



Photograph V-15: A Grid Was Laid Out at the Main Landfarm For Sampling



Photograph V-16: View of the Main Landfarm From the Station Summit

Table V-9: Results of Analyses of Soil Samples for TPH from the Main Landfarm

| Sample Number | Tag Number | Depth (cm) | TPH -Fuel (ug/g) |
|---------------|------------|------------|------------------|
| RI04-599 | 7447 | 30-60 | 2850 |
| RI04-682 | 6767 | 0-20 | 1440 |
| RI04-683 | 6715 | 0-30 | 2550 |
| RI04-684 | 6744 | 0-20 | 3030 |
| RI04-685 | 7458 | 0-50 | 2130 |
| RI04-686 | 6815 | 0-25 | 3830 |
| RI04-688 | 6808 | 0-20 | 3080 |
| RI04-689 | 6731 | 0-30 | 2670 |
| RI04-691 | 6963 | 0-30 | 1840 |
| RI04-692 | 7448 | 0-25 | 5180 |
| RI04-693 | 7448 | 25-50 | 4860 |
| RI04-694 | 6761 | 0-30 | 3520 |
| RI04-695 | 6761 | 30-60 | 4830 |
| RI04-697 | 7505 | 0-50 | 4380 |
| RI04-698 | 6802 | 0-50 | 3640 |
| RI04-699 | 6762 | 0-50 | 4220 |
| RI04-700 | 6771 | 0-50 | 3140 |
| RI04-701 | 6942 | 0-50 | 2490 |
| RI04-702 | 6734 | 0-50 | 4670 |
| RI04-703 | 6801 | 0-50 | 3220 |
| RI04-704 | 6892 | 0-20 | 2520 |
| RI04-705 | 6921 | 0-20 | 2690 |
| RI04-706 | 6736 | 0-30 | 2370 |
| RI04-707 | 6932 | 0-35 | 2980 |
| RI04-709 | 6844 | 0-50 | 2680 |
| RI04-710 | 7444 | 0-50 | 3070 |
| RI04-711 | 6826 | 0-50 | 5550 |
| RI04-712 | 6759 | 0-50 | 5070 |

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| Sample Number | Tag Number | Depth (cm) | TPH -Fuel (ug/g) |
|---------------|------------|------------|------------------|
| RI04-713 | 6770 | 0-50 | 2320 |
| RI04-715 | 6886 | 0-50 | 3180 |
| RI04-717 | 6775 | 0-50 | 3870 |
| RI04-718 | 6775 | 0-50 | 3510 |
| RI04-719 | 6737 | 0-50 | 3540 |
| RI04-720 | 6778 | 0-50 | 3780 |
| RI04-721 | 7447 | 0-50 | 2810 |
| RI04-722 | 6740 | 0-50 | 3930 |
| RI04-723 | 6768 | 0-50 | 2810 |
| RI04-724 | 6737 | 30-70 | 2400 |
| RI04-725 | 6844 | 30-40 | 2760 |

G. Laboratory Landfarm Study

In order to gain a better understanding of the roles of aeration and bioremediation, laboratory studies were initiated in 2003 in order to mimic the field trial plots. The idea was that the experimental setup so developed could then be used to optimize parameters and conditions for rapid remediation of the diesel contaminated fuel at the site and elsewhere in the Canadian Arctic.

