during APEG treatment require unusual treatment because the KPEG reagent is somewhat destructive to standard gas chromatography (GC) equipment. Care must be taken so that reagent is not included in the extract that is injected into the GC. This method has been developed to provide accurate results in the shortest time possible.

1. Soil Wash Step (not required for final treated soil or untreated soil)

Samples of the reaction slurry are collected directly in tared 8 mL vials. The samples are about 4-5 mL volume. The vials are re-weighed to obtain the weight of slurry used. Water is added to the vials to fill them up to the "shoulders" and the vials are capped with solid, teflon lined caps and shaken vigorously using a vortex mixer to suspend the soil. The vial is then centrifuged at 3200 rpm for two minutes. The supernatant is transferred into a 24 mL collection vial. Two additional aliquots of deionized water (4-5 mL each) are used to wash the soil by the same method. The vial containing the wet soil is re-weighed to obtain a wet soil weight.

2. Extraction Step

After the final water wash is carefully decanted, 1 mL of methanol and 3 mL of hexane are added to the 8 mL vials containing the soil samples. (Dry, untreated soil is weighed in a tared vial and saturated with 1:4 water:methanol. Hexane is then added as for treated samples). The vials are shaken vigorously on the vortex mixer to suspend the soil from the bottom of the vial. Shaking is continued for an additional minute. The vials are then centrifuged for two minutes as described above. The hexane layer is carefully transferred into a 15 mL graduated centrifuge tube with a teflon lined screw cap (or a 10 mL volumetric flask) using a disposable Pasteur pipette. Two additional aliquots of hexane, 3 mL each, are used to extract the soil+water+methanol slurry by the same method. Each hexane layer is transferred into the centrifuge tube. After the third extraction, the volume in the centrifuge tube is adjusted to 10 mL with fresh hexane. The centrifuge tube is mixed gently on the vortex mixer and 2-3 mL of the extract are transferred to a 4 mL vial with a teflon lined screw cap which contains 1 mL of concentrated sulfuric acid. The vial is shaken and the phases are allowed to separate. This procedure usually produces a colorless, dry extract. The sulfuric acid wash may be repeated if necessary. The clean extract is used for GC analysis. The remainder of the unwashed extract is transferred into an 12 mL storage vial and retained until a satisfactory analytical result is obtained for that sample. The level of extract in the 12 mL vial is marked as a precaution against solvent evaporation.

The vial containing the extracted soil is left uncapped in a hood or over a steam bath to dry. The vial and soil are then re-weighed to obtain the dry soil weight.

3. Chromatography

The acid washed sample extracts are diluted as required and used for GC analysis. PCB standards and blanks are injected on the same day as samples to provide instrument response data for calculations and adequate quality control for the analysis. Samples, standards and blanks are injected into one of two chromatographic systems. Conditions for each instrument are as follows.

Hewlett Packard 5890A
Injector temp.: 250°C, Manual injections with solvent flush
Column: 30 m x 0.543 mm ID fused silica coated with 1.5 µm DB-1
Carrier gas: nitrogen at 25 mL/min through column, makeup to 60 ml/min.
Temperature Program: 170°C, 25°C/min. to 270°C, hold 6 min.
Detector: Ni-63 ECD, base: 300°C
Integrator: Hewlett Packard 59970C Chemstation with GC software.
Threshold: 0, atten 2^6 (adjust as needed), Report mode: Area%

Shimadzu GC9A
Injector/detector block: 300°C, Manual injections with solvent flush
Column: 30 m x 0.543 mm ID fused silica coated with 1.5 µm DB-1
Carrier gas: nitrogen at 30 mL/min.
Temperature Program: 170°C, 5°C/min. to 230°C, hold 7 min.
Detector: Ni-63 ECD, saturation current : 1 nA, range: 10^1
Integrator:Shimadzu C-R5A Chromatopac, width: 10, slope: 320 (adjust as needed) min. area: 1000, speed: 10, atten: 2^4

4. Data Reduction

The APEG process causes unique problems in the area of data reduction because in many cases some PCB congeners react more rapidly than others. The differences in reaction rate result in a disruption of the usual aroclor peak pattern. Since the samples do not have the usual peak pattern, the normal methods of quantitation by comparison to standard aroclors are not appropriate.

The concentration of PCB in samples is calculated according to the procedure of Webb and McCall as described in the EPA method for PCB analysis (EPA 600/4-81-045). Each PCB peak is treated as a separate compound and is quantified individually. The total PCB concentration in a sample is the sum of the concentrations represented by the various peaks. This method provides a much more accurate estimate of the PCB concentrations in treated samples. It also provides accurate results for aroclors and mixtures of aroclors.

Chromatograms of samples and standards are studied and peak names, based on the relative retention times (RRT's) given in the EPA method, are assigned. Peaks 11 and 14 represent monochlorobiphenyls, peaks 16 and 21 represent dichlorobiphenyls and so on.

The standards used for calculations are hexane solutions of a 1:1 mixture of aroclors 1242 and 1260 at various concentrations. The nanograms of PCB represented by a given peak (ngix) within a standard is calculated as follows.

$$ngix = Cx * Vl * (Mia + Mib)/200$$

where Cx = the total PCB concentration of standard x in ng/µL,
Vl = the injection volume of the standard in µL, and
Mia = the mean weight percent of peak i in Aroclor 1242, taken from Table 3 of the EPA method.
Mib = the mean weight percent of peak i in Aroclor 1260, taken from Table 6 of the EPA method

The nanograms of PCB represented by each peak in a sample is calculated by linear interpolation between two standards having the same peak at higher and lower concentrations. The equation for this calculation is:

$$ng_{is} = ng_{ih} - [(A_{ih}-A_{il})(ng_{ih}-ng_{il})/(A_{ih}-A_{is})]$$

where i refers to a peak name,
s refers to the sample,
h refers to the higher standard,
l refers to the lower standard, and
A is a peak area.

The total nanograms of PCB in a sample injection, (ng_t) is the sum of the nanograms of the individual PCBs (ng_{is}).

$$ng_t = \Sigma\, ng_{is}.$$

The PCB concentration in the soil is calculated from the nanograms in the sample injection as follows:

$$C = ng_t * V * D/(VJ * W)$$

where C = the concentration of PCB in soil in mg/kg (ppm)
V = the total sample extract volume
W = the weight of soil, in grams and
D = any additional dilution factor required, e.g. if a 1/100 dilution of the extract is injected, D=100

In order to speed up calculations without requiring a large computer, a spreadsheet program was developed for doing the Webb McCall calculations on a Macintosh 540K microcomputer using a spreadsheet. After peak identities are assigned by trained personnel, peak areas and concentrations of standards and dilution information for each sample are typed into the spreadsheet. The spreadsheet is programmed to calculate ng injected for each PCB peak by linear interpolation, to add up the total ng of PCB injected, to correct for the injection volume, sample dilution, extract volume and sample weight, and to report concentrations in ppm by weight in the soil. The instructions used for training analysts to identify peaks and use the spreadsheet are attached.

When rapid sample turn around is critical, as in process monitoring, it is not possible to obtain dry soil weights for the individual samples in the required time. In order to supply useful data as quickly as possible, half of the wet soil weight (after washing) is used to approximate the dry soil weight, and the data are reported verbally as approximate. GRC's experience has shown that half of the wet soil weight is a good apporoximation of the dry soil weight in this analysis. The soil samples are dried after extraction and re-weighed to obtain the dry soil weight before final data are reported.

How to Assign Peak Names to PCB Chromatograms

1. Assign Names to Standard Peaks

In assigning peak names to standards, it is important to look at the peak pattern. Standards have a fixed number of compounds in fixed concentration ratios. Retention times on the various GCs are not consistent, but the standard peak pattern is.

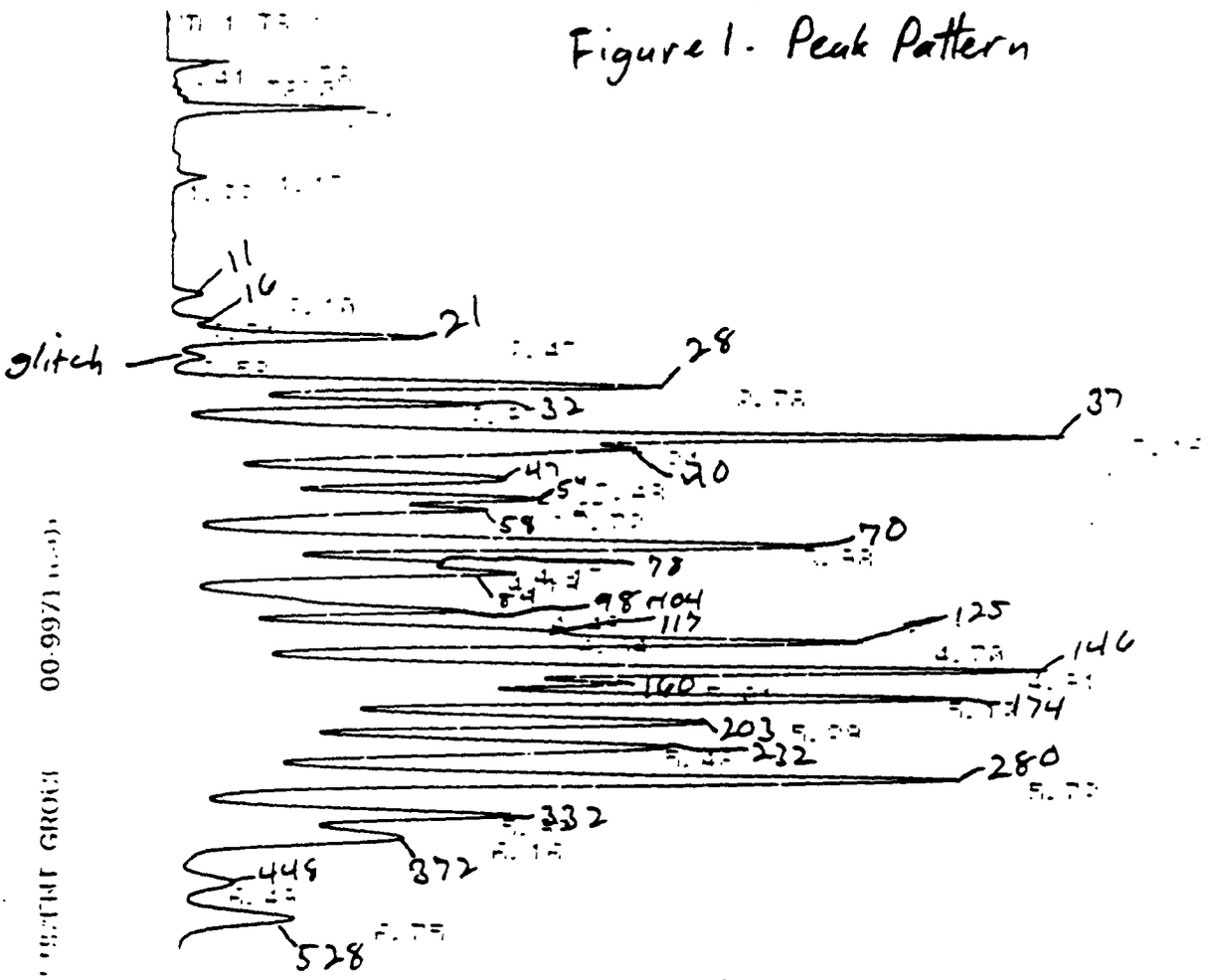
Figure 1 is a picture of a typical 1:1 1242:1260 standard with the peaks marked. This sample was run on the Tracor GC with the Spectra Physics integrator. Notice that there is a un-named peak between peaks 21 and 28. In this particular standard, peak 78 is slightly separated from peak 84 and peak 117 is slightly separated from peak 125. That's OK, it will simply be necessary to add up the two areas in those peak groups when typing them into the spreadsheet. Sometimes, peaks do not separate as well as they are in this chromatogram. It is not unusual for peak 16 to become a shoulder of peak 21 or for peak 160 to be lost between peaks 146 and 174. In those cases, the samples will not separate any better than the standards and peak groups can be used for calculation purposes. Small peaks such as #16 and #160 make a small contribution to the total PCB concentration. Errors in calculating their concentrations will not add significantly to the total error involved in this analysis.

When the Spectra Physics integrator is used it is sometimes difficult to tell which peak a retention time (printed on the chromatogram) is referring to. Remember that this integrator must decide that a peak has passed its maximum before it can assign a retention time. A line that just skims the top of the numbers should intersect the downward slope of the peak it refers to. Figure 2 is a picture of the same chromatogram with the retention times labeled. Figure 3 is the same chromatogram that you can copy and practice on.

2. Assign Names to Sample Peaks

Peak retention times on the Hewlett Packard and Shimadzu GCs are relatively stable throughout a given day. The retention times on the Tracor GC are not particularly stable. There may be as much as half a minute differences. When analyzing reaction samples, start with the initial samples which have the least peak pattern disruption and try to follow the changes in the peak pattern through the reaction. That way, when you get down to the last three or four peaks, you will be more likely to identify them correctly. Pattern is slightly more reliable than retention time for the Tracor. Retention time is more reliable for the other GCs. When you have finished a group of samples from a single reaction, check the sequence and make sure that peaks have not disappeared and then re-appeared due to incorrect identification.

Figure 1. Peak Pattern

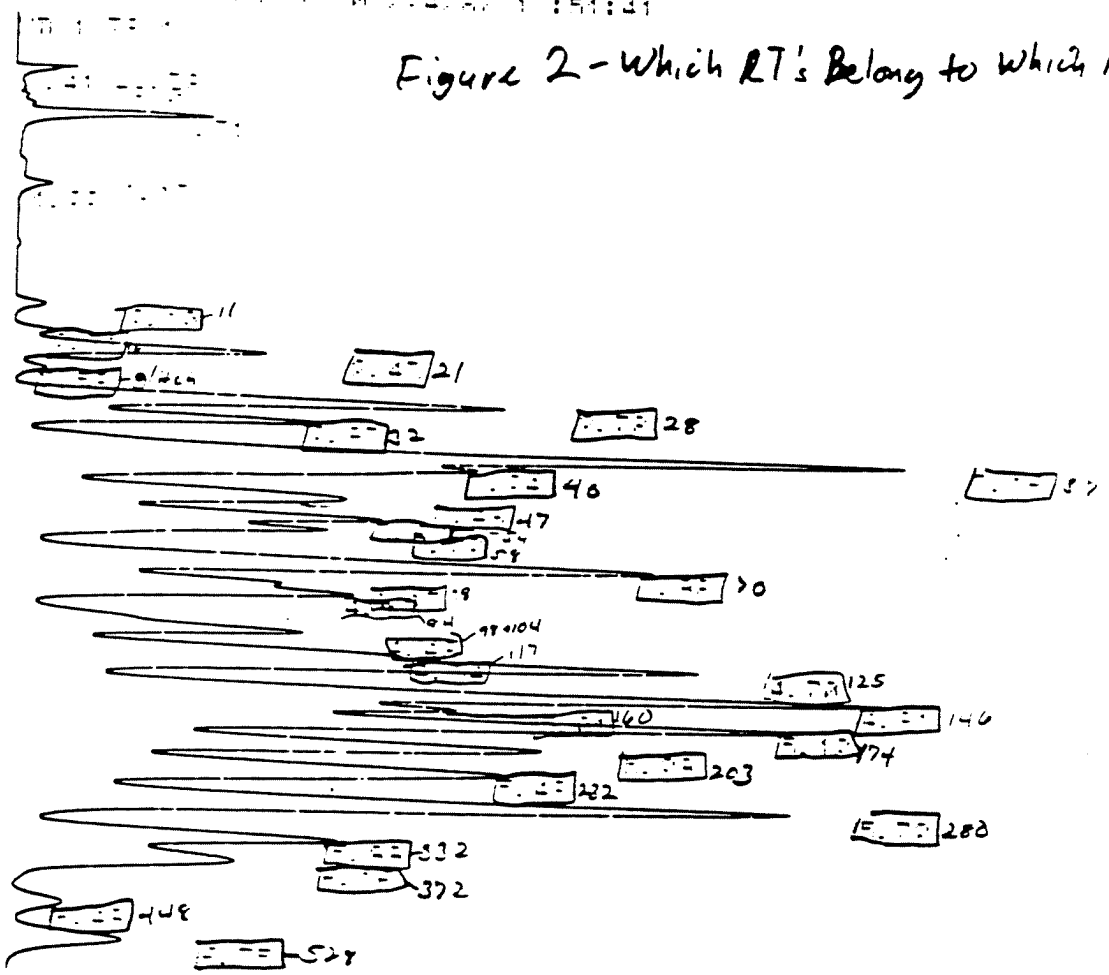


TEST AT 12/07 13:51:41 CH= "C"

