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Treatability Study of the Graces
Quarters Contaminated Aquifer
Using Vitamin B12-Catalyzed
Reductive Dechlorination Part I:
Microcosms

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NWRI Contribution No. 99-224

**Treatability study of the Graces Quarters Contaminated Aquifer using
Vitamin B12-Catalyzed Reductive Dechlorination Part 1: Microcosms**

Final Report

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MANAGEMENT PERSPECTIVE

- Title:** **Treatability Study of the Graces Quarters Contaminated Aquifer using Vitamin B12-Catalyzed Reductive Dechlorination. Part 1: Microcosms.**
- Author(s):** **S. Lesage, K. Millar, S. Brown, and H. Steer**
- NWRI Publication #:**
- Citation:** **Report to Dames and Moore subcontract number BAL-S-0198-0121 on U.S. DOE contract DEAC05-84OR21400**
- EC Priority/Issue:** This work is being conducted under the "Clean Environment" issue. The paper presents the results of a contract obtained from the U.S. Department of Energy through Dames and Moore Inc. to conduct a treatability study for groundwater at from Graces Quarters, Aberdeen Proving Ground, a U.S. Military research facility in Maryland. This contract was obtained to test the vitamin B12 technology for the remediation chlorinated compounds, for which a patent was received last year.
- Current Status:** The microcosms study is complete. It was followed by a column test which is a separate report.
- Next Steps:** This work was conducted as part of a larger feasibility study to evaluate all existing technologies at the site. The results were very positive and the technology has been selected for a pilot scale study to be conducted at the site. The project, which is in the planning stage, will be conducted by Dames and Moore, with our technical advice.

ABSTRACT

The use of vitamin B12 reduced by titanium (III) citrate for the reductive dechlorination of chlorinated aliphatic hydrocarbons has been the subject of numerous microcosm studies, many suggesting possible *in situ* field applications. However, most of the work has been conducted in the aqueous phase with carbon tetrachloride or tetrachloroethene. This paper describes a laboratory treatability study consisting of eight treatment conditions conducted with aquifer material and contaminated site groundwater from Graces Quarters, Aberdeen Proving Ground, MD. The purpose of the study was to look at the effect of combining the use of vitamin B12 and titanium citrate with various carbon sources to evaluate the combination of chemical and biological treatments. The site groundwater is contaminated with a mixture of 1,1,2,2-tetrachloroethane, carbon tetrachloride, chloroform, tetrachloroethene, and trichloroethene.

The eight treatment conditions evaluated were: (1) vitamin B12 (10 mg/L), titanium citrate, and glucose; (2) titanium citrate and glucose; (3) titanium citrate and yeast extract; (4) γ -irradiated sterile control with vitamin B12 (10 mg/l), titanium citrate, and glucose; (5) glucose only; (6) vitamin B12 (10 mg/l), titanium citrate and glucose, with yeast extract added after 2 days; (7) an alternate chelating agent and vitamin B12; and (8) vitamin B12, titanium citrate, and yeast extract.

Each treatment combination was conducted using site aquifer material and highly contaminated groundwater (2000 - 4000 $\mu\text{g/L}$) or less contaminated groundwater (<1,000 $\mu\text{g/L}$). Each was run in triplicate, for a total of six active microcosms per treatment condition. In addition, two microcosms consisting of aquifer material and highly or less-contaminated groundwater (with nothing else added) were used as controls for each microcosm condition. In addition to monitoring for the chlorinated hydrocarbons, the microcosms were monitored for titanium, methane, ethane, ethene, acetylene, glucose, citrate, lactate, acetate, and propionate.

This series of experiments showed that the vitamin B12/titanium citrate reaction was effective in treating all the compounds initially present in the site groundwater. When vitamin B12 was present, chlorinated alkanes were fully degraded within 5 minutes, whereas the half-life for tetrachloroethene was 33 minutes. The final products of the 1,1,2,2-tetrachloroethane degradation were *cis*- and *trans*-DCE, which remained above their maximum acceptable concentration after 28 days. Some vinyl chloride was formed, but did not persist. First order reaction rate constants were not dependant upon contaminant concentration. The addition of titanium citrate alone, without vitamin B12, did degrade the chlorinated methanes and ethanes (but not the ethenes), but at a much reduced rate. The γ -irradiated control showed that most of the dechlorination observed was abiotic; however, vinyl chloride degraded more rapidly in the non-sterilized microcosms. In the treatment containing glucose only, carbon tetrachloride was dechlorinated to chloroform, but no other reaction was observed. This means that the addition of a carbon source alone would not be sufficient to induce bacterial populations capable of degrading all the contaminants. The degradation of glucose under these anerobic conditions produced large amounts of CO_2 , which contributed to the removal of some of the chlorinated products.

List of abbreviations

| | |
|-------|--|
| B12 | Vitamin B12 |
| DCE | dichloroethene |
| DCM | dichloromethane |
| DUP | duplicate |
| ECD | electron capture detector |
| FID | flame ionization detector |
| GC/MS | gas chromatography/mass spectrometry |
| L | liter |
| MDL | method detection limit |
| mM | millimolar |
| PERC | perchloroethylene or tetrachloroethene |
| ppm | parts-per-million |
| PQL | practical quantitation limit |
| TCD | thermal conductivity detector |
| TCE | trichloroethene |
| TeCA | 1,1,2,2-tetrachloroethane |
| µg | microgram |
| VC | vinyl chloride |
| VFA | volatile fatty acid |

Table of Contents

| | |
|--|-----------|
| <i>i. List of Figures</i> | <u>4</u> |
| <i>ii. List of Tables</i> | <u>4</u> |
| <i>I. Introduction</i> | <u>5</u> |
| <i>II. Methods</i> | <u>8</u> |
| <i>III. Results</i> | <u>10</u> |
| 1. Preliminary experiments | <u>10</u> |
| 2. Microcosm 1 (titanium citrate, 10 mg/L vitamin B12 and glucose) | <u>10</u> |
| 3. Microcosm 2 (titanium citrate and glucose) | <u>17</u> |
| 4. Microcosm 3 (titanium