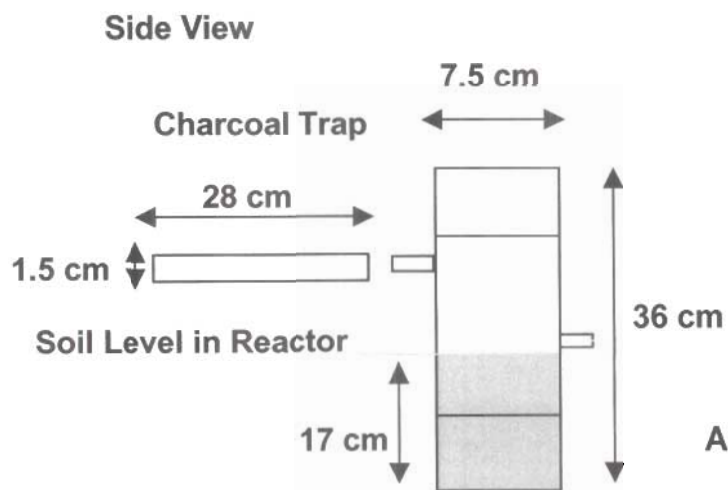
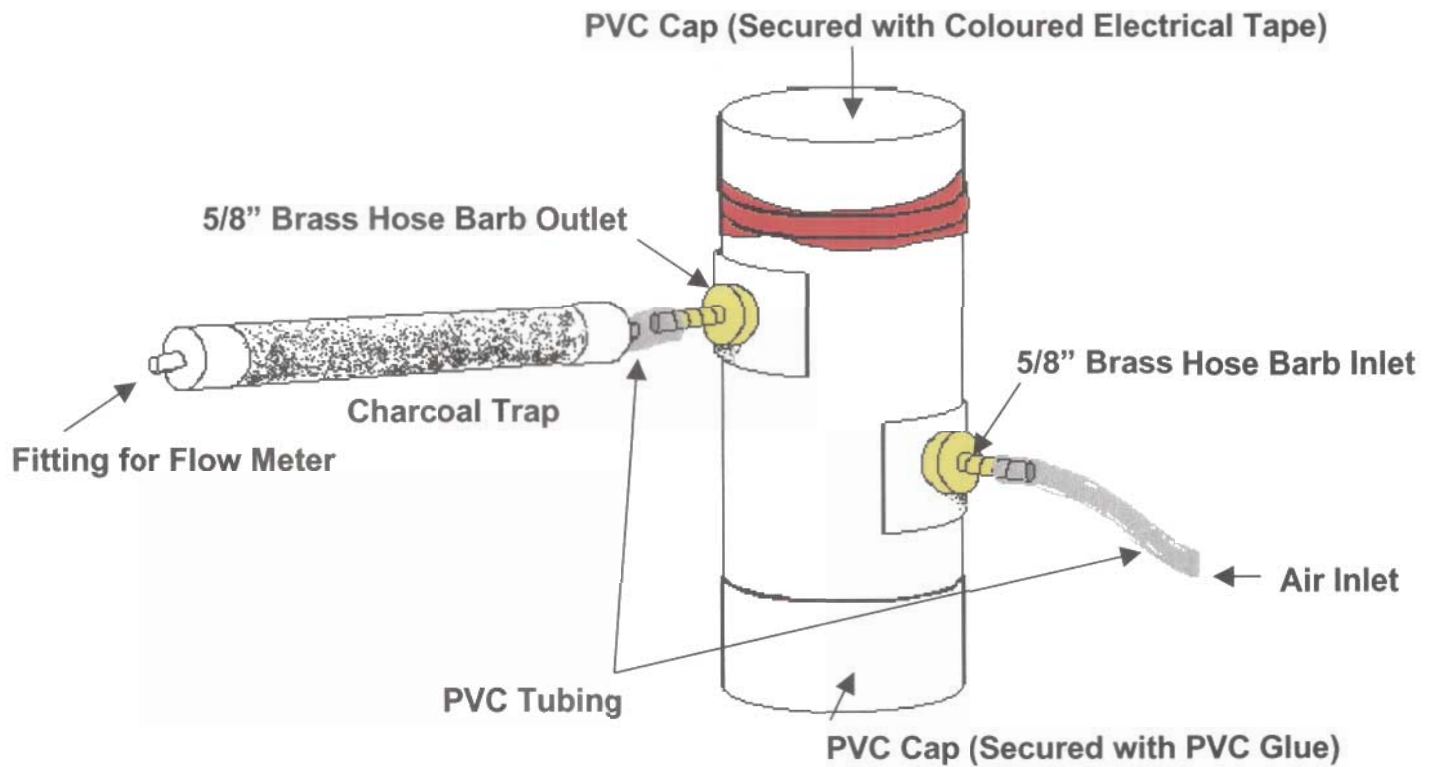
Eighty seven small reactors were constructed for the laboratory experiment. Each contained diesel contaminated soil from Resolution island in a closed vessel. Air was blown through the vessel and any TPH that evaporated was collected on charcoal tubes. Analysis of these charcoal tubes enabled the amount of TPH lost through aeration to be measured. The TPH level in the soil was determined periodically. This permitted the amount of TPH lost to both aeration and bioremediation to be determined. TPH degradation through bioremediation could therefore be calculated and this could also be observed by inspection of the C_{17}/Pr ratios. Three sets of reactors were kept at different temperatures and moisture levels were maintained.