FILE 1. METHOD A. RUN 28 INDEX 28

PEAK#	AREA%	RT	AREA BC
1	0.107	0.38	414 B2
2	0.098	0.41	376 B2
3	0.657	0.71	2596 B1
4	0.079	1.13	302 B2
5	0.211	1.22	813 B2
6	0.161	2.1	619 B1
7	0.185	2.31	714 B2
8	1.833	2.42	7064 B2
9	0.187	2.58	707 B2
10	3.758	2.78	14479 B2
11	2.17	2.93	8369 B2
12	7.718	3.14	29207 B2
13	4.711	3.34	18158 B2
14	3.895	3.48	15014 B2
15	2.971	3.63	11457 B2
16	2.513	3.79	10093 B2
17	8.497	3.96	34679 B2
18	1.858	4.17	7159 B2

Figure 2 - Which RT's Belong to Which Peak

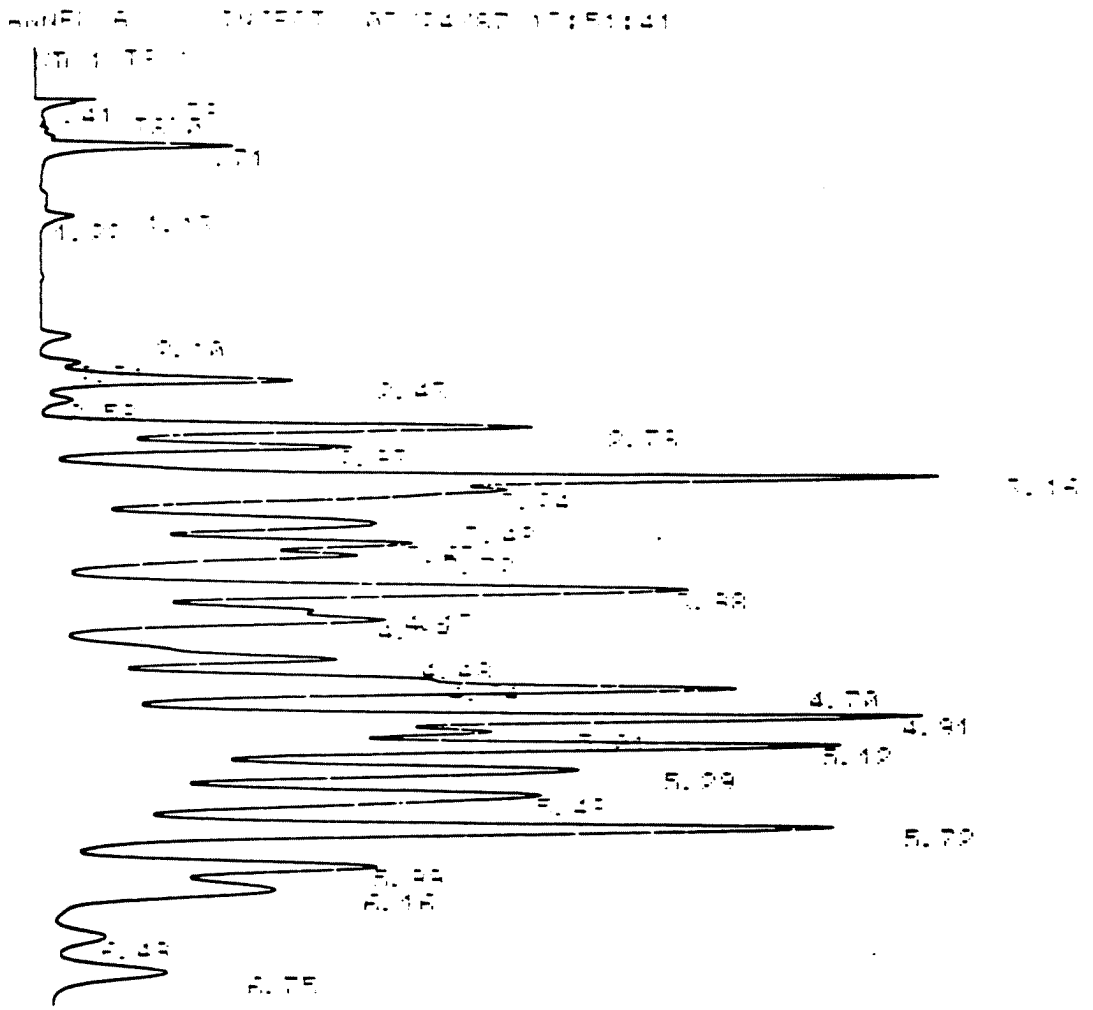


TEST 03/04/87 13:51:41 CHR= "S"

FILE 1. METHOD A. RUN 78 INDEX 78

PEAK#	AREA%	RT	NAME	AREA RD
1	A.187	A.38		414 R2
2	A.898	A.41		378 R2
3	A.933	A.71		2596 R1
4	A.879	1.13		382 R2
5	A.211	1.29		813 R2
6	A.161	2.1	#11	619 R1
7	A.185	2.31	#16	714 R2
8	1.833	2.43	#21	7864 R2
9	A.183	2.58	glitch	787 R2
10	3.798	2.78	28	14679 R2
11	2.13	2.93	32	8289 R2
12	7.318	3.1537		28287 R2
13	4.711	3.3440		18158 R2
14	4.115	3.4847		15814 R2
15	2.571	3.6754		11453 R2
16	2.519	3.7258		10893 R2
17	A.487	3.8870		24279 R2
18	1.352	4.1799		7152 R2

Figure3- Copy for practice



TST 07/24/87 17:51:41 CH= "A" PF= 1.

TIF 1. METHOD A. RUN 70 INDEX 70

PAK#	AREAX	RT	AREA BC
1	0.107	0.38	414 B2
2	0.068	0.41	276 B3
3	0.933	0.71	3596 B1
4	0.079	1.13	303 B2
5	0.211	1.22	813 B3
6	0.161	2.1	619 B1
7	0.185	2.31	714 B2
8	1.823	2.43	7064 B2
9	0.157	2.58	787 B3
10	3.758	2.78	14579 B2
11	2.13	2.93	8299 B2
12	7.718	3.16	29987 B2
13	4.711	3.34	18158 B2
14	3.955	3.48	15814 B2
15	2.871	3.63	11453 B2

How to Webb-McCall

After peak names have been assigned to the peaks in both sample and standard chromatograms, we need to use the peak names and areas to calculate the PCB content of the samples.

The APEG process causes unique problems in the area of data reduction because in many cases some PCB congeners react more rapidly than others. The differences in reaction rate result in a disruption of the usual aroclor peak pattern. Since the samples do not have the usual peak pattern, the normal methods of quantitation by comparison to standard aroclors are not appropriate.

The concentration of PCB in samples is calculated according to the procedure of Webb and McCall as described in the EPA method for PCB analysis (ref.1). Each PCB peak is treated as a separate compound and is quantified individually. The total PCB concentration in a sample is the sum of the concentrations represented by the various peaks. This method provides a much more accurate estimate of the PCB concentrations in treated samples. It also provides accurate results for aroclors and mixtures of aroclors.

As a general rule for gas chromatography, standards should bracket samples. In other words, it is best to have a standard of higher concentration and a standard of lower concentration for calculation of sample concentration. That way the sample concentration can be calculated by linear interpolation, which will greatly reduce inaccuracy caused by nonlinear detector response. The nanograms (ng) of PCB represented by each peak in a sample is calculated by linear interpolation between two standards having the same peak at higher and lower concentrations. The equation for this calculation is:

$$ng_{is} = ng_{ih} - [(A_{ih}-A_{il})(ng_{ih}-ng_{il})/(A_{ih}-A_{is})]$$

where i refers to a peak name,
s refers to the sample,
h refers to the higher standard,
l refers to the lower standard, and
A is a peak area.

The nanograms of PCB represented by a given peak (ng_i) within our 1:1 1242:1260 standard is calculated as follows.

$$ng_{ix} = C_x * V_I * M_i/100$$

where C_x is the concentration of Aroclor in standard x in ng/µL,
V_I is the injection volume of the standard in µL, and
M_i is the mean weight percent of peak i in the standard, given in the table below.

Composition of a 1:1 Mixture of Aroclors 1242 and 1260

Peak	Percent	Peak	Percent	Peak	Percent
11	0.55	58	2.8	203	4.65
16	1.45	70	6.5	232+244	4.9
21	5.65	78	1.8	280	5.5
28	5.5	84	3.7	332	2.1
32	3.05	98+104	3.45	372	2
37	5.75	117+125	7.8	448	0.3
40	5.55	146	7.55	528	0.75
47	4.4	160	2.45		
54	3.4	174	6.2	Total	97.75

(These percentages were obtained by adding up the Aroclor 1242 and 1260 percentages, taken from Tables 3 and 6 of the EPA method 600/4-81-045 and dividing by 2.)

The PCB concentration in the sample (C) is calculated from the nanograms of each peak injected, the injection volume, and the dilution factors involved.

$$C = \Sigma\, ng_{is} * V * D / V_J * W$$

where C is the concentration of PCB in the soil in mg/kg (ppm),
V_J is the sample injection volume,
V is the total sample extract volume,
W is the weight of soil extracted in grams and
D is any additional dilution factor required.

Usually, we inject only one dilution of a sample, but sometimes there is a need to use 2 dilutions and calculate accordingly.

Not surprisingly, manual Webb-McCall calculations are extremely slow and tedious. To speed up the calculations, a computer program has been created by writing the various equations into a Microsoft Multiplan spreadsheet. The Multiplan file is named "WM mix." It is designed to do the Webb-McCall calculations for PCBs in soil, but can easily be adjusted for oil or other matrices.

Master copies of this spreadsheet are in the Utilities files on Roger Gall's hard disk and on Edwina Milicic's XL hard disk. (There is also a backup copy on Edwina's Multiplan application disk.) Before entering any data into the spreadsheet, make a copy of it by highlighting the icon and selecting "duplicate" under the file menu. Then click into the title line of the duplicate icon and rename the duplicate 'WM mm/dd" using the month (mm), and date (dd) that the samples were injected. Open the new file you have created, select "page setup" and type "samples injected mm/dd/yy" into the footer space. This way, there will always be a blank copy of the spreadsheet to use as a template for new batches of samples and it will be easy to associate file copies of the spreadsheet with the right chromatograms.

The file copy consists of 28 rows and 10 columns. This size is enough for all calculations for three standards (1:1 mixtures of aroclor 1242 and aroclor 1260) and two samples. The number of standards is fixed, but the number of samples can be adjusted as needed. The first page of the spreadsheet (without input data) is shown in illustration 1.

The first seven columns are dedicated to standards. The analyst types in the concentration (in ng/ul), the injection volume (in ul) and peak areas for the various peaks for all three standards. The program calculates ng/injection for each peak and displays them is columns 3, 5, and 7. Illustration 2 shows the first three columns with equations for calculating ng/injection. The first factor in all the calculations (in column 3) refers to the concentration of the standard. The second factor refers to the injection volume and the third factor is the decimal fraction of the aroclor accounted for by the peak in question (taken from the EPA method).

Sample calculations take up 3 columns per sample. To describe the calculations, columns 8, 9, and 10 will be discussed. These three columns, with their equations, have been printed in illustration 3. Column 8 is where the peak areas for the sample are keyed in. The peak ID for any row is fixed and is given in column 1 of the spreadsheet. There are spaces at the top of column 9 for the sample identification number, dilution used and the injection volume. "Dilution used" refers to how much the concentrated soil extract was diluted before injection. This is placed at the top of the spreadsheet because it is most commonly written on the chromatogram, while the mass of soil extracted (row 33) and the volume of the concentrated extract (row 34) are recorded in the analyst notebook, but not written on the chromatogram. When entering the dilution used it is necessary to use an equals sign; e.g. for a 1/25 dilution, the analyst types "=1/25 [return]" and the value "0.04" appears on the screen.

Rows 5-16 and 21-34 are where the ng/injection values for the sample are calculated. The equations are too long to be printed out in spreadsheet form. Illustration 3 contains only the first 40 characters of the equations, but illustration 3 does show that the equations are alike except for the row number used. Row numbers in this part of the spreadsheet are associated with peak ID numbers. For example, row 21 is associated with peak #160. The complete equations for peak 11 (row 5) and peak 528 (row 29) are given below.

Column 9, row 5 =IF(RC[-1]>R5C6,R5C7+(R5C7-R5C5)*(RC[-1]-R5C6)/(R5C6-R5C4), IF(RC[-1]>R5C4,(R5C7-R5C5)*(RC[-1]-R5C4)/(R5C6-R5C4)+R5C5, IF(RC[-1]>R5C2,(R5C5-R5C3)*(RC[-1]-R5C2)/(R5C4-R5C2)+R5C3, R5C3*RC[-1]/R5C2)))

Column 9, row 29 =IF(RC[-1]>R29C6,R29C7+(R29C7-R29C5)*(RC[-1]-R29C6)/(R29C6-R29C4),IF(RC[-1]>R29C4,(R29C7-R29C5)*(RC[-1]-R29C4)/(R29C6-R29C4)+R29C5, IF(RC[-1]>R29C2,(R29C5-R29C3)*(RC[-1]-R29C2)/(R29C4-R29C2)+R29C3, R29C3*RC[-1]/R29C2)))

The logic behind these equations is "If x, Then y, Else z." The "If" statements are nested so that the directions given to the computer by these equations are as follows: If the peak area of this sample for this peak ID (same row, one column back) is greater than the peak area of the high standard for this peak ID, then calculate by linear extrapolation from the high and middle standards. If the peak area is greater than that of the middle standard (and less than or equal to that of the high standard), calculate by linear interpolation between the middle and high standards. If the peak area is greater than that of the low standard (and less than or equal to that of the middle standard), calculate by linear interpolation between the low and middle standards. Otherwise, (i.e. if the peak area is less than that of the low standard), calculate by proportion to the low standard.

Row 31 is where all the peak contributions are added up. That equation is also too long to be printed by the spreadsheet and is given below.

Column 9, row 31= R[-26]C+R[-25]C+R[-24]C+R[-23]C+R[-22]C+R[-21]C +R[-20]C +R[-19]C+R[-18]C+R[-17]C+R[-16]C+R[-15]C+R[-14]C+R[-13]C +R[-12]C+R[-11]C+R[-10]C+R[-9]C+R[-8]C+R[-7]C+R[-6]C+R[-5]C+R[-4]C +R[-3]C+R[-2]C

Row 36 of column 9 is where the final soil concentration in soil is calculated. The equation for this calculation is;

$$\text{ppm in soil} = \frac{\text{total ng/injection x extract volume}}{\text{injection volume x soil mass x dilution}}$$

The purpose of column 10 is to indicate how the ng/injection for each peak was calculated. If the peak area is lower than that of the lowest standard for that peak, a "0" will be printed in column 10. If the peak area is higher than that of the highest standard, a "2" will be printed. If the peak area is within the range of the standards, a "1" will be printed. Samples with more than one or two 2's should be diluted further and re-injected. Samples with 0's that account for more than 25% of the total ng should be injected in a more concentrated form unless the soil concentration is below the desired detection limit.

Columns 8, 9, and 10 can be copied and pasted on at the end of the spreadsheet to make room for the number of samples to be calculated. References to the peak and ng/injection values for the standards are references to fixed locations on the spreadhseet so that correct equations will be used at any location in the spreadsheet. Before entering data, copy and paste enough sets of sample columns for the number of samples in the set. If there are more than 17 samples, you will have to split them into two spreadsheets, Multiplan will not accomodate more than 63 columns in this program. When splitting a large group of samples, you can save some time by copying the standard section and pasting it into the second spreadsheet. The formulas will not transfer, but once the calculations for the ng/injection are done correctly, the formulas are not needed. Copy only the active area (R1C1 - R29C7), not the whole columns; otherwise the clipboard will be too large.