1. First Set of Reactors Experiments

a) Reactor Design

In order to compare the field trial landfarm with a laboratory scale set of reactors, it was decided that the first set of experiments would use the same four regimes as in the field trial. Three sets of 29 reactors were constructed so that the experiments could be carried out at the three temperatures of 18 °C, 8 °C, and 5 °C. Since a large number of reactors were needed suitably priced materials were required. These were obtained from hardware, plumbing or fish tank stores. Figure V-7 shows the individual reactors that were designed and constructed and Photograph V-17 one of the sets of reactors in operation.

Figure: V-7: Diagram of a Laboratory Reactor





Photograph V-17: Some of the Reactors in the Cold Room at 8 C: The Coloured Tape was Used to Distinguish the Different Treatment Regimes

The body of the reactors was constructed of 3 inch PVC sewer pipe cut into 28 cm lengths. One end cap was glued in place while the other removable cap was press fitted by wrapping electrical tape around the top of the tube. Various inserts were added as described below and the assembly pressure tested for leaks. The length of the tube was chosen as 28 cm in order to accommodate the contaminated soil below the mid-point air inlet. In order to simulate the rototilling of the landfarm, tubes were inverted, and then turned back to their original position once all the soil was at the other end. Care was taken to prevent any soil coming in contact with the exit tubes and valve.

The body of the reactors was filled with approximately 1.2 kg of soil. The soil was excavated from near the imploded tank at Resolution Island and shipped back to Queen's University in six coolers. The soil was emptied on to a tarp on the floor of a cold room (8°C), screened to ½ inch and mixed by transferring portions from cooler to cooler. Analysis of the resulting homogenized soil showed that the TPH concentration was about 3700 ppm. Therefore, 1.2 kg of this soil would contain about 4000 mg of TPH. This mass of TPH was of a practical amount, such that if it all volatilized, it could reasonably be collected on granulated activated charcoal (GAC). The TPH concentration was also at the right level to match the field trial and represent TPH levels likely to be encountered in landfarms in the Arctic. Higher levels might be encountered in the field since a 8000 ppm criteria is likely for most contaminated locations but at such high levels it is more difficult to obtain homogeneous TPH levels and such soils are generally more difficult to work with.

Several parameters needed to be set for the experiments. Wind is a factor in removal of TPH by aeration and air is therefore needed to be blown through the reactors to simulate this. It is very difficult to determine the air flow through the apparatus to simulate a particular wind speed. It was decided to use 1 mL/min of air and this was passed in through the reactors at mid-height using a 5/8 inch brass hose barb fitting screwed into the tapped pipe with Teflon tape. The air flowed out the top of the reactor through another brass fitting and then through a GAC trap. Each trap was 20 cm long with an internal diameter of 1.5 cm and filled with approximately 20 g of 12 × 40 mesh granulated activated charcoal from Carbon and Filtration Products, Toronto. Some reactors had additional GAC traps of a smaller size attached so that when TPH was detected on these traps all the main GAC traps were replaced with ones filled with fresh GAC. It was determined through experimentation that 20 g of GAC would capture at least 600 mg of TPH. Air was supplied by compressors located in two buildings at

Queen's University, the Biosciences Complex and Ellis Hall. The air was first passed through 8 m of ¼ inch diameter copper coil in ensure that it was at the temperature of the reactors before entering them. Then it passed into a manifold constructed of 4 inch ABS pipe into which 29 valves were fitted. The valves were aquatic air regulators. Each could be adjusted to maintain the correct air flow. Air flow was carefully measured and maintained on a daily basis using a portable flow meter which was attached in turn to the end of each GAC tube.

Moisture content of the soil had to be controlled otherwise the soil would dry out and bioremediation would be adversely affected. It was decided to control the moisture content between 10 % and 15 % in order to promote bioremediation: the original soil had a moisture content in this range. A ball valve was glued into position at the top of each reactor. After determination of moisture content of the reactors, an appropriate amount of deionised water was introduced into the reactors to return the moisture content to 15 %. If the moisture content approached 10 % or fell below that target, then water was added. Problems were encountered with the 8 °C set as the air supply initially contained too much moisture. This problem was solved by drying the air with molecular sieves before it reached the air manifold.

Three sets of 29 reactors were attached to each of three manifolds and set up in three different locations at different temperatures. These temperatures were 18 °C, 8 °C, and 5 °C. These temperatures were chosen to cover the range that might be expected for soils in the Arctic and were also determined by the availability of cold rooms at specific temperatures. The sets were set up on 6 July 2004. Each set of 29 reactors was composed as follows:

- Six reactors for a control set. These were not rotated.
- Six reactors that were rotated every day.
- Six reactors that were rotated every four days.
- Six reactors to which fertilizer was added and these were also rotated every four days. The amount of fertilizer added was 0.710 g of urea and 0.112 g of diammonium phosphate (DAP) per reactor.