Save the file frequently during work sessions so that, in the event of a system crash or lockup, you will not have to start from scratch. The master copies are set on "manual calculation" so that the computer will not recalculate the entire spreadsheet after each peak area is entered, but it will calculate the entire spreadsheet whenever it is saved. One save per chromatogram is usually enough.

After you are finished, generate a hard copy of the spreadsheet and file it in the Webb-McCall notebook above Edwina's desk. Also make sure that the date is clearly marked on the chromatograms, and file the chromatograms so that they can be located easily in the future. Your notebook should contain sample weights, extract volumes (at least one per page if many samples were done the same way) GC conditions (or at least reference to standard GC conditons), results in ppm PCB and reference to the chromatogram date and the calculation file. All of this information is very important for our QA program and for our ability to figure out what was done and how it worked out when we look at the notebook five years from now.

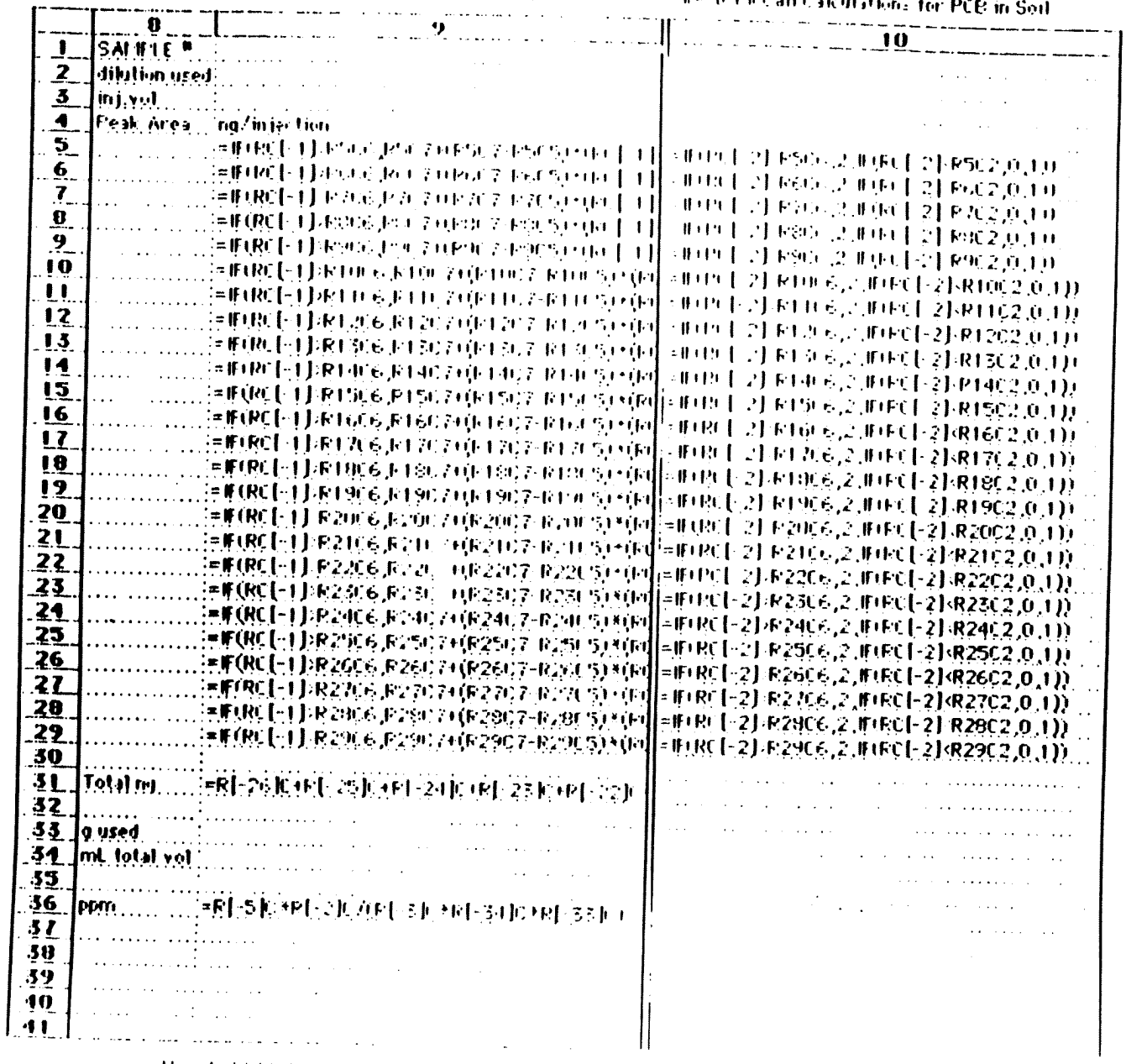
Webb McCall Calculations for PCB in Soil

	1	2	3	4	5	6	7	8	9	10	11	12	13
1	STANDARDS (1:1 mixtures of aroclors 1242 and 1260)							SAMPLE #			SAMPLE #		
2	Total PCB Conc (ng/ul)							dilution used			dilution used		
3	inj. vol							inj vol			inj vol		
4	peak #	Peak Area	ng/injection	Peak Area	ng/injection	Peak Area	ng/injection	Peak Area	ng/injection		Peak Area	ng/injection	
5	11		0		0		0		#DIV/0!			#DIV/0!	
6	16		0		0		0		#DIV/0!			#DIV/0!	
7	21		0		0		0		#DIV/0!			#DIV/0!	
8	28		0		0		0		#DIV/0!			#DIV/0!	
9	32		0		0		0		#DIV/0!			#DIV/0!	
10	37		0		0		0		#DIV/0!			#DIV/0!	
11	40		0		0		0		#DIV/0!			#DIV/0!	
12	47		0		0		0		#DIV/0!			#DIV/0!	
13	54		0		0		0		#DIV/0!			#DIV/0!	
14	59		0		0		0		#DIV/0!			#DIV/0!	
15	70		0		0		0		#DIV/0!			#DIV/0!	
16	78		0		0		0		#DIV/0!			#DIV/0!	
17	84		0		0		0		#DIV/0!			#DIV/0!	
	98+104		0		0		0		#DIV/0!			#DIV/0!	
	117+125		0		0		0		#DIV/0!			#DIV/0!	
	146		0		0		0		#DIV/0!			#DIV/0!	
21	160		0		0		0		#DIV/0!			#DIV/0!	
22	174		0		0		0		#DIV/0!			#DIV/0!	
23	203		0		0		0		#DIV/0!			#DIV/0!	
24	232+244		0		0		0		#DIV/0!			#DIV/0!	
25	290		0		0		0		#DIV/0!			#DIV/0!	
26	332		0		0		0		#DIV/0!			#DIV/0!	
27	372		0		0		0		#DIV/0!			#DIV/0!	
28	448		0		0		0		#DIV/0!			#DIV/0!	
29	528		0		0		0		#DIV/0!			#DIV/0!	
30													
31								Total ng	#DIV/0!		Total ng	#DIV/0!	
32													
33								g used			g used		
34								mL total vol			mL total vol		
35													
36								ppm	#DIV/0!		ppm	#DIV/0!	
37													
38													
39													
40													
41													

How to Webb McCall Worksheet 1

Worksheet for PCB Calculations for PCB in Soil

	1	2	3
1	STANDARD AROCLOR 1260		
2	Total PCB Conc. (ug/L)		
3	Inj. Vol.		
4	peak ID	Peak Area	ng/injection
5	11		=R[-3]C*R[-2]C*0.0055
6	16		=R[-4]C*R[-3]C*0.0145
7	21		=R[-5]C*R[-4]C*0.0565
8	28		=R[-6]C*R[-5]C*0.055
9	32		=R[-7]C*R[-6]C*0.0305
10	37		=R[-8]C*R[-7]C*0.0575
11	40		=R[-9]C*R[-8]C*0.0555
12	47		=R[-10]C*R[-9]C*0.044
13	54		=R[-11]C*R[-10]C*0.034
14	59		=R[-12]C*R[-11]C*0.029
15	70		=R[-13]C*R[-12]C*0.065
16	78		=R[-14]C*R[-13]C*0.018
17	84		=R[-15]C*R[-14]C*0.037
18	90+94		=R[-16]C*R[-15]C*0.0345
19	117+125		=R[-17]C*R[-16]C*0.078
20	146		=R[-18]C*R[-17]C*0.0755
21	160		=R[-19]C*R[-18]C*0.0245
22	174		=R[-20]C*R[-19]C*0.062
23	203		=R[-21]C*R[-20]C*0.0465
24	232+244		=R[-22]C*R[-21]C*0.049
25	280		=R[-23]C*R[-22]C*0.055
26	332		=R[-24]C*R[-23]C*0.021
27	370		=R[-25]C*R[-24]C*0.02
28	440		=R[-26]C*R[-25]C*0.003
29	528		=R[-27]C*R[-26]C*0.0075
30			
31			
32			
33			
34			
35			
36			
37			
38			
39			
40			
41			



	8	9	10
1	SAMPLE #		
2	dilution used		
3	inj.vol		
4	Peak Area	ng/injection	
5		=IF(RC[-1]<R5C6,R5C7*(R5C7-R5C5)*(RC[-1]	=IF(RC[-2]<R5C6,2,IF(RC[-2]<R5C2,0,1))
6		=IF(RC[-1]<R6C6,R6C7*(R6C7-R6C5)*(RC[-1]	=IF(RC[-2]<R6C6,2,IF(RC[-2]<R6C2,0,1))
7		=IF(RC[-1]<R7C6,R7C7*(R7C7-R7C5)*(RC[-1]	=IF(RC[-2]<R7C6,2,IF(RC[-2]<R7C2,0,1))
8		=IF(RC[-1]<R8C6,R8C7*(R8C7-R8C5)*(RC[-1]	=IF(RC[-2]<R8C6,2,IF(RC[-2]<R8C2,0,1))
9		=IF(RC[-1]<R9C6,R9C7*(R9C7-R9C5)*(RC[-1]	=IF(RC[-2]<R9C6,2,IF(RC[-2]<R9C2,0,1))
10		=IF(RC[-1]<R10C6,R10C7*(R10C7-R10C5)*(R	=IF(RC[-2]<R10C6,2,IF(RC[-2]<R10C2,0,1))
11		=IF(RC[-1]<R11C6,R11C7*(R11C7-R11C5)*(R	=IF(RC[-2]<R11C6,2,IF(RC[-2]<R11C2,0,1))
12		=IF(RC[-1]<R12C6,R12C7*(R12C7-R12C5)*(R	=IF(RC[-2]<R12C6,2,IF(RC[-2]<R12C2,0,1))
13		=IF(RC[-1]<R13C6,R13C7*(R13C7-R13C5)*(R	=IF(RC[-2]<R13C6,2,IF(RC[-2]<R13C2,0,1))
14		=IF(RC[-1]<R14C6,R14C7*(R14C7-R14C5)*(R	=IF(RC[-2]<R14C6,2,IF(RC[-2]<R14C2,0,1))
15		=IF(RC[-1]<R15C6,R15C7*(R15C7-R15C5)*(R	=IF(RC[-2]<R15C6,2,IF(RC[-2]<R15C2,0,1))
16		=IF(RC[-1]<R16C6,R16C7*(R16C7-R16C5)*(R	=IF(RC[-2]<R16C6,2,IF(RC[-2]<R16C2,0,1))
17		=IF(RC[-1]<R17C6,R17C7*(R17C7-R17C5)*(R	=IF(RC[-2]<R17C6,2,IF(RC[-2]<R17C2,0,1))
18		=IF(RC[-1]<R18C6,R18C7*(R18C7-R18C5)*(R	=IF(RC[-2]<R18C6,2,IF(RC[-2]<R18C2,0,1))
19		=IF(RC[-1]<R19C6,R19C7*(R19C7-R19C5)*(R	=IF(RC[-2]<R19C6,2,IF(RC[-2]<R19C2,0,1))
20		=IF(RC[-1]<R20C6,R20C7*(R20C7-R20C5)*(R	=IF(RC[-2]<R20C6,2,IF(RC[-2]<R20C2,0,1))
21		=IF(RC[-1]<R21C6,R21C7*(R21C7-R21C5)*(R	=IF(RC[-2]<R21C6,2,IF(RC[-2]<R21C2,0,1))
22		=IF(RC[-1]<R22C6,R22C7*(R22C7-R22C5)*(R	=IF(RC[-2]<R22C6,2,IF(RC[-2]<R22C2,0,1))
23		=IF(RC[-1]<R23C6,R23C7*(R23C7-R23C5)*(R	=IF(RC[-2]<R23C6,2,IF(RC[-2]<R23C2,0,1))
24		=IF(RC[-1]<R24C6,R24C7*(R24C7-R24C5)*(R	=IF(RC[-2]<R24C6,2,IF(RC[-2]<R24C2,0,1))
25		=IF(RC[-1]<R25C6,R25C7*(R25C7-R25C5)*(R	=IF(RC[-2]<R25C6,2,IF(RC[-2]<R25C2,0,1))
26		=IF(RC[-1]<R26C6,R26C7*(R26C7-R26C5)*(R	=IF(RC[-2]<R26C6,2,IF(RC[-2]<R26C2,0,1))
27		=IF(RC[-1]<R27C6,R27C7*(R27C7-R27C5)*(R	=IF(RC[-2]<R27C6,2,IF(RC[-2]<R27C2,0,1))
28		=IF(RC[-1]<R28C6,R28C7*(R28C7-R28C5)*(R	=IF(RC[-2]<R28C6,2,IF(RC[-2]<R28C2,0,1))
29		=IF(RC[-1]<R29C6,R29C7*(R29C7-R29C5)*(R	=IF(RC[-2]<R29C6,2,IF(RC[-2]<R29C2,0,1))
30			
31	Total ng	=R[-26]C+R[-25]C+R[-24]C+R[-23]C+R[-22]	
32			
33	g used		
34	mL total vol		
35			
36	ppm	=R[-5]C*R[-2]C/R[-3]C*R[-30]C*R[-33]C	
37			
38			
39			
40			
41			

Analytical Procedures for PCB in Reagent

GRC's method for analyzing reagent for PCB is similar to the method used for soil. A sample of reagent is weighed accurately, diluted with enough 1:4 aqueous methanol to reduce the viscosity roughly to that of water. The reagent solution is extracted three times with a volume of hexane roughly equal to the volume of the reagent solution. After each extraction, the hexane layer is transferred to a volumetric flask or graduated receiver. After the third extraction, the hexane extract is brought up to a known volume. The sample is cleaned up by shaking 2 mL of the hexane extract briefly with 1 mL of concentrated sulfuric acid. The extract is then injected into a GC with ECD.

The chromatographic technique for the hexane solution was the same as for the hexane extract from a soil sample. PCB concentration is calculated as described for soil samples.

Analytical Procedure for PCB in Wash Water

Wash water from the APEG process is difficult to extract with organic solvents such as methylene chloride because residual PEG and TMH in the wash water act as surfactants and cause formation of emulsions. For that reason, solid phase extraction is used.

A disposable 6 ml octyl (C8) solid phase extraction column (Baker #7087-6 or equivalent) is connected to a 250 mL filtering flask or to the SPE system manifold (Baker #7018-0 or equivalent). The column is conditioned with two column volumes of methanol followed by one column volume of deionized water. The column is not permitted to run dry during conditioning.

A 75 mL reservoir and a filtration column are attached to the conditioned column. Two 50 ml aliquots of the sample (washwater) are pipetted into the reservoir and drawn through the column. The entire column, including the reservoir is rinsed with two 3 mL portions of deionized water. The reservoir and filtration column are removed and the C8 column is allowed to dry with the vacuum on for at least 10 minutes.