- Three reactors to which silver nitrate was added and these were also rotated every four days. Silver nitrate was added to each tube for a final concentration of 0.3 %.
- Two reactors to which fresh diesel was added to slightly TPH contaminated Resolution Island soil. These were rotated every four days.

The amount of fertilizer added was based on the ratio of C:N:P of 100:7.5:0.5. Three had silver nitrate added to them. The silver will inhibit bioremediation so that for these three reactors it was known that no bioremediation was occurring. We were interested in knowing whether the weathered diesel fuel found in the Resolution Island soil would behave in the same way as fresh diesel fuel; some literature reports had suggested otherwise. A batch of Resolution Island soil which contained <100 ppm TPH was spiked with diesel fuel in a methanol solution. After thorough mixing, the methanol was allowed to evaporate overnight in a fume hood to give a final TPH concentration of about 5000 ppm. This soil was placed in the two remaining reactors in each set.

Maintaining the three sets of reactors required considerable time and effort. The soil in each reactor was analysed periodically by shutting off the air supply, removing the lid and rapidly taking about 10 g of soil on a spatula and placing it in a pre-weighed conical flask which was then sealed. Each set of soils was then immediately extracted and analysed. Moisture content was initially measured in all reactors or a sub-section of the reactors approximately once a week. Once the reactors had been in operation for some time, the rate of loss of water from the soil at the three different temperatures could be fairly accurately estimated. Similarly the rate at which TPH was collected on the GAC traps was found to be predictable after an initial period of several weeks. The traps were replaced three or four times during the course of the experiment depending on the temperature. Each day six reactors had to be rotated per set and the air flow was checked on all reactors daily. Every fourth day, twenty three reactors were rotated. The experiment was operated for about 150 days per set at which time it was judged that enough data had been collected to allow useful deductions to be made and thus allow a second set of experiments to be initiated.

Problems with initial TPH measurements were encountered because the silica tubes used to remove naturally occurring organic materials were defective. As a result no TPH data on the original soil samples was available. However, since no significant bioremediation occurred in most reactors, the original TPH concentration could be

calculated by adding the amount trapped on the GAC tubes which was accurately known. This was also checked by plotting the TPH in soil as a function of time, assuming the rate of loss of TPH was linear, and determining the intercept at time zero. The average initial diesel concentration was determined to be 3710 ± 730 ppm; the 18 °C fertilized set was not used to obtain this average because significant bioremediation was occurring in this set. It should also be mentioned here that it is very difficult to obtain a homogeneous mixture of TPH contaminated soil. Generally, the variability of results of 25 % or more can be expected. Results presented here are the average of the six or, for the silver added set, three results.

Results from the two reactors per set in which fresh diesel were added did not give any definitive results and are therefore are not presented here. This is probably due to the heterogeneity of the samples and the fact that only two replicates were used.

b) GAC Traps

The amount of TPH found on the charcoal traps after each replacement with fresh tubes is reported in Tables V-10A, V-10B and V-10C for the three temperatures at which the experiment was conducted. In each table the TPH collected each day in the period for which the charcoal tube was in place has been calculated. Table V-11 shows the average total TPH collected for each set of reactors at each temperature. As would be expected the amount of TPH aerated increased as the temperature increased and also increased with the frequency of rotating the tubes. Thus, for the six control samples averaged in the first line of Table V-11, the amount of TPH aerated for the three temperatures of 18 °C, 8 °C, and 5 °C were 1200, 460 and 390 mg respectively. Also in the first column of data in Table V-11, one observes that for the 18 °C reactor sets, the amount of TPH aerated was 1200 mg for the control samples, 2270, 1960 and 1990 for the three sets rotated every four days and 2580 mg for the set rotated every day. Similar trends are seen for all sets. In Tables V-10, the rate of aeration can be seen to decrease with time by inspecting the amounts of TPH aerated each day. This is to be expected as the amount of TPH remaining decreases and the volatility of the remaining TPH also decreases. For the fertilized set at 18 °C this decrease with time is more pronounced as significant bioremediation occurred in this set; this decreased the TPH level and thus the amount of TPH left to evaporate. Thus in Table V-10A one sees the rate of TPH loss per day decreases from 21 mg/day down to 5 mg/day whereas for the other two sets that were rotated every four days this decrease is not so pronounced. This effect is also seen to a

lesser degree for the 8 °C set but not for the 5 °C set, pointing to a decrease in bioremediation in the fertilized soil as the temperature decreased.

Table V-10A: Mass of TPH Collected on Charcoal Traps in the Reactors at 18 °C from First Set of Experiments

| Reactor Type | Mass of TPH Collected During Experiment per Day (mg) | | | | |
|--------------|--|------------|------------|-------------|--------------|
| | Days From Start of Experiment | | | | |
| | 1-30 days | 31-51 days | 52-77 days | 78-126 days | 127-169 days |
| Control | 9 | 6 | 4 | 10 | 5 |
| Everyday | 24 | 17 | 16 | 16 | 8 |
| Every 4 days | 22 | 11 | 11 | 14 | 9 |
| Fertiliser | 21 | 12 | 11 | 10 | 5 |
| Added Silver | 16 | 8 | 8 | 15 | 10 |

Table V-10B: Mass of TPH Collected on Charcoal Traps in the Reactors at 8 °C from First Set of Experiments

| Reactor Type | Mass of TPH Collected During Experiment per Day (mg) | | | |
|--------------|--|------------|-------------|--------------|
| | Days From Start of Experiment | | | |
| | 1-36 days | 37-90 days | 91-142 days | 143-169 days |
| Control | 2 | 2 | 2 | 6 |
| Everyday | 11 | 7 | 11 | 3 |
| Every 4 days | 5 | 9 | 7 | 5 |
| Fertiliser | 11 | 7 | 5 | 3 |
| Added Silver | 6 | 7 | 8 | 7 |

Table V-10C: Mass of TPH Collected on Charcoal Traps in the Reactors at 5 °C from First Set of Experiments

| Reactor Type | Mass of TPH Collected During Experiment per Day (mg) | | | |
|--------------|--|-------------|--------------|--------------|
| | Days From Start of Experiment | | | |
| | 1-49 days | 50-111 days | 112-145 days | 146-169 days |
| Control | 2 | 2 | 2 | 3 |
| Everyday | 10 | 8 | 10 | 6 |
| Every 4 days | 8 | 6 | 12 | 3 |
| Fertiliser | 10 | 5 | 8 | 3 |
| Added Silver | 9 | 5 | 10 | 3 |

Table V-11: Summary of Total Mass of TPH Collected on Charcoal Traps in the Reactors from First Set of Experiments

| Reactor Type | Total Mass of TPH Collected on Charcoal Traps (mg) | | |
|--------------|--|------|------|
| | 18 °C | 8 °C | 5 °C |
| Control | 1200 | 460 | 390 |
| Everyday | 2580 | 1420 | 1510 |
| Every 4 days | 2270 | 1180 | 1210 |
| Fertiliser | 1960 | 1100 | 1160 |
| Added Silver | 1990 | 1170 | 1200 |

c) Soil TPH Levels

The TPH levels at the start and end of the experiment, as presented in Table V-12, were calculated as previously discussed. Most show a decrease with time, with the control samples, which were not rotated, showing the least decrease of the four regimes. The fertilized reactors clearly exhibited the largest decrease in TPH concentration over the course of the experiment for all temperatures.

Table V-12: Summary of TPH Concentrations in the Reactors at the Start and End of the First Set of Experiments

| Reactor Type | TPH Concentration in Soil in the Reactors (ppm) | | | | | |
|--------------|---|------|-------|------|-------|------|
| | 18 °C | | 8 °C | | 5 °C | |
| | Start | End | Start | End | Start | End |
| Control | 3710 | 2530 | 3710 | 3880 | 3710 | 3190 |
| Everyday | 3710 | 1660 | 3710 | 2960 | 3710 | 1920 |
| Every 4 days | 3710 | 1750 | 3710 | 3200 | 3710 | 2430 |
| Fertiliser | 3710 | 810 | 3710 | 2390 | 3710 | 1830 |
| Added Silver | 3710 | 2250 | 3710 | 3570 | 3710 | 1790 |

The amounts of TPH in the reactors can be calculated from the TPH concentrations and masses of soil in each reactor. These differences between the starting mass and final mass, which represent the loss of TPH by all mechanisms, are presented in Table V-13. The results show that the fertilised reactors lose the largest mass of TPH and that daily aeration is the next best treatment. However, at 5 °C and 8 °C they are generally not very useful or informative in showing any trends. This is due to the large error in each value in Table V-13 associated with the difficulty in obtaining representative samples from the reactors for analysis and in obtaining homogeneous soil. The initial concentration in all reactors was 3710 ppm with a standard deviation of 730 ppm. Since the numbers in Table V-13 are the difference between two concentrations and two times the standard deviation is often used to represent the precision of data, it is not surprising that the error associated with these numbers is high. However the reactors clearly show that by aerating every 4 days and fertilizing, the TPH contaminated soil can be remediated at all 3 temperatures. In approximately 5 months, 78 % of the TPH had been remediated from the soil at 18 °C. The TPH in the soil at 8 °C and 5 °C was

remediated 36 % and 51 % respectively. The remediation at these lower temperature is important because it demonstrates that this regime is applicable to sites with colder temperatures than Resolution Island.