When the column is dry, a receiver is positioned at the end of the column. The column is then eluted with three, approximately 0.5 ml aliquots of hexane. The column is allowed to dry for two to three minutes and the receiver is removed. The hexane solution in the receiver is cleaned up by shaking briefly with 0.5 mL of concentrated sulfuric acid. When the phases have separated, the hexane layer is transferred to a small vial with a teflon lined screw cap. The receiver and acid layer are rinsed with two 0.5 mL aliquots of hexane, which are added to the vial. The solution in the vial is reduced to incipient dryness under a gentle stream of nitrogen. The contents of the vial are then dissolved in 0.5 mL of hexane and analyzed by GC as described for soil extracts.

Analytical Procedure for PCB in Process Distillates

In most cases, solid phase extraction cannot be used to extract process distillates because there is water insoluble phase that would overload the extraction column. In lab scale reactions, PCBs tend to adsorb onto glassware rather than remain suspended in water. Therefore, it is necessary to extract the entire amount of distillate produced in a lab reaction and rinse the condensate receiver with the extraction solvent to assure complete recovery of the PCBs.

The distillate is transferred to the appropriately sized separatory funnel. Methylene chloride is used to rinse the distillate receiver and the rinse is added to the same separatory funnel. The distillate is extracted three times with portions of methylene chloride about one fifth the volume of the distillate. The extracts are collected in a Kuderna Danish apparatus and are concentrated to a final volume of 5-10 mL with solvent exchange to hexane. The hexane extract is cleaned up and analyzed in the same way as a hexane extract from soil.

In pilot or full scale operations, there is enough distillate for the two liquid phases to be analyzed separately. A sample of the water layer is collected carefully, to avoid contamination by the organic layer, and placed in a glass bottle. The water is analyzed according to the procedure for distillates, given above. The organic layer is analyzed by weighing a small portion of the organic layer and diluting to a known volume with hexane. The hexane solution is cleaned up and analyzed in the same way as a hexane extract from soil.

Analytical Procedure for KOH in Liquid Samples

A portion of sample is weighed in a glass vessel. If the sample is opaque, it may be diluted with distilled or deionized water. A few drops of phenolphthalein indicator are added. The sample is titrated with acid solution of known concentration (expressed as Normality) until the pink color is discharged. If the sample is highly colored, a pH meter may be used to monitor the titration and the sample is titrated to a pH of 8.2. The concentration of KOH in the sample is calculated as follows.

mg KOH/g sample = mL titrant x titrant normality x 56 / sample weight (g)

Analysis of Reagent and Washes for PEG, TMH, DMSO and SFLN

HPLC Set-up

Any HPLC system equivalent to the following may be used, provided that its performance is checked.

Mainframe: Hewlett Packard 1090L
Automatic injector capable of 1-25 ul injections (usually set at 10 ul)
Mobile Phase: 10% methanol in HPLC grade water (isocratic)
Flow rate: 2 ml/min, Max Pressure set at 400 bar
Guard Column: 7 cm x 2 mm ID stainless steel packed with 37-53 µm silica gel (Whatman 4390-411 or equivalent) frits and packing changed when pressure exceeds 200 bar.
Analytical column: 25 cm x 4.6 mm ID stainless steel packed with 10 µm silica gel (Whatman 4216-001 or equivalent)
Detector: Hewlett Packard 1037A refractive index detector, set at 30°C
Integrator: Hewlett Packard 3393A, atten: 2^6, area reject: 10000, peak width: 0.10, Threshold 3, chart speed: 1 cm/min, zero at 10% of full scale, Mode: peak height, Events: baseline hold 0.5 - 3.8 min, baseline next valley at 3/8 min, baseline all valleys after 5 min, stop at 20 min.

Turn the detector power on and allow it to warm up for two hours (total) before injecting samples. During this time, purge the mobile phase with helium for 15 minutes to remove traces of air, turn on mainframe power, allow the instrument to go through its self test program and pump mobile phase through the columns for 30 minutes to assure good equilibration. Check the "zero" frequently throughout the day and adjust it as necessary so that it is between -2 and 2 at all times.

Sample Preparation:

Mix the samples well. Weigh 1-2 g of reagent or 5-6 g of wash water into a centrifuge tube with a teflon lined screw cap. Add deionized water to the reagent samples so that the volume is 5-6 mL and mix on the vortex mixer. Bring the pH to <8 by dropwise addition of 25% sulfuric acid, using Hydrion paper to check the pH. Bring the sample volume up to 10 mL with deionized water. Mix on the vortex mixer. Record the sample weight and solution volume (10 mL) in your notebook. The pH adjustment may produce copious quantities of precipitate. Use the centrifuge to settle the precipitate and filter about 2 mL of the liquid using a filter syringe (Lid-X/AQOR .45 or equivalent). Pour the filtered sample into an auto injector vial and seal it with a crimp cap.

Standard Preparation

Use disposable microbore pipettes to measure the desired quantities of the analytes you need into 10 mL volumetric flasks and bring them up to the mark with deionized water. The following table lists volumes of analytes and

the concentrations they will produce in the volumetric flasks. You may mix two glycols and one sulfoxide together into mixed standards, the HPLC will separate them and you will need fewer injections. It is recommended that the mixed standard be kept in the 1:1:2 PEG:TMH:sulfoxide ratio anticipated for the samples. That way any cross interference will be cancelled out.

Table of Concentrations for Standards in 10 mL Flasks

	Standard #1		Standard #2		#3**
Analyte*	µl used	mg/ml	µl used	mg/ml	mg/ml
PEG	100	11.25	50	5.625	1.13
TMH	100	10.54	50	5.27	1.05
DMSO	200	22.02	100	11.01	2.20
SFLN	200	25.22	100	12.61	2.52

*listed in reverse order of elution from HPLC
**standard #3 is a 1/10 dilution of standard #1
This table assumes that analytes are at room temp (25°C)

Inject samples and standards into the HPLC. Write the injection volume and the sample identification number or standard identification number on the chromatogram. If a sample produces a peak area higher than that of the most concentrated standard, it is necessary to use a smaller injection volume. If that doesn't bring the peak area low enough, dilute the sample. Use a 1 mL disposable pipette to measure 0.5 or 1 mL of sample solution into a 10 mL test tube and bring the volume up to 3-10 mL as desired. Record all dilution volumes in your notebook.

Data Reduction

A copy of a standard chromatogram with peaks marked is attached to this method. DMSO and Sulfolane produce only one peak each. TMH produces 2 peaks, but only the largest one is used for quantitation. PEG produces a series of peaks. The heights of the four largest peaks are summed and used for quantitation. These four peaks are marked in the chromatogram attached to this method.

The number of micrograms of analyte "i" in an injection is calculated from the concentration of that analyte in the standard and the injection volume as follows. (Remember that mg/mL = ug/uL)

$$ug_{is} = C_{is} * V_{js}$$

where ug_{is} = the micrograms of analyte "i" in standard injection
C_{is} = the concentration of "i" in the standard (in mg/mL)
V_{js} = the injection volume for the standard.

The micrograms of analyte "i" in a sample injection is calculated by linear interpolation between standards of higher and lower concentration. The equation for this calculation

$$ug_{ix} = ug_{ih} - [(H_{ih}-H_{il})(ug_{ih}-ug_{il})/(H_{ih}-H_{ix})]$$

where i refers to a peak name,
x refers to the sample,
h refers to the higher standard,
l refers to the lower standard, and
H is a peak height.

The peaks generated in this method are broad and the integrator baseline correction is not consistent. Therefore integrated peak areas vary randomly and produce inconsistent results. Peak height was found to produce more reliable results. Therefore peak height is used instead of peak area for this analysis. Since peak height is a function of the voltage difference between the "zero" voltage and the voltage at the tip of the peak, it is very important that the detector "zero" be checked frequently throughout the day and adjusted as required.

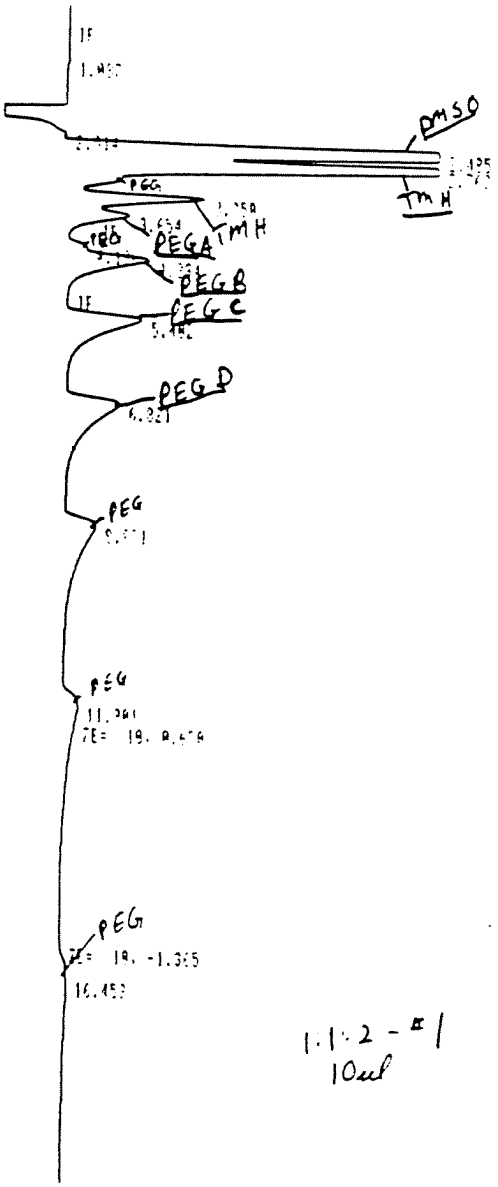
The concentration of analyte "i" in the reagent is calculated from the micrograms in the injection, the sample injection volume, the sample solution volume (usually 10 mL), and the sample weight. It is most useful to report concentrations in mg analyte per gram of reagent or wash water. The equation for that is given below.

$$\text{"i" (mg/g)} = (ug_{ix} * V)/(V_{jx} * W)$$

where V is the sample solution volume in mL (usually 10 mL)
V_{jx} is the sample injection volume and
W is the sample weight in grams.

A spreadsheet program which does these calculations automatically has been developed using Microsoft Excel. The program is quite similar to the Webb McCall spreadsheet used to calculate PCB concentrations in soil samples. A copy of the first page of a blank calculation file is attached.

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	STANDARDS							SAMPLE #			SAMPLE #		
2													
3	Standard Number		3		2		1	dilution used			dilution used		
4	Inj. Vol.		10		10		10	inj.vol			inj.vol		
5	compound	Peak Height	µg/injection	Peak Height	µg/injection	Peak Height	µg/injection	Peak Height	µg/injection	Peak Area	µg/injection		
6	Sulfolane		25.22		126.1		252.2		#DIV/0!	1	#DIV/0!	1	
7	DMSO		22.02		110.1		220.2		#DIV/0!	1	#DIV/0!	1	
8	TMH		10.54		52.27		105.4		#DIV/0!	1	#DIV/0!	1	
9	PEGa		NA		NA		NA						
10	PEGb		NA		NA		NA						
11	PEGc		NA		NA		NA						
12	PEGd		NA		NA		NA						
13	PEG SUM	0	11.25	0	56.25	0	112.5	0	#DIV/0!	1	0	#DIV/0!	1
14													
15							g used				g used		
16							mL prep vol				mL prep vol		
17													
18								compound	#amp mg/g		compound	#amp mg/g	
19								SFLN	#DIV/0!		SFLN	#DIV/0!	
20								DMSO	#DIV/0!		DMSO	#DIV/0!	
21								TMH	#DIV/0!		TMH	#DIV/0!	
22		Approximate detection limits: PEG: 10 mg/g, TMH: 2 mg/g, DMSO: 2 mg/g.						PEG	#DIV/0!		PEG	#DIV/0!	



1.1.2 - #1
10ul

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PE./TMH/DMSO ANALYSIS

HE.GHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
2.314	84327	BH	.252	1.34967
2.485	3855050	SHH	.116	61.70080
2.763	1089260	SHH	.168	17.43381
3.350	199417	VH	.210	3.19171
3.654	162721	HH	.356	2.60438
4.196	109674	HH	.194	1.75535
4.394	186578	HH	.516	2.90622
5.492	177792	SHH	.991	2.84559
6.821	77221	BH	1.491	1.23594
8.871	120012	HH	2.242	1.92081
11.981	99687	HH	3.473	1.59533
16.453	86316	I HH	4.829	1.38158

TOTAL HEIGHT=6247974
MUL FACTOR=1.0000E+00

Appendix 4.

Results of PCB Analysis

PCB Analysis Done for Wide Beach Bench Study

Reaction #1, Soil A

Reactor samples	Description	ppm PCB	Comments
87112501*	Initial	1200	
R70108711300005JRa	1 hour	620	
R70108711300105JRa	2 hours	590	
R70108711300205JRa	3 hours	Not Analyzed	
R70108711300305JRa	4 hours	140	
R70108711300405JRa	5 hours	92	
R70108711300505JR	6 hours	66	
R70108711300605JR	7 hours	38	
R70108711300705JR	8 hours	32	
R70108711300805JR	9 hours	Not Analyzed	
R70108711300905EM	10 hours	32	
R70108711301105EM	12 hours	24	

Final Outputs from reaction 1	Description	ppm PCB	Comments
R70108712020001RJR	Reagent	43	
R70108712020030WJR	Wash 1	1.6	
R70108712020156WJR	Wash 2	0.8	
R70108712020247WJR	Wash 3	0.8	
R70108711301105CEM	Condensate	<18	interference
R70108712040303SRG	Finished soil	<59	Interference

Reaction #2, Soil B

Reactor samples	Description	ppm PCB	Comments
87112502*	Initial	24	
R70108711300005JRb	1 hour	16	
R70108711300105JRb	2 hours	21	
R70108711300205JRb	3 hours	Not Analyzed	
R70108711300305JRb	4 hours	1.8	
R70108711300405JRb	5 hours	0.99	

Final Outputs	Description	ppm PCB	Comments
R70108711300430CJR	Condensate	<42	interference
R70108712020430RJR	Reagent	3.6	
R70108712020520WJR	Wash 1	1.2	
R70108712020555WJR	Wash 2	0.2	
R70108712020647WJR	Wash 3	0.3	
R70108712040308SRG	Finished soil	1.6	

*old sample numbering system used before 11/27

Reaction #3, Soil C, DMSO

Reactor samples	Description	ppm PCB	Comments
R70108712020445SJR	Initial	620	
R70108712022315SJR	1 hour	550	
R70108712030015SJR	2 hours	670	
R70108712030115SJR	3 hours	430	
R70108712030215SJR	4 hours	480	
R70108712030315SJR	5 hours	35	
R70108712030415SJR	6 hours	20	
R70108712030515SJR	7 hours	5.7	
R70108712030615SJR	8 hours	5.7	
R70108712030715SJR	9 hours	5.1	
R70108712030815SJR	10 hours	6.5	

Final Outputs	Description	ppm PCB	Comments
R70108712030815CJR	Condensate	< 24	interference
R70108712040605RRG	Reagent	1.5	
R70108712040640WJR	Wash 1	0.7	
R70108712040725WJR	Wash 2	0.7	
R70108712040810WJR	Wash 3	0.4	
R70108712062315SRG	Finished soil	6.8	

Reaction #4, Soil C, Sulfolane

Reactor samples	Description	ppm PCB	Comments
R70108712020448SJR	Initial	490	
R70108712022318SJR	1 hour	380	
R70108712030018SJR	2 hours	1260	
R70108712030118SJR	3 hours	760	
R70108712030218SJR	4 hours	460	
R70108712030318SJR	5 hours	59	
R70108712030418SJR	6 hours	42	
R70108712030518SJR	7 hours	65	
R70108712030618SJR	8 hours	2.6	
R70108712030718SJR	9 hours	6.7	
R70108712030818SJR	10 hours	3.8	

Final Outputs	Description	ppm PCB	Comments
R70108712030818CJR	Condensate	<120	Interference
R70108712040845RJR	Reagent	4.2	
R70108712040922WJR	Wash 1	0.3	
R70108712041013WJR	Wash 2	0.2	
R70108712041058WJR	Wash 3	0.5	
R70108712062320SRG	Finished soil	4.2	

Reaction #5, Soil A, 150°C

Reactor samples	Description	ppm PCB	Comments
R70108712062358SJR	Initial	690	
R70108712072315SJR	1 hour	740	
R70108712080015SJR	2 hours	790	
R70108712080115SJR	3 hours	156	
R70108712080215SJR	4 hours	24	
R70108712080315SJR	5 hours	9.8	
R70108712080415SJR	6 hours	8.3	
R70108712080515SJR	7 hours	6.2	
R70108712080615SJR	8 hours	5.6	

Final Outputs	Description	ppm PCB	Comments
R70108712080708CJR	Condensate	<390	interference
R70108712090105RJR	Reagent	3.2	
R70108712090125WJR	Wash 1	0.5	
R70108712090144WJR	Wash 2	0.7	
R70108712090219WJR	Wash 3	0.1	
R70108712090257WJR	Wash 4	1.4	
R70108712090530SJR	Finished soil	<15	interference

Reaction #6, Soil A, 160°C

Reactor samples	Description	ppm PCB	Comments
R70108801140436SJR	Initial	640	
R70108801150000SJR	1 hour	690	
R70108801150100SJR	2 hours	Not Analyzed	
R70108801150200SJR	3 hours	Not Analyzed	
R70108801150300SJR	4 hours	Not Analyzed	
R70108801150400SJR	5 hours	Not Analyzed	
R70108801150500SJR	6 hours	140	
R70108801150600SJR	7 hours	140	
R70108801150700SJR	8 hours	120	
R70108801150800STG	9 hours	63	
R70108801150900STG	10 hours	Not Analyzed	
R70108801151000STG	11 hours	49	
R70108801151100STG	12 hours	71	

Final Outputs	Description	ppm PCB	Comments
R70108801151225CEM	Condensate	<46	interference
R70108801151445REM	Reagent	26	
R70108801151550WEM	Wash 1	38	
R70108801151645WEM	Wash 2	13	
R70108801151705WEM	Wash 3	4	
R70108801201406SRG	Finished soil	<240	Interference

Appendix 5.