Table V-13: Summary of Total Mass of TPH Lost From the Reactors During the First Set of Experiments

| Reactor Type | Total Mass of TPH Lost from the Reactors (mg) | | |
|--------------|---|--------|------|
| | 18 °C | 8 °C | 5 °C |
| Control | 1540 | -220 * | 670 |
| Everyday | 2670 | 980 | 2320 |
| Every 4 days | 2550 | 670 | 1670 |
| Fertiliser | 3770 | 1710 | 2450 |
| Added Silver | 1900 | -260 * | 2490 |

* negative numbers are left to show variability in TPH soil data

d) Aeration Versus Bioremediation

The mass of TPH lost through aeration is presented in Table V-11 and the mass lost through both aeration and bioremediation given in Table V-13. Table V-14 gives the differences in these masses, which might be considered to represent the mass of TPH lost due to bioremediation. The fertilizer added reactors at 18 °C showed the highest value where one would expect bioremediation to be most active. Unfortunately these numbers are not as meaningful at the 2 lower temperatures as one might have hoped for reasons discussed above.

Table V-14: Calculated Masses of TPH Lost Through Bioremediation

| Reactor Type | Calculated Difference in Mass of TPH From Total Mass Loss From TPH Soil Levels and Mass Collected on GAC Tubes (mg) | | |
|--------------|---|---------|------|
| | 18 °C | 8 °C | 5 °C |
| Control | 340 | -690 * | 280 |
| Everyday | 80 | -450 * | 810 |
| Every 4 days | 280 | -510 * | 450 |
| Fertiliser | 1810 | 610 | 1290 |
| Added Silver | -90 * | -1430 * | 1290 |

* negative numbers are left to show variability in TPH soil data

It is clear from the masses of TPH collected on the GAC traps that the fertilized reactors show a change that can be attributable to bioremediation. This can be further explored by examining the C_{17}/Pr ratios and results are presented in Figures V-8 and V-9. Figure V-8 gives the results for the 8 °C set. As can be seen there is no change in the C_{17}/Pr ratio for any of the sets except the reactors to which nutrients were added which show a steady decrease in this ratio. Table V-9, which plots the change in the C_{17}/Pr ratios with time for all the every 4 day reactors and the fertilized reactors at all three temperatures, shows this even more effectively. None of the every 4 day reactors show a change in the C_{17}/Pr ratios while the fertilized reactors all show a decrease. Moreover, this decrease is faster and more pronounced for the fertilized reactors at 18 °C while the 8 °C set shows a slower decrease and the 5 °C set an even slow change.

Figure V-10 displays chromatograms obtained from the initial soil placed in the reactors and soil at the end of the experiment from the aerated daily reactors and the fertilizer added reactors. The plots, as presented, are on the same vertical and horizontal scales. For the aerated daily reactors there is a decrease in peak size due to loss of material but no readily discernable change in pattern. For the fertilized reactors there is a noticeable shift to lighter hydrocarbons as well as a decrease in intensity.

The first set of experiments therefore show that TPH is lost in the fertilized reactors by both aeration and bioremediation while all other reactors only exhibit loss through aeration. These experiments clearly show that both mechanisms are occurring even at temperatures as low as 5 °C and support the results obtained in the field study at Resolution Island

Figure V-8: Change of C_{17}/Pr Ratio with Time for the 8°C Reactor Set

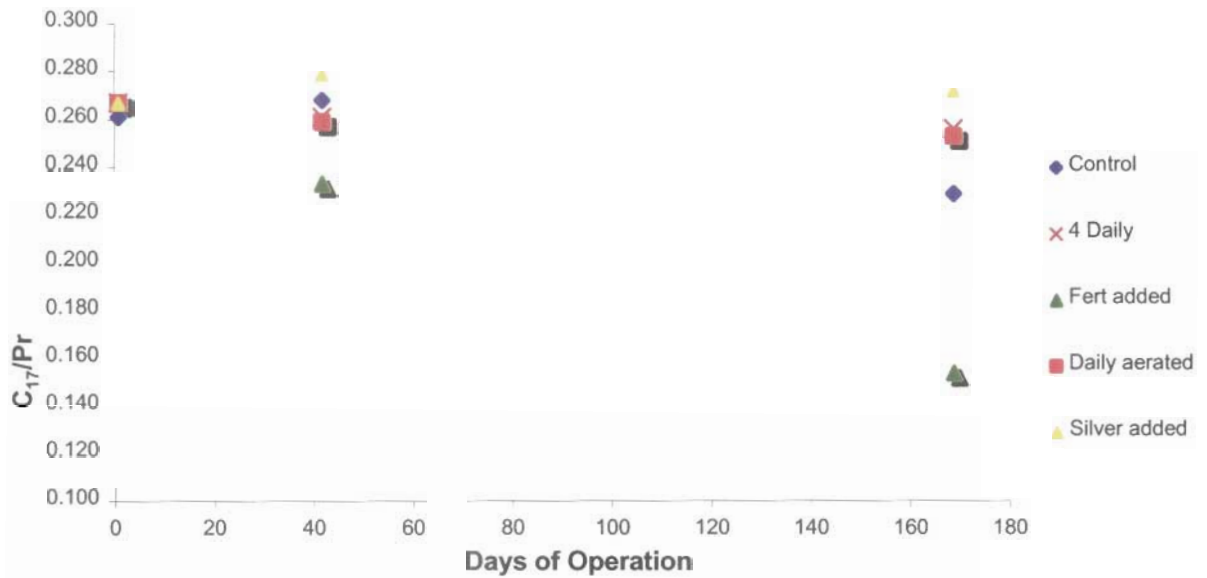


Figure V-9: Change of C_{17}/Pr Ratio with Time for Every Four Days and Fertiliser Added Reactor Sets

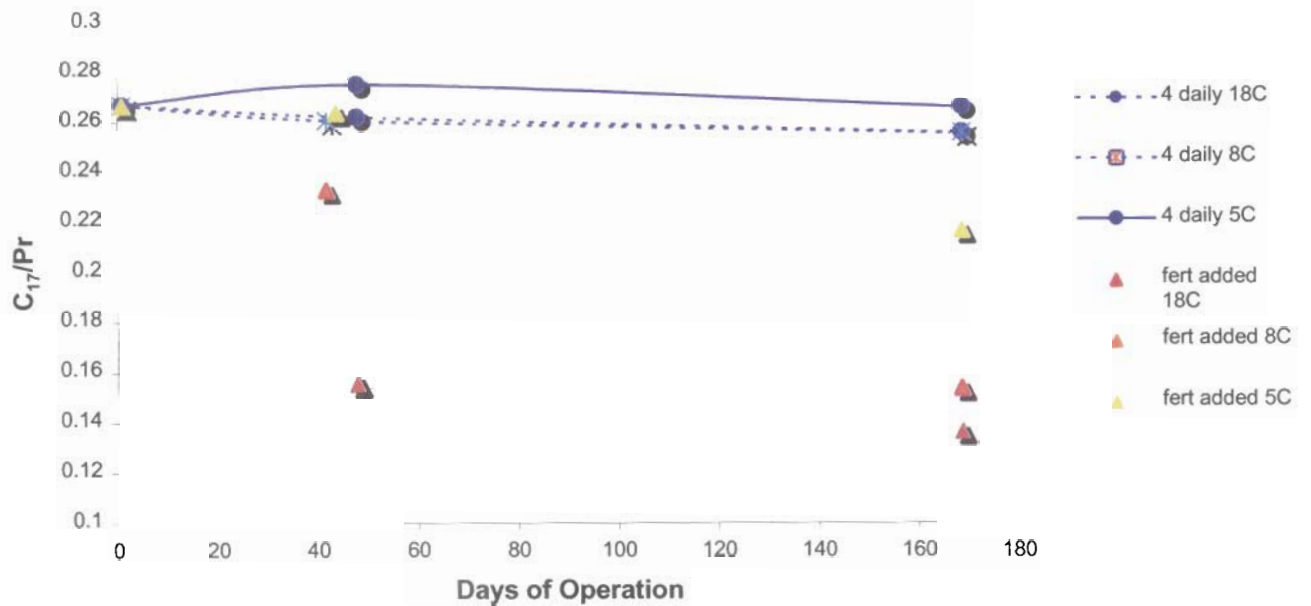
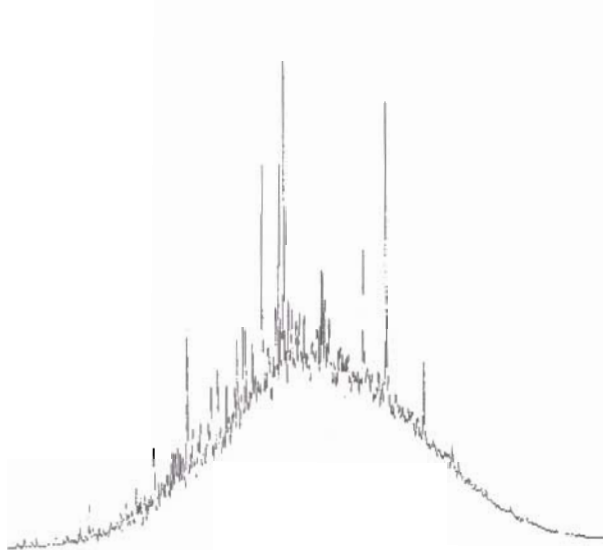
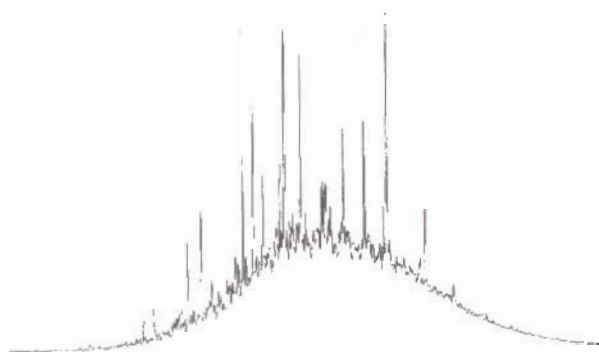