Results of Total Organic Carbon Analysis

Adirondack
Environmental Services Inc.

P.O. Box 265 | 298 Riverside Avenue | Rensselaer, NY 12144 | (518) 434-4546

LABORATORY REPORT

for

Galson Research Corporation
6601 Kirkville Road
East Syracuse, NY 13057

Attention: Edwina Milicic

Report date: 02/03/88
Number of samples analyzed: 9
AES Project ID: 880125 D

Adirondack
Environmental Services Inc.

P.O. Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R701087111712I2RP
AES sample #: 880125 D01
Samples taken by: Client
MATRIX: soil
Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	1,140	ug/g	MF	02/01

Adirondack Environmental Services Inc.

PO Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R70108711171145RP
AES sample #: 880125 D02 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	1,600	ug/g	MF	02/01

Adirondack Environmental Services PC

PO Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R701087111711130RP
AES sample #: 880125 D03
Samples taken by: Client
MATRIX: soil
Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	700	ug/g	MF	02/01

Adirondack Environmental Services Inc.

PO Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R70108801201406SRG
AES sample #: 880125 D04 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	17,600	ug/g	MF	02/01

Adirondack Environmental Services Inc.

P.O Box 265 296 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R701087120403O3SRG
AES sample #: 880125 D05 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	18,400	ug/g	MF	02/01

Adirondack Environmental Services Inc.

P.O Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R701087120403085RG
AES sample #: 880125 D06 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	13,700	ug/g	MF	02/01

Adirondack Environmental Services Inc.

PO Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R70108712062315SRG
AES sample #: 880125 D07 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	4,850	ug/g	MF	02/01 8

Adirondack Environmental Services Inc.

PO Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R70108712062320SRG
AES sample #: 880125 D08 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DAT
Total Organic Carbon	EPA-9060	29,000	ug/g	MF	02/01

Adirondack Environmental Services Inc.

P.O. Box 265 298 Riverside Avenue Rensselaer, NY 12144 (518) 434-4546

CLIENT: Galson Research Corporation
CLIENT'S SAMPLE ID: R70108712090530SRG
AES sample #: 880125 D09 Samples taken by: Client
MATRIX: soil

Date Sampled: Unknown
Date sample received: 01/25/88
Location: E. Syracuse
grab

PARAMETER PERFORMED	METHOD	RESULT	UNITS	NOTEBK REF	TEST DATE
Total Organic Carbon	EPA-9060	1,850	ug/g	MF	02/01/88

APPROVED BY: Frank Rinaldi
Report date: 02/03/88

Appendix 6.

Results of Toxicity and Mutagenicity Testing

INDUSTRIAL CONFIDENTIAL

IITRI Project No. L08190
Study No. 1

ACUTE ORAL TOXICITY STUDY (LIMIT TEST) OF PROCESSED SOIL IN GUINEA PIGS

FINAL REPORT

IITRI Project No. L08190

STUDY NO. 1

Contractor:

IIT Research Institute
Life Sciences Research Department
10 West 35th Street
Chicago, Illinois 60616

Sponsor:

Galson Research Corporation
6601 Kirkville Road
East Syracuse, NY 13057

Attention: Ms. Edwina Milicic

April 27, 1988

IITRI Project No. L08190

Acute Oral Toxicity Study of "PROCESSED SOIL" in Guinea Pigs

EXECUTIVE SUMMARY

This report describes an acute oral toxicity study of "PROCESSED SOIL" in guinea pigs conducted by IIT Research Institute (IITRI) for the Galson Research Corporation. E.M. Furedi-Machacek, D.V.M., served as Study Director. J. Brooks Harder, D.V.M., was responsible for the supervision of Animal Care personnel. Helen V. Ratajczak, Ph.D. performed the dose administration. Mr. Anatol Oleksijew, B.S. and Mr. Ronald Weinberg were responsible for the technical aspects of the study conduct and for the data generation. Mr. Larry G. Derrick, B.S., served as manager of the Quality Assurance Unit.

The "limit test" was conducted by administering 5000 mg/kg of "PROCESSED SOIL" suspended in 1% CMC by oral gavage to one group of five male and five female Hartley stain of guinea pigs in a constant volume of 10 ml/kg. The animals were observed for 14 days after dose administration and all guinea pigs were killed at the end of the 14-day observation period and were subjected to a limited gross necropsy.

No mortality occured during the study and consequently the acute oral LD_{50} for the "PROCESSED SOIL" for male and female guinea pigs was estimated to be greater than 5000 mg/kg. All animals appeared clinically to be normal and no gross lesions were noted during the necropsy.

E. M. Furedi-Machacek 4-27-88

E.M. Furedi-Machacek, D.V.M. Date
Study Director
Life Sciences Research

James D. Fenters 4-27-88

James D. Fenters, Ph.D. Date
Head, Toxicology and
Environmental Health
Life Sciences Research

TABLE OF CONTENTS

Acute Oral Toxicity Study of "PROCESSED SOIL" in Guinea Pigs

I. INTRODUCTION

The purpose of this study was to determine the acute oral toxicity of "PROCESSED SOIL" in guinea pigs.

II. MATERIALS AND METHODS

A. **Test Article:** Approximately one-hundred grams of "PROCESSED SOIL" received on January 9, 1988 from Galson Research corp. was stored in the dark at 4°C. The test article was a dark tan powder. The Sponsor was responsible for the performance of all necessary analytical chemical analyses on the test article.

B. **Dose Formulations and Administration:** A base suspension of 500 mg/ml of "PROCESSED SOIL" in 1% CMC (Sigma, Lot No. 124F0407) was prepared by pulverizing the test article, and then transferred to a graduated cylinder and diluting until the final concentration was achieved. Dose level of 5000 mg/kg for the limit test was prepared. The suspension was continuously mixed with a magnetic stirrer prior and during the dosing. The test article was administered by oral gavage in a constant volume of 10 ml/kg each, on April 7, 1988 for the limit test.

C. **Animals:** Groups of 6 male and 6 female Hartley guinea pigs were received on March 30, 1988 for the Limit Test, at age of 4-6 weeks, from Charles River Breeding Lab., Portage, MI facility. Male guinea pigs weighed 311 to 335 grams, while female guinea pigs weighed 281 to 308 grams at the time of dosing for the Limit Test. Each animal was identified by a study-unique test animal number by a metal ear tag.

D. **Housing, Food and Water:** The guinea pigs were individually housed in polypropylene cages (16.5" x 8.5" x 7.5") throughout the study. The guinea pigs were transferred weekly to clean cages with clean bedding (Beta Chips, Northeastern Products Corp., Warrensburg, NY). Animal rooms were maintained at temperature of 18° to 27°C and relative humidity of 19% to 46%. Fluorescent lighting was provided for 12 hours of light followed by 12 hours of darkness.

The animals received Purina Guinea Pig Chow 5011 (Ralston Purina Co., St. Louis, MO) *ad libitum*, except for a fasting period of approximately 16 hours immediately prior to dosing and 4 hours after dosing. City of Chicago drinking tap water, supplied by plastic

bottles with sipper tubes, was available *ad libitum*. Fresh water was supplied to the guinea pigs twice weekly.

E. Pretest Methods:

1. Quarantine: The guinea pigs were quarantined for approximately one week prior to their assignment to test groups. During the quarantine, the animals were observed daily, and at the end of the period received a thorough physical examination to ensure their suitability for use as test animals.

2. Randomization: For the Limit Test suitable guinea pigs were assigned to one dose group of five guinea pigs per sex by draw of random numbers. There was no separate control group.

F. Limit Test:

1. Clinical Observations: All guinea pigs were observed at approximately 1, 3, and 5 hour intervals on the day of dosing and twice daily on week-days and at least once daily on week-ends for mortality and signs of toxicity.

2. Body Weights: All guinea pigs were weighed immediately prior to dosing and the weights were used for dose calculations. Body weights were measured also at test day 13 and at the study termination prior to necropsy.

3. Necropsies: All guinea pigs were subjected to a limited gross necropsy on April 21, 1988.

III. RESULTS

A. Limit Test:

1. Clinical Observations: No clinical signs of toxicity were noted during the study.

2. Mortality: No mortality occured on the study.

3. Body Weights: Mean body weights at the study initiation for male and female guinea pigs were 322 $\pm$ 11 g and 295 $\pm$ 10 g, respectively. At the study conclusion, mean body weights for male and female guinea pigs were 425 $\pm$ 9 g and 396 $\pm$ 11 g, respectively. The total body weight gain was 103 $\pm$ 14 g and 100 $\pm$ 11 for the male and female animals respectively. Further evaluation of these data was percluded by the absence of a control group.

4. Gross Necropsy Observations: No gross lesions were noted during the necropsy.

IV. EVALUATION AND CONCLUSIONS

Based on the results of this study using one dose levels of 5000 mg/kg, the acute oral LD_{50} for "PROCESSED SOIL" for male and female guinea pigs was estimated to be greater than 5000 mg/kg. No treatment related clinical or necropsy observations were noted.

All raw data generated in the conduct of this study will be maintained in the IITRI life Sciences Archives.

V. QUALITY ASSURANCE STATEMENT

Study Title: Oral Toxicity Study of "PROCESSED SOIL" in Guinea Pigs

Project Number: L08190

Study Number: 1

Study Director: E. Marianna Furedi-Machacek

Initiation Date: 4/7/88

Report Audit Date: 4/27/88

This study has been divided into a series of phases. Using a random sampling approach, Quality Assurance monitors each of these phases over a series of studies. Procedures, documentation, equipment, etc., are examined in order to assure that the study is conducted according to EPA Good Laboratory Practice regulations (40CFR792) and to the protocol.

The following are the inspection dates, phases inspected, auditor, and report dates of QA inspections submitted to management:

			Inspection Report(s) Submitted to:	
Date	Phase	Auditor	Study Director	Management
4/1/88	Body weights	Derrick	4/27/88	4/27/88
4/1/88	Quarantine	Derrick	4/27/88	4/27/88
4/7/88	Dosing	Derrick	4/27/88	4/27/88

This report describes the methods and procedures used in the study ar the reported results accurately reflect the raw data of the study.

Larry S. Derrick 4/27/88

Larry S. Derrick, B.S. Date
Manager, Quality Assurance

Industrial Confidential

IITRI Project No. L08190
Study No. SN01GRC

FINAL REPORT

Ames Salmonella Mammalian Microsomal Reverse Mutation Analysis of Test Article: Processed Soil

IIT Research Institute
Life Sciences Research
10 West 35th Street
Chicago, Illinois 60616

Peter V. Barbera, Study Director

Prepared for:

Galson Research Corporation
6601 Kirkville Road
East Syracuse, NY 13057

Attn: Ms. Edvina Milicic

Date:

April 13, 1988

IIT RESEARCH INSTITUTE

TABLE OF CONTENTS

IITRI Project No. L08190
Study No. SN01GRC

Ames Salmonella Mammalian Microsomal Reverse Mutation Analysis of Test Article: Processed Soil

SUMMARY

Processed Soil was tested for mutagenicity at doses up to 5.0 mg/plate and was found non-mutagenic, with and without metabolic activation. Doses ranging from 0.05 mg to 5.0 mg/plate were toxic to tester strain TA98.

IITRI Project No. L08190
Study No. SN01GRC

Ames Salmonella Mammalian Microsomal Reverse Mutation Analysis of Test Article: Processed Soil

Final Report

I. PURPOSE

The purpose of this study was to assess the mutagenicity of Test Article: Processed Soil using the Ames reverse mutation assay with a rat S9 activation system.

II. RATIONALE

The Ames test is an in vitro assay used to detect mutagens by their ability to cause base-pair and frameshift mutations in histidine auxotrophic (his.-) strains of Salmonella typhimurium. While five auxotrophic strains of Salmonella, which are deficient in the enzymes necessary to synthesize histidine, are normally used to measure DNA damage, two tester strains were used in the study. Although Ames and co-workers originally proposed the use of five tester strains in the bioassay, strain TA98 and TA100 are the most sensitive for mutagen detection (Ames, et al., Mutation Res. 31: 347-364, 1975). In addition, the use of both strains allows for the detection of the two mutational events - frameshift (TA98) and base-pair substitution (TA100) that constitute the basis of the Ames test. Strain TA98 is in fact the same as strain TA1538 but with an added resistance transfer (R) factor. Strain TA100 likewise is the same as strain TA1535 with the additionally added resistance transfer (R) factor. Ames has suggested in his methodology paper, cited above, that for all practical purposes strain TA1538 may be deleted as strain TA98 appears to be a more sensitive mutagen detector. Pelroy R.A., and Peterson, M.R. (Environ. Health Perspect. 30: 191-203, 1979) selected these two strains, TA98 and TA100, as the basis for evaluating five shale oil fractions: acidic, basic, neutral, polynuclear aromatic (PNA) and a tar fraction. These two selected strains were also utilized by Wei, E.T. et al., (J. Air Pollution Control Assoc. 30: (3) 267-271, 1980) in their evaluation of deisel emissions using the Ames test. Wang, et al., (Can. Res. 35: 3611-3717, 1975) utilized both of these selected strains in their evaluation of thirty-two heterocyclic compounds after noting that some of the nitroheterocycles failed to demonstrate mutagenic activity with strains TA1535, TA1537 and TA1538 but did demonstrate mutagenicity in other test systems. This approach has also been recognized by the

Department of Health, Education and Welfare Food and Drug Administration in their RFP No. 223-80-2339 entitled "Comparison of Activiation by Microsomes from Target and Test Species in Ames/Salmonella Assays".

When mutations occur in the histidine operon, the bacteria are reverted to the histidine independent (his.+) prototrophic wild type. These "reverse mutants" can grow in histidine deficient medium. The his.+ revertants are easily scored as colonies against a slight background bacterial lawn. The background lawn results from addition of trace amounts of histidine to the medium which enables all of the his.- bacteria to undergo a few cell divisions before the histidine is depleted; this growth is essential for the expression of any mutagenic events. Mammalian liver microsomes (S9) are also added to the cultures to mimic the in vivo activation pathways necessary for activating promutagenic agents.

When mutagens are added to the agar medium, the number of mutant colonies is increased over spontaneous background levels. The difference in number of mutant colonies in the exposed cultures over the negative control (spontaneous revertants) is the measure of the test.

III. MATERIALS

A. Test Article: The test article stability was the responsibility of the Sponsor.

- Date received: Ninety-six grams were received on January 26, 1988. Two grams were assigned for the Mutagenicity Test.
- Identification code: Processed Soil
- Handling: The test article was mixed thoroughly and a reference sample taken. The test article was stored in the vessel in which it was received at 3°C ± 1°C and protected from light.
- Physical characteristics: The test article is a cocoa-colored finely divided powder.
- Dosage Formulations: The test article was suspended and diluted in dimethyl sulfoxide. The test article was partially insoluble. Since particulates would likely have obscured background colonies, the suspension was filtered through a Teflon 0.45 µ filter unit (Gelman 4219). The dimethyl sulfoxide was assumed to have acted as an extractant. The dose levels tested for mutagenicity were 5.0, 1.0, 0.5, 0.05 and 0.005 mg/plate.

B. Test Control Substances:

POSITIVE CONTROLS
Non-Activated System (Without S9)

Strain	Compound	Conc.	Acceptable Revertants*
TA98	2-Nitrofluorene (2NF)	10 µg	500- 900
TA100	Sodium Azide (NAZ)	10 µg	700-1200

Activated System (With S9)

Strain	Compound	Conc.	Acceptable Revertants*
TA98	2-Anthramine (2A)	10 µg	1400-2000
TA100	2-Anthramine (2A)	10 µg	1500-2300

Negative Control

DMSO 100 µl/plate

* based on internal historical data

C. S. typhimurium Tester Strains:

Two mutant strains of Salmonella typhmurium (TA98 and TA100) were used in the test. The two strains were originally supplied by Dr. Bruce Ames, University of California Berkeley, Berkeley, California in 1978. The use of these specific strains are recommended by NIEHS (Science 203:563-565, 1979) and by the EPA (Federal Register, Part IV, July 29, 1979). These strains are also recommended in FDA Document 600/9-79-027, Sept. 1978. The two strains detect reverse mutations which are of both frameshift and base-pair substitution types. The utility of the test has been validated by among others, Bartsch, H.C. et al (Mut. Res. 75, 1-50, 1980). Stock cultures were prepared, frozen at -70°C and then tested for genotypic characteristics (his. rfa, uvr B, bio) and for the pKM101 plasmid as described in Ames et al. (Mutation Research 31:347-354, 1975).

Stock Cultures. Stock cultures derived from the stock received from Dr. Ames were prepared by growing the organisms in nutrient broth No. 2 (Oxoid Ltd. Wade Road,

Basingstoke Hampshire, U.K.) at 37°C overnight. After incubation, 9% v/v DMSO was added and the culture aliquoted in 1 ml volumes into Cryotubes II (Nunc, Denmark). The cryotubes were placed at -70°C and the frozen stock tested for genotypic characteristics at that time. The strains have the following characteristics in addition to the mutation at the histidine operon:

Strain name	Gene affected	Additional Mutations LPS	Repair	R factor	Mutation Type Detected	Spontaneous revertants*
TA98	his D	rfa	uvr B	pKM101	Frameshift	14- 50
TA100	his D	rfa	uvr B	pKM101	Base-pair substitution	120-200

* based on historical data with and without S9

The uvr- B (uv-repair) mutation decreases the organism's ability to repair some forms of genetic damage caused by certain chemical and physical agents. The rfa- (deep rough) mutation leads to defective lipopolysaccharide cell wall formation by the bacteria thereby making the cell walls more permeable to larger molecular weight agents. The pKM101 resistant transfer factor plasmid (R factor) confers resistance to ampicillin and is thought to cause an increase in error-prone DNA repair. All of these mutations make the strains more susceptible to genetic damage and therefore make the bacteria more sensitive organisms for identifying DNA damaging agents.

Working Stock Culture. Working stock cultures for the assay were prepared by transfer of a 1 ml -70°C stock culture into nutrient broth No. 2 and incubating with aeration on a shaker platform at 37°C for 16 hrs on March 29, 1988.

D. Media:

Mutagenicity test. The minimal glucose agar medium used in this study consisted of 20 ml of Vogel-Bonner medium E (Vogel and Bonner, J. Biol. Chem. 218:97-106, 1956) with 1.5% agar-agar and 2% dextrose.

The top overlay agar contained the folloving per 100 ml volume as per Ames, et al., (Mutation Research 31:347-354, 1975):

o	agar agar	0.6 g
o	0.5 mM L-Histidine	10.0 ml
o	0.5 mM Biotin	
o	sodium chloride	0.5 g
o	distilled water	100 ml

E. Mixed Function Oxidase Activation System:

S9 Homogenate. The rat liver S9 was purchased from Organon Teknika Cappel, Irving, Texas. The 9,000 x g microsomal fraction was prepared from male Sprague-Davley rats. The rats received a single intraperitoneal injection of Aroclor 1254, then the 9,000 x g microsomal fraction was prepared using the procedure of Ames et al. (Mutation Research 31:347-354, 1975). Upon receipt the S9 (Lot No. 07414) was stored at -70°C until used.

S9 Mix. The S9 mix was prepared just prior to use following the method of Ames, et al., (Mutation Research 31:347-354, 1975). The S9 mix was filter sterilized through a 0.45 um membrane filter, then was mixed with the S9 homogenate just prior to use. The final S9 reaction mixture contained the following concentration of each component per ml:

o	NADP (sodium salt)	4 µM	0.45 ml
o	D-glucose-6-phosphate	5 µM	
o	MgCl	8 µM	
o	KCl	33 µM	0.45 ml
o	Sodium phosphate buffer (pH 7.4)	100 µM	
o	S9 Homogenate	0.1 ml	

The amount of hepatic S9 mix used (0.5 ml/tube) represented 450 µl of core reaction mixture and 50 µl of rat liver S9 product.

IV. METHODS

A. Mutagenicity Testing: The Ames assay was performed on March 30, 1988 following the procedures outlined in IITRI SOP No. MBGT53R2. The plate incorporation procedures are based on those of Ames et al. (Mutation Research 31:347-364, 1975). Briefly, pour plates were made by adding, in order, 0.1 ml of a 16-hr Oxoid nutrient broth culture, 20 ml volume (incubated with shaking in 50 ml flask) of the tester strain to 2 ml of molten top-agar (45°C) in sterile unused 16x150 mm test tubes followed by 0.1 ml of the control or test article to be tested and 0.5 ml of the described S9 mix kept in ice bath. The preparation of the

working cultures were based on Ames' recommendations and complied with NIEHS International collaborative study recommendations Anderson, D. et al (Mutation Research 130:1-10, 1984). Each plate was inoculated with 10^8 viable cells. The small amount of histidine and biotin added to the top agar alloved the bacteria to undergo several divisions. The tubes were vortex mixed then the contents were poured onto the surface of prelabeled minimal medium agar plates. All test and control plates were in triplicate. The plates were incubated in an inverted position at 37°C in the dark for 2 days after which the number of revertant colonies were counted with a Biotran™ II colony counter on April 1, 1988. The plates were hand counted when there were fever than 50 colonies. Simultaneously, observations for a decrease in background lawn were made. These were used as an index of toxicity. Upon completion of the study all plates were disposed of by incineration on April 1, 1988.

As a check for bacterial contamination, sterility tests were performed simultaneously on the overlay agar, biotin-histidine, solvent and S9 components using triplicate plates. A sterility test, using a single plate/dose, was also performed on the test article. An additional 5% of the uninoculated minimal medium agar plates were evaluated for sterility.

B. Interpretation of Data: The data are reported as average number of revertants per dose.

1. A test was considered valid if the following criteria were met:

   - o No evidence of general microbial contamination.
   - o The average number of revertants for the S9 positive control and the direct acting positive controls were in the expected range (see Section III-B).
   - o The average number of revertants for the solvent control background spontaneous revertants were within the expected range (see Section III-C).

2. A test was considered positive under the following conditions:

   - o The average number of revertants was dose responsive and at least one dose was >2X the solvent control spontaneous revertant value for at least one tester strain.

- o A non-dose responsive test was considered positive only if the positive dose was the high dose or if the subsequent doses showed a plateau response or a corresponding toxic response as evidenced by a decrease in background lawn.
- o A test result was considered suspicious if the average number of revertants was dose responsive but all doses were <2X the solvent control spontaneous revertant value. Conclusions on the mutagenicity of such test articles would require further testing in other test systems or within a more narrow concentration range.

3) A test was considered negative if the average number of revertants was not dose responsive and all doses were <2X the solvent control spontaneous revertants value for each tester strain.

V. SUPERVISORY PERSONNEL USED IN THIS STUDY

James D. Fenters, Ph.D.	Head, Toxicology and Environmental Health
Peter W. Barbera	Study Director
Charles Gradle	Research Associate
Larry G. Derrick	Manager, Quality Assurance

VI. SUMMARY AND ANALYSIS OF DATA

- o Background toxicity test: The results of the background lawn toxicity test are presented in Table 1. The test article was toxic to Tester Strain TA98 at the 5.0 to 0.5 mg/plate level without metabolic activation, and toxic at the 5.0 to 0.05 mg/plate level with metabolic activation. The effect of the toxic response can be seen in the reduced colony counts as compared to the spontaneous revertant count (Table 3). Toxicity was not seen with Tester Strain TA100 at any dose evaluated.

- o Mutagenicity test: There was no evidence of contamination from either the test article or the assay components. The positive and negative assay controls were all within the limits set for the test (Tables 2-3).

  Since a toxic response was seen with Tester Strain TA98, an evaluation for mutagenicity could not be performed for dose

Since a toxic response was seen with Tester Strain TA98, an evaluation for mutagenicity could not be performed for dose levels 5.0 to 0.5 mg/plate without activation or dose levels 5.0 to 0.05 mg/plate with activation. A mutagenic response was not seen at non-toxic levels. Additionally, a mutagenic response was not seen with Tester Strain TA100 at any of the doses tested (Table 3). The standard deviations for the test article and assay controls are presented in Table 4.

- Hepatic S9: The S9 product yielded the following for Lot 07414.

  1. Alkoxyphenoxazone Dealkylase activity - (P448):4835 pmol/min./mg protein.

  2. Protein (Lovry, et al., J. Biol. Chem. 192:265, 1951): 43.0 mg/ml.

  3. Benzo(α)pyrene 5 ug/plate activity curve with tester strain TA98 (Ames, et al., Mut. Res. 32:347, 1975): 770 revertant colonies.

  4. Benzo(α)pyrene 5 ug/plate activity curve with tester strain TA100 (Ames, et al., Mut. Res. 32:347, 1975): 952 revertant colonies.

VII. CONCLUSIONS

Since there was no evidence of microbial contamination and the positive and solvent controls were within the range set for the assay, the test was considered valid. Within the doses of test article that could be evaluated for mutagenicity, in consideration of the observed toxicity, Processed Soil was found not to be mutagenic against either of the tester strains but toxic to Tester Strain TA98 at dose levels ranging from 5.0 mg to 0.05 mg/plate. It is of interest to note the lack of an increase in average colony counts with decreasing test article toxic dose levels in Tester Strain TA98. This type of response could result from a competition between mutagenicity and toxicity.

VIII. SIGNATURE OF SCIENTISTS INVOLVED IN THIS STUDY

James Fenters
James D. Fenters, Ph.D.
Toxicology & Environmental Health

4/14/88
Date

Peter W. Barbera
Peter W. Barbera
Study Director

4/14/88
Date

IX. STORAGE OF DATA AND REPORTS

All rav data generated during the course of this study were retained in the IITRI Life Sciences archives as specified by government regulations. The original and one copy of final report was submitted to the Sponsor and one copy of the report was retained in the IITRI archives, Department L files and one by the program director.

X. QUALITY ASSURANCE

Quality Assurance Statement: This study was conducted in accordance with EPA Good Laboratory Practice (GLP) Regulations (40 CFR 160). The study has been subjected to inspections, and this report has been audited by the IITRI Quality Assurance Unit. The report accurately reflects the rav data obtained during the study. There were no significant deviations from GLP regulations which would have affected the integrity of the study. Phases inspected, dates of inspections, auditor and the individual who audited the final report are listed below.

Phase	Date	Auditor
Ames Assay	March 30, 1988	M. Marlov
Plate Counts	April 1, 1988	M. Marlov
Final Report Audit	April 13, 1988	M. Marlov

Larry A. Derrick
Larry A. Derrick, B.S.
Manager, Quality Assurance

4/13/88
Date

XI. TABLES

TABLE 1

Toxicity (Background) Test Results

Test Article Concentration (mg/plate)	TA98 -S9	TA98 +S9	TA100 -S9	TA100 +S9
5.0	T	T	N	N
1.0	T	T	N	N
0.5	T	T	N	N
0.05	N	T	N	N
0.005	N	N	N	N
Control				
DMSO Solvent 100 µl	N	N	N	N

-S9 = Non-activated
+S9 = Activated
T = Toxic Response
N = No Toxic Response

TABLE 2

Mutagenicity Test Control Results

	Revertants/Plate (X)			
	TA98		TA100	
Controls	-S9	+S9	-S9	+S9
Positive				
2NF 10 µg	875 251 818 (648)			
NAZ 10 µg			1043 1119 1040 (1067)	
2A 10 µg		1804 1580 1388 (1591)		2041 2309 2524 (2291)

+S9 = activated
-S9 = non-activated
2NF = 2-nitrofluorene
NAZ = sodium azide
2A = 2-anthramine
(X) = mean of triplicate plate counts

TABLE 3

Mutagenicity Test Results for the Test Article

	Revertants/Plate (X)			
	TA98		TA100	
Controls	-S9	+S9	-S9	+S9
Spontaneous	18	29	124	185
Revertants	18	25	120	181
Solvent Control	19	30	123	188
(100 µl)	(18)	(28)	(122)	(185)
Test Article				
Conc. mg/plate				
5.0	11	16	160	159
	17(15)	16(17)	133(153)	167(173)
	18	20	165	193
	(NE)	(NE)	(--)	(--)
1.0	12	18	142	148
	10(13)	16(20)	144(139)	225(187)
	17	27	130	188
	(NE)	(NE)	(--)	(--)
0.5	8	20	188	100
	10(9)	17(17)	170(173)	185(146)
	8	15	162	152
	(NE)	(NE)	(--)	(--)
0.05	16	19	215	208
	19(16)	24(20)	201(199)	199(211)
	13	18	180	227
	(--)	(NE)	(--)	(--)
0.005	19	28	191	178
	18(18)	27(29)	184(199)	211(196)
	17	32	223	199
	(--)	(--)	(--)	(--)

(+) = mutagenic -S9 = non-activated
(-) = non-mutagenic +S9 = activated
(X) = mean of triplicate plate counts
(NE) = not evaluated, toxicity present

Table 4

STANDARD DEVIATIONS (S.D ±)

	TA98		TA100	
	-S9	+S9	-S9	+S9
Positive Controls				
2NF 10 µg	345.0			
NAZ 10 µg			44.8	
2A 10 µg		208.2		242.0
Spontaneous Revertants Solvent Control (100 µl)	0.6	2.6	2.1	3.5
Test Article				
Conc. mg/plate				
5.0	3.8	2.3	17.2	17.8
1.0	3.6	5.9	7.6	38.5
0.5	1.2	2.5	13.3	42.9
0.05	3.0	3.2	17.6	14.3
0.005	1.0	2.7	20.8	16.7

Appendix 7.

Results of Particle Size Analysis

EMPIRE
SOILS INVESTIGATIONS INC

REPORT OF MATERIAL TESTING

PROJECT: Material Testing
CLIENT: Glason Research Corporation

The ASTM procedures for Hydrometer Analysis and Liquid/Plastic Limits had to be adjusted due to the nature of the contamination in the sample. Therefore, normal accuracy cannot be expected.

Because of the adjustments, the Liquid Limit and Plasticity Index are lower than what might be expected had ASTM procedures been followed exactly.

The Hydrometer Analysis shows somewhat larger particle size than would be expected had ASTM procedures been followed exactly.