Figure V-10: Chromatograms of the TPH Present in Soil From the Reactors a) Initially, b) Aerated Daily after 169 days and c) Fertilizer Added After 169 Days.

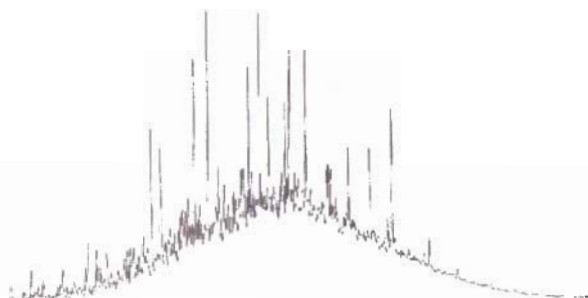
(a)



(b)



(c)



2. Second Set of Reactors Experiments

The second set of laboratory reactor experiments are designed to confirm results found in the first set and to explore the possibility of in situ bioremediation. The sets of reactors were set up on 14 February 2005.

TPH contaminated soil was excavated from > 8000 ppm area near the imploded tank on 25 August 2004 and shipped back to the laboratory where it was stored at - 18 °C. In early January it was removed to an 8 °C cold room and allowed to thaw. On 9 February when the temperature in Kingston was near the freezing point, the soil was emptied into a robust child's paddling pool and thoroughly mixed. The reactors were then filled and the experiment initiated on 14 February 2005 at which time each was sampled. Results of the analyses of the 72 samples so obtained showed that the soil was relatively homogeneous and somewhat more contaminated than the first set with a concentration of 7200 ± 920 ppm TPH.

Three sets of 24 reactors were attached to each of the three manifolds and set up in three different locations at 18 °C, 8 °C, and 5 °C. Each set of 24 reactors was composed as follows:

- Four reactors for a control set. These were not rotated.
- Four reactors with fertilizer for a control set. These were not rotated.
- Four reactors that were rotated every four days
- Four reactors with fertilizer that were rotated every four days
- Four reactors that were rotated every twelve days
- Four reactors with fertilizer that were rotated every twelve days

The rotation schedule was chosen to investigate the effect of longer times between rototilling on the rate of loss of TPH. It would obviously be beneficial to operate a large scale landfarm with longer periods between aeration and be more practical especially in the case of remote, uninhabited Arctic sites. Results from the first reactor set established that the addition of fertiliser enhanced bioremediation, duplicating the result found in the field. Ideally one would like not to have to excavate fuel contaminated soils but to treat them in place by stimulating bioremediation by the in situ application of nutrients. The

experiment is also designed to explore this possibility at three different temperatures. It is also planned to conduct experiments in the field to investigate this near the Beach POL tanks.