Respectfully submitted,

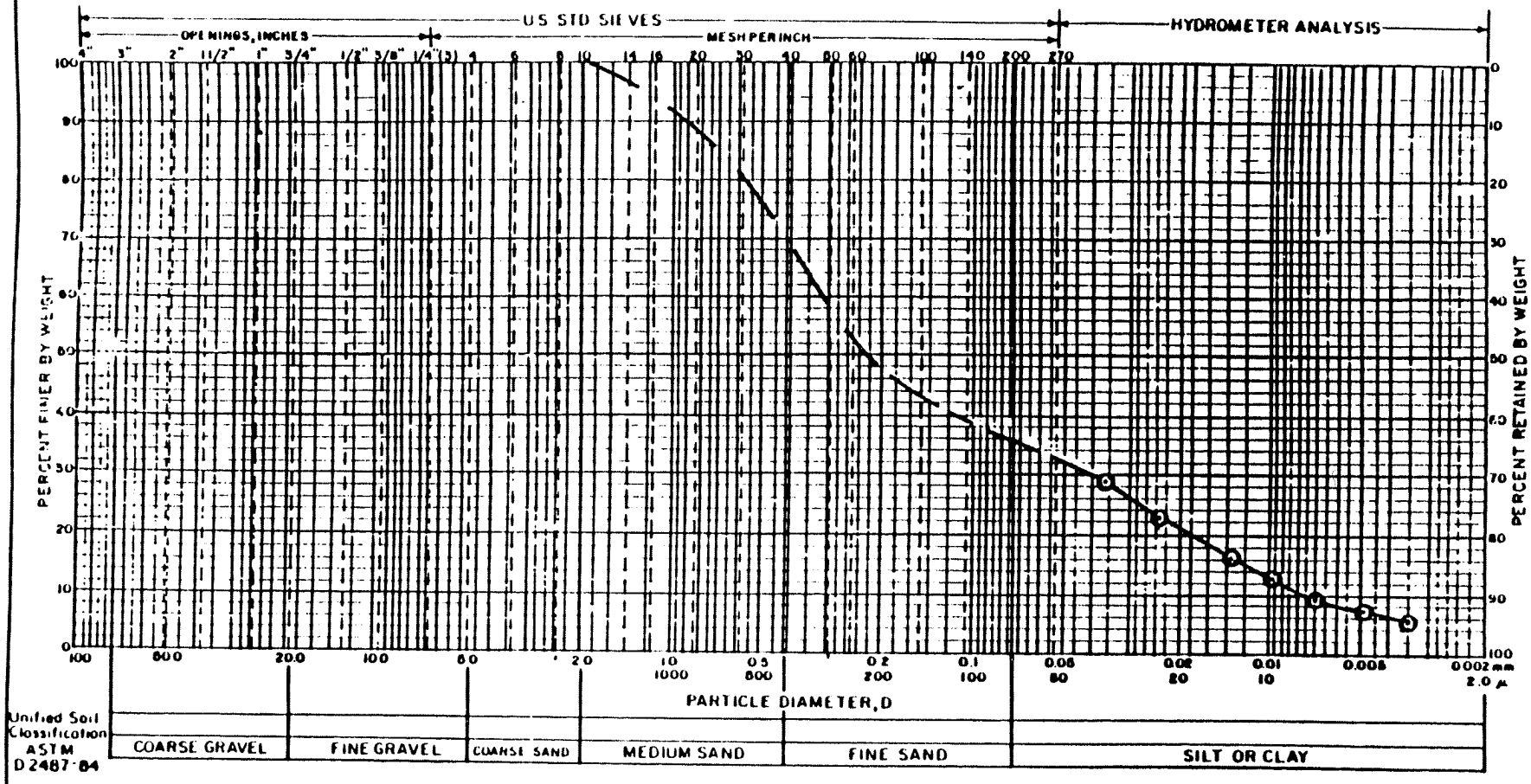
EMPIRE SOILS INVESTIGATIONS, INC.

Thomas Hamilton

Thomas A. Hamilton
Administrator of Technical Services

1-5-88
GT-87-170

PARTICLE SIZE DISTRIBUTION CURVE



SAMPLE INFORMATION:

Liquid Limit:	60
Plastic Limit:	36
Plasticity Index:	24

Note: Dashed line represents a visual estimate of particle size.

☐ Insufficient Sample Size per ASTM D 422

EMPIRE SOILS INVESTIGATIONS INC

PARTICLE SIZE ANALYSIS

MATERIAL TESTING
EAST SYRACUSE, NEW YORK
GALSON RESEARCH CORPORATION

DR. BY: NG	CK'D.	DATE: 12/87	PROJ. NO. GT-87-170

Appendix 8.

Mass Balance Spreadsheets

Mass Balance for Wide Beach Reaction #1

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300 5	Treated Soil	155 5		Max temp, °C	140
5	1.1:2 mix	200 4	Reagent	162 9		Soil ID	Wide Beach "A"
6	45% KOH	100 2	Reactor samps	64 5	list below	Sulfoxide used	DMSO
7	Wash 1	240	Wash 1	108 7			
8	Wash 2	240	Wash 2	348			
9	Wash 3	240	Wash 3	174.9			
10	replace cond	53 7	condensate	53.2			
11	Total Inputs	1374 8	Total Outputs	1067 7			
12	Total inputs -	total outputs	307 1				
13							
14		Samples	g from reactor	soil weight			
15	Initial	87112501*	3 6	3 6			
16	R70108711	300005JRa	*5 6*	2 8			
17	R70108711	300105JRa	5 1	2 5			
18	R70108711	300205JRa	4 6	2 2			
19	R70108711	300305JRa	3 7	3 3			
20	R70108711	300405JRa	5 6	3			
21	R70108711	300505JR	7	4 2			
22	R70108711	300605JR	8 2	4 6			
23	R70108711	300705JR	3 5	2 9			
24	R70108711	300805JR	7 2	4 3			
25	R70108711	300905EM	4 3	*2 15*			
26	R70108711	301105EM	6 1	*3 05*			
27		subtotal	64 5	38 6			
28							
29		*old sample numbering system used before 11/27					
30		Weights in italics are estimated (missing data)					
31							
32	GROSS MASS BALANCE FOR REACTION						
33							
34	Total mass weighed into reactor (in grams)			601 1			
35	Total mass of samples removed from reactor			64 5			
36	Total mass in reactor at end of reaction			397 6			
37	Mass of condensate collected from reactor			53 2			
38	*Mass lost during reaction and sampling*			*85.8*			
39							
40	GROSS MASS BALANCE FOR SOIL WASHING						
41							
42	Mass in reactor after reaction			397 6			
43	Mass of water added to replace condensate			53 7			
44	Total mass in reactor before filtration			451 3			
45	Mass of reagent recovered			162 9			
46	*Mass in apparatus after reagent removal*			*288 4*			
47							
48	Mass of wash 1 added			240			
49	Mass of wash 1 recovered			108.7			
50	*Mass in apparatus after Wash #1*			*419.7*			
51							
52	Mass of wash 2 added			240			
53	Mass of wash 2 recovered			348			
54	*Mass in apparatus after Wash #2*			*311.7*			
55							
56	Mass of wash 3 added			240			
57	Mass of wash 3 recovered			174.9			
58	*Mass in apparatus after Wash #3*			*376.8*			
59							
60	Dry soil recovered			155.5			
61	*Mass lost in filtration and soil drying*			*221.3*			
62							
63	*TOTAL LOSSES (SAMPLING, FILTRATION AND DRYING)*				307 1		

	A	B	C	D	E	F	G
64	SOIL AND CHEMICAL MASS BALANCES						
65							
66	SOIL	INPUT	SAMPLES	FINAL			
67	total mass	300	NA	155 5			
68	% moisture	23 6	NA	0			
69	dry mass	229 2	38 6	155 5			
70	% Recovery, dry basis		84 6858639				
71							
72	REAGENT INPUT						
73	component	PEG	TMH	DMSO	KOH		
74	mass used	50 1	50 1	100.2	100.2		
75	% purity	100	100	100	45		
76	Actual g used	50 1	50 1	100.2	45.09		
77							
78	REAGENT OUTPUT		SAMPLE #	R70108712020001RJR			
79	total mass	162 9					
80	mg/g	193	172	314	8.4		
81	g found	31 4397	28 0188	51 1506	1.36836		
82	% recovery	62 7538922	55 9257485	51 048503	3.03473054		
83							
84	WASH 1 OUTPUT		SAMPLE #	R70108712020030WJR			
85	total mass	108 7					
86	mg/g	12	27	51	53 8		
87	g found	1 3044	2 9349	5 5437	5.84806		
88	% recovery	2 6035928	5 85808383	5 53263473	12 9697494		
89							
90	WASH 2 OUTPUT		SAMPLE #	R70108712020156WJR			
91	total mass	348					
92	mg/g		12	26	40.3		
93	g found	0	4 176	9 048	14 0244		
94	% recovery	0	8 33532934	9 02994012	31 1031271		
95							
96	WASH 3 OUTPUT		SAMPLE #	R70108712020247WJR			
97	total mass	174 9					
98	mg/g		5	15	22.4		
99	g found	0	0 8745	2.6235	3.91776		
100	% recovery	0	1 74550898	2 61826347	8.68875582		
101							
102	TOTAL %R	65 357485	71 8646707	68 2293413	55.7963628		
103							
104							
105	*Detection Limits*						
106		*PEG 10 mg/g*	*TMH 1mg/g*	*DMSO 1mg/g*			

Mass Balance for Wide Beach Reaction #2

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300.4	Treated Soil	225.8		Max temp, °C	140
5	1:1:2 mix	200	Reagent	194.3		Soil ID	Wide Beach "B"
6	45% KOH	100	Slurry samps	27.3	list below	Sulfoxide used	DMSO
7	Wash 1	240	Wash 1	89.2			
8	Wash 2	120	Wash 2	233.4			
9	Wash 3	240	Wash 3	219.7			
10	replace cond	33.4	condensate	33.4			
11	Total Inputs	1233.8	Total Outputs	1023.1			
12	Total Inputs - total outputs		210.7				
13							
14		Samples	g from reactor	soil weight			
15		87112502*	3.7	3.7			
16	R70108711	300005JRb	*4*	2			
17	R70108711	300105JRb	3.8	1.3			
18	R70108711	300205JRb	6	2.4			
19	R70108711	300305JRb	4.6	3.5			
20	R70108711	300405JRb	5.2	4.5			
21		subtotal	27.3	17.4			
22							
23		*old sample numbering system used before 11/27					
24		Weights in italics are estimated (missing data).					
25							
26	MASS BALANCE FOR REACTION						
27							
28	Total mass weighed into reactor (in grams)			600.4			
29	Total mass of samples removed from reactor			27.3			
30	Total mass in reactor at end of reaction			505			
31	Mass of condensate collected from reactor			33.4			
32	*Mass lost during reaction and sampling*			*34.7*			
33							
34	MASS BALANCE FOR SOIL WASHING						
35							
36	Mass in reactor after reaction			505			
37	Mass of water added to replace condensate			33.4			
38	Total mass in reactor before filtration			538.4			
39	Mass of reagent recovered			194.3			
40	*Mass in apparatus after reagent removal*			*344.1*			
41							
42	Mass of wash 1 added			240			
43	Mass of wash 1 recovered			89.2			
44	*Mass in apparatus after Wash #1*			*494.9*			
45							
46	Mass of wash 2 added			120			
47	Mass of wash 2 recovered			233.4			
48	*Mass in apparatus after Wash #2*			*381.5*			
49							
50	Mass of wash 3 added			240			
51	Mass of wash 3 recovered			219.7			
52	*Mass in apparatus after Wash #3*			*401.8*			
53							
54	Dry soil recovered			225.8			
55	*Mass lost in filtration and soil drying*			*176*			
56							
57	*TOTAL LOSSES (SAMPLING, FILTRATION AND DRYING)*				210.7		

	A	B	C	D	E	F	G
58	SOIL AND CHEMICAL MASS BALANCES						
59							
60	SOIL	INPUT	SAMPLES	FINAL			
61	total mass	300.4	NA	225.8			
62	% moisture		NA	0			
63	dry mass	300.4	17.4	225.8			
64	% Recovery, dry basis		80.9587217				
65							
66	REAGENT INPUT						
67	component	PEG	TMH	DMSO	KOH		
68	mass used	50	50	100	100		
69	% purity	100	100	100	45		
70	Actual g used	50	50	100	45		
71							
72	REAGENT OUTPUT		SAMPLE #	R701087120	20430RJR		
73	total mass	194.3					
74	mg/g	226	168	311	54.8		
75	g found	43.9118	32.6424	60.4273	10.64764		
76	% recovery	87.8236	65.2848	60.4273	23.6614222		
77							
78	WASH 1 OUTPUT		SAMPLE #	R701087120	20520WJR		
79	total mass	89.2					
80	mg/g	16	41	84	117.6		
81	g found	1.4272	3.6572	7.4928	10.48992		
82	% recovery	2.8544	7.3144	7.4928	23.3109333		
83							
84	WASH 2 OUTPUT		SAMPLE #	R701087120	20555WJR		
85	total mass	233.4					
86	mg/g		11	24	33.6		
87	g found	0	2.5674	5.6016	7.84224		
88	% recovery	0	5.1348	5.6016	17.4272		
89							
90	WASH 3 OUTPUT		SAMPLE #	R701087120	20647WJR		
91	total mass	219.7					
92	mg/g			2	10.1		
93	g found	0	0	0.4394	2.21897		
94	% recovery	0	0	0.4394	4.93104444		
95							
96	TOTAL %R	90.678	77.734	73.9611	69.3306		
97							
98							
99	*Detection Limits*						
100		*PEG: 10 mg/g*	*TMH: 1mg/g*	*DMSO: 1mg/g*			

Mass Balance for Wide Beach Reaction #3

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300	Treated Soil	141.5		Max temp, °C	140
5	1:1:2 mix	200	Reagent	155.3		Soil ID	Wide Beach °C°
6	45% KOH	100	Slurry samps	64.3	list below	Sulfoxide used	DMSO
7	Wash 1	240.1	Wash 1	282.3			
8	Wash 2	240	Wash 2	152.2			
9	Wash 3	240	Wash 3	152.9			
10	replace cond.	121	condensate	121			
11	Total Inputs	1441.1	Total Outputs	1069.5			
12	Total inputs -	total outputs	371.6				
13							
14		Samples	g from reactor	soil weight			
15	R70108712	020445SJR	3.4	3.4			
16	R70108712	022305SJR	4.9	2.1			
17	R70108712	030015SJR	4.5	2			
18	R70108712	030115SJR	6	3.1			
19	R70108712	030215SJR	7.2	4.3			
20	R70108712	030315SJR	7.5	4.7			
21	R70108712	030415SJR	5.6	3.4			
22	R70108712	030515SJR	6.2	4.4			
23	R70108712	030615SJR	7.4	3.4			
24	R70108712	030715SJR	8.5	5.3			
25	R70108712	030815SJR	6.5	4.1			
26		subtotal	64.3	36.8			
27							
28	MASS BALANCE FOR REACTION						
29							
30	Total mass weighed into reactor (in grams)			600			
31	Total mass of samples removed from reactor			64.3			
32	Total mass in reactor at end of reaction			314.3			
33	Mass of condensate collected from reactor			121			
34	*Mass lost during reaction and sampling*			*100.4*			
35							
36	MASS BALANCE FOR SOIL WASHING						
37							
38	Mass in reactor after reaction			314.3			
39	Mass of water added to replace condensate			121			
40	Total mass in reactor before filtration			435.3			
41	Mass of reagent recovered			155.3			
42	*Mass in apparatus after reagent removal*			*280*			
43							
44	Mass of wash 1	added		240.1			
45	Mass of wash 1	recovered		282.3			
46	*Mass in apparatus after Wash #1*			*237.8*			
47							
48	Mass of wash 2	added		240			
49	Mass of wash 2	recovered		152.2			
50	*Mass in apparatus after Wash #2*			*325.6*			
51							
52	Mass of wash 3	added		240			
53	Mass of wash 3	recovered		152.9			
54	*Mass in apparatus after Wash #3*			*412.7*			
55							
56	Dry soil recovered			141.5			
57	*Mass lost in filtration and soil drying*			*271.2*			
58							
59	*TOTAL LOSSES (SAMPLING, FILTRATION AND DRYING)*				371.6		
60							

	A	B	C	D	E	F	G
61	SOIL AND CHEMICAL MASS BALANCES						
62							
63	SOIL	INPUT	SAMPLES	FINAL			
64	total mass	300	NA	141.5			
65	% moisture		NA	0			
66	dry mass	300	36.8	141.5			
67	% Recovery, dry basis		59.4333333				
68							
69	REAGENT INPUT						
70	component	PEG	TMH	DMSO	KOH		
71	mass used	50	50	100	100		
72	% purity	100	100	100	45		
73	Actual g used	50	50	100	45		
74							
75	REAGENT OUTPUT		SAMPLE #	R70108712040605RRG			
76	total mass	155.3					
77	mg/g	282	212	134	28		
78	g found	43.7946	32.9236	20.8102	4.3484		
79	% recovery	87.5892	65.8472	20.8102	9.66311111		
80							
81	WASH 1 OUTPUT		SAMPLE #	R70108712040640WJR			
82	total mass	282.3					
83	mg/g	13	30	26	31.4		
84	g found	3.6699	8.469	7.3398	8.86422		
85	% recovery	7.3398	16.938	7.3398	19.6982667		
86							
87	WASH 2 OUTPUT		SAMPLE #	R70108712040725WJR			
88	total mass	152.2					
89	mg/g		3	4	6.7		
90	g found	0	0.4566	0.6088	1.01974		
91	% recovery	0	0.9132	0.6088	2.26608889		
92							
93	WASH 3 OUTPUT		SAMPLE #	R70108712040810WJR			
94	total mass	152.9					
95	mg/g			1	5.6		
96	g found	0	0	0.1529	0.85624		
97	% recovery	0	0	0.1529	1.90275556		
98							
99	TOTAL %R	94.929	83.6984	28.9117	33.5302222		
100							
101	*Detection Limits*						
102		*PEG 10 mg/g*	*TMH: 1mg/g*	*DMSO: 1mg/g*			

Mass Balance for Wide Beach Reaction #4

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300	Treated Soil	161.5		Max temp, °C	140
5	1:1:2 mix	201	Reagent	65.7		Soil ID	Wide Beach "C"
6	45% KOH	100	Slurry samps	50.9	list below	Sulfoxide used	Sulfolane
7	Wash 1	240	Wash 1	334.2			
8	Wash 2	240	Wash 2	151.6			
9	Wash 3	240	Wash 3	161.6			
10	replace cond.	47.1	condensate	47.1			
11	Total Inputs	1368.1	Total Outputs	972.6			
12	Total inputs - total outputs		395.5				
13							
14		Samples	Slurry weight	Soil weight			
15	R7010871	2022318SJR	6.1	3.4			
16	R7010871	2030018SJR	5.1	1.1			
17	R7010871	2030118SJR	4.4	1.9			
18	R7010871	2030218SJR	5.1	2.2			
19	R7010871	2030318SJR	5	4.7			
20	R7010871	2030418SJR	4.7	3.5			
21	R7010871	2030518SJR	5.2	2.4			
22	R7010871	2030618SJR	6.4	4.7			
23	R7010871	2030718SJR	4.8	3.1			
24		2030818SJR	4.1	2.7			
25		subtotal	50.9	29.7			
26							
27	MASS BALANCE FOR REACTION						
28							
29	Total mass weighed into reactor (in grams)			601			
30	Total mass of samples removed from reactor			50.9			
31	Total mass in reactor at end of reaction			392.9			
32	Mass of condensate collected from reactor			47.1			
33	*Mass lost during reaction and sampling*			*110.1*			
34							
35	MASS BALANCE FOR SOIL WASHING						
36							
37	Mass in reactor after reaction			392.9			
38	Mass of water added to replace condensate			47.1			
39	Total mass in reactor before filtration			440			
40	Mass of reagent recovered			65.7			
41	*Mass in apparatus after reagent removal*			*374.3*			
42							
43	Mass of wash 1	added		240			
44	Mass of wash 1	recovered		334.2			
45	*Mass in apparatus after Wash #1*			*280.1*			
46							
47	Mass of wash 2	added		240			
48	Mass of wash 2	recovered		151.6			
49	*Mass in apparatus after Wash #2*			*368.5*			
50							
51	Mass of wash 3	added		240			
52	Mass of wash 3	recovered		161.6			
53	*Mass in apparatus after Wash #3*			*446.9*			
54							
55	Dry soil recovered			161.5			
56	*Mass lost in filtration and soil drying*			*285.4*			
57							
58	*TOTAL LOSSES (SAMPLING, FILTRATION AND DRYING)*				395.5		

Mass Balance for Wide Beach Reaction #4

	A	B	C	D	E	F	G
59	SOIL AND CHEMICAL MASS BALANCES						
60							
61	SOIL	INPUT	SAMPLES	FINAL			
62	total mass	300	NA	165.1			
63	% moisture		NA	0			
64	dry mass	300	29.7	165.1			
65	% Recovery, dry basis		64.9333333				
66							
67	REAGENT INPUT						
68	component	PEG	TMH	SFLN	KOH		
69	mass used	50.25	50.25	100.5	100		
70	% purity	100	100	100	45		
71	Actual g used	50.25	50.25	100.5	45		
72							
73	REAGENT OUTPUT		SAMPLE #	R70108712040845RJR			
74	total mass	65.7					
75	mg/g	325	181	407	9		
76	g found	21.3525	11.8917	26.7399	0.5913		
77	% recovery	42.4925373	23.6650746	26.6068657	1.314		
78							
79	WASH 1 OUTPUT		SAMPLE #	R70108712040922WJR			
80	total mass	334.2					
81	mg/g	25	59	130	42.6		
82	g found	8.355	19.7178	43.446	14.23692		
83	% recovery	16.6268657	39.239403	43.2298507	31.6376		
84							
85	WASH 2 OUTPUT		SAMPLE #	R70108712041013WJR			
86	total mass	151.6					
87	mg/g		13	31	13.4		
88	g found	0	1.9708	4.6996	2.03144		
89	% recovery	0	3.92199005	4.67621891	4.51431111		
90							
91	WASH 3 OUTPUT		SAMPLE #	R70108712041058WJR			
92	total mass	161.6					
93	mg/g		2	11	7.8		
94	g found	0	0.3232	1.7776	1.26048		
95	% recovery	0	0.64318408	1.76875622	2.80106667		
96							
97	TOTAL %R	59.119403	67.4696517	76.2816915	40.2669778		
98							
99							
100	*Detection Limits*						
101		*PEG: 10 mg/g*	*TMH: 1 mg/g*	*DMSO: 1 mg/g*			

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300 8	Treated Soil	154 1		Max temp, °C	155
5	1 1:2 mix	200	Reagent	228 9		Soil ID	Wide Beach "A"
6	45% KOH	100	Slurry samps	38 9	list below	Sulfoxide used	DMSO
7	Wash 1	240	Wash 1	266.2			
8	Wash 2	240	Wash 2	253.3			
9	Wash 3	240	Wash 3	222 9			
10	Wash 4	240	Wash 4	264.9			
11	replace cond	183 6	condensate	183 6			
12	bump wash	50					
13	Total Inputs	1794 4	Total Outputs	1612.8			
14	Total inputs -	total outputs	181 6				
15							
16		Samples	Slurry weight	Soil weight			
17	R70108712	072315SJR	4 4	2.2			
18	R70108712	080015SJR	3 1	1.5			
19	R70108712	080115SJR	3 8	1.6			
20	R70108712	080215SJR	6	3 8			
21	R70108712	080315SJR	6 5	4 4			
22	R70108712	080415SJR	5 2	3 9			
23	R70108712	080515SJR	3 8	1 7			
24	R70108712	080615SJR	6 1	3 4			
25		subtotal	38 9	22 5			
26							
27	MASS BALANCE FOR REACTION						
28							
29	Total mass weighed into reactor (in grams)			600 8			
30	Water used to rinse condensor after a bump			50			
31	Total mass of samples removed from reactor			38 9			
32	Total mass in reactor at end of reaction			386 1			
33	Mass of condensate collected from reactor			183 6			
34	*Mass lost during reaction and sampling*			*42.2*			
35							
36	MASS BALANCE FOR SOIL WASHING						
37							
38	Mass in reactor after reaction			386 1			
39	Mass of water added to replace condensate			183 6			
40	Total mass in reactor before filtration			569 7			
41	Mass of reagent recovered			228 9			
42	*Mass in apparatus after reagent removal*			*340.8*			
43							
44	Mass of wash 1 added			240			
45	Mass of wash 1 recovered			266.2			
46	*Mass in apparatus after Wash #1*			*314.6*			
47							
48	Mass of wash 2 added			240			
49	Mass of wash 2 recovered			253.3			
50	*Mass in apparatus after Wash #2*			*301.9*			
51							
52	Mass of wash 3 added			240			
53	Mass of wash 3 recovered			222.9			
54	*Mass in apparatus after Wash #3*			*318.4*			
55							
56	Mass of wash 4 added			240			
57	Mass of wash 4 recovered			264.9			
58	*Mass in apparatus after Wash #4*			*293.5*			
59							
60	Dry soil recovered			154.1			
61	*Mass lost in filtration and soil drying*			*139.4*			
62							
63	*TOTAL LOSSES (SAMPLING, FILTRATION AND DRYING)*				181.6		

	A	B	C	D	E	F	G
64	SOIL AND CHEMICAL MASS BALANCES						
65							
66	SOIL	INPUT	SAMPLES	FINAL			
67	total mass	300 8	NA	154 1			
68	% moisture	23 6	NA	0			
69	dry mass	229 8112	22 5	154 1			
70	% Recovery, dry basis		76 845689				
71							
72	REAGENT INPUT						
73	component	PEG	TMH	DMSO	KOH		
74	mass used	50	50	100	100		
75	% purity	100	100	100	45		
76	Actual g used	50	50	100	45		
77							
78	REAGENT OUTPUT		SAMPLE #	R70108712090105RJR			
79	total mass	228 9					
80	mg/g	243	119	256	10.1		
81	g found	55 6227	27 2391	58 5984	2.31189		
82	% recovery	111 2454	54 4782	58 5984	5 13753333		
83							
84	WASH 1 OUTPUT		SAMPLE #	R70108712090125WJR			
85	total mass	266 2					
86	mg/g		10	28	21.3		
87	g found	0	2 662	7 4536	5 67006		
88	% recovery	0	5 324	7 4536	12 6001333		
89							
90	WASH 2 OUTPUT		SAMPLE #	R70108712090144WJR			
91	total mass	253 3					
92	mg/g		1	5	11.2		
93	g found	0	0 2533	1 2665	2 83696		
94	% recovery	0	0 5066	1 2665	6 30435556		
95							
96	WASH 3 OUTPUT		SAMPLE #	R70108712090219WJR			
97	total mass	222 9					
98	mg/g			2	4.5		
99	g found	0	0	0 4458	1.00305		
100	% recovery	0	0	0 4458	2.229		
101							
102	WASH 4 OUTPUT		SAMPLE #	R70108712090257WJR			
103	total mass	264 9					
104	mg/g				3.4		
105	g found	0	0	0	0.75786		
106	% recovery	0	0	0	1.68413333		
107							
108							
109	TOTAL %R	111.2454	60.3088	67.7643	27.9551556		
110							
111							
112	*Detection Limits*						
113		*PEG: 10 mg/g*	*TMH: 1 mg/g*	*DMSO: 1 mg/g*			

Mass Balance for Wide Beach Reaction #6

	A	B	C	D	E	F	G
1	GRAVIMETRIC MASS BALANCE						
2							
3	Inputs	grams	Outputs	grams		Other Parameters	
4	Untreated Soil	300	Treated Soil	130.1		Max temp, °C	160
5	1:1:2 mix	200	Reagent	147.8		Soil ID	Wide Beach "A"
6	45% KOH	100	Slurry samps	59.5	list below	Sulfoxide used	DMSO
7	Wash 1	303	Wash 1	251			
8	Wash 2	303.2	Wash 2	321.7			
9	Wash 3	304.3	Wash 3	316.9			
10	replace cond +	181.7	condensate	149.8			
11	Total Inputs	1692.2	Total Outputs	1376.8			
12		Input-output	315.4				
13							
14		Samples	g from reactor	Soil weight			
15	R70108801	140436SJR	3.4	3.4			
16	R70108801	150000SJR	3.6				
17	R70108801	150100SJR	3.9				
18	R70108801	150200SJR	4.6				
19	R70108801	150300SJR	6.9				
20	R70108801	150400SJR	7.4				
21	R70108801	150500SJR	4.6				
22	R70108801	150600SJR	6.5				
23	R70108801	150700SJR	4.2				
24	R70108801	150800STG	4				
25	R70108801	150900STG	3.8				
26	R70108801	151000STG	3.1				
27	R70108801	151100STG	3.5				
28		subtotal	59.5	3.4			
29							
30	MASS BALANCE FOR REACTION						
31							
32	Total mass weighed into reactor (in grams)			600			
33	Water used to rinse condenser after bumps						
34	plus water added to replace condensate			181.7			
35	Total mass of samples removed from reactor			59.5			
36	Total mass in reactor before reagent decant			471.3			
37	Mass of condensate collected from reactor			149.8			
38	*Mass lost during reaction and sampling*			*101.1*			
39							
40	MASS BALANCE FOR SOIL WASHING				checked		
41				by calculation	by weighing	cumulative loss	
42					the reactor		
43	Total mass in reactor before decantation			471.3	471.3		
44	Mass of reagent recovered			147.8			
45	*Mass in apparatus after reagent removal*			*323.5*	318.5	5	
46							
47	Mass of wash 1 added			303			
48	Mass of wash 1 recovered			251			
49	*Mass in apparatus after Wash #1*			*375.5*	323.5	52	(spills)
50							
51	Mass of wash 2 added			303.2			
52	Mass of wash 2 recovered			321.7			
53	*Mass in apparatus after Wash #2*			*357*	301.3	55.7	
54							
55	Mass of wash 3 added			304.3			
56	Mass of wash 3 recovered			316.9			
57	*Mass in apparatus after Wash #3*			*344.4*	290.1	54.3	
58							
59	Dry soil recovered				130.1		
60	Water lost in soil drying				160		
61							
62	*TOTAL LOSSES (SAMPLING, WASHING, AND DRYING)*				315.4	=101.1+54.3+160	

	A	B	C	D	E	F	G
63	SOIL AND CHEMICAL MASS BALANCES						
64							
65	SOIL	INPUT	SAMPLES	FINAL			
66	total mass	300	NA	130.1			
67	% moisture	23.6	NA	0			
68	dry mass	229.2	3.4	130.1			
69	% Recovery, dry basis		58.2460733				
70							
71	REAGENT INPUT						
72	component	PEG	TMH	DMSO	KOH		
73	mass used	50	50	100	100		
74	% purity	100	100	100	45		
75	Actual g used	50	50	100	45		
76							
77	REAGENT OUTPUT		SAMPLE #	R70108801151445REM			
78	total mass	147.8					
79	mg/g	221	146	303	5.6		
80	g found	32.6638	21.5788	44.7834	0.82768		
81	% recovery	65.3276	43.1576	44.7834	1.83928889		
82							
83	WASH 1 OUTPUT		SAMPLE #	R70108801151550WEM			
84	total mass	251					
85	mg/g	69	41	96	26.8		
86	g found	17.319	10.291	24.096	6.7268		
87	% recovery	34.638	20.582	24.096	14.9484444		
88							
89	WASH 2 OUTPUT		SAMPLE #	R70108801151645WEM			
90	total mass	321.7					
91	mg/g	18	20	39	8.96		
92	g found	5.7906	6.434	12.5463	2.882432		
93	% recovery	11.5812	12.868	12.5463	6.40540444		
94							
95	WASH 3 OUTPUT		SAMPLE #	R70108801151705WEM			
96	total mass	316.9					
97	mg/g		7	14	6.72		
98	g found	0	2.2183	4.4366	2.129568		
99	% recovery	0	4.4366	4.4366	4.73237333		
100							
101	TOTAL %R	111.5468	81.0442	85.8623	27.9255111		
102							
103							
104	*Detection Limits*						
105		*PEG 10 mg/g*	*TMH 1 mg/g*	*DMSO 1 mg/g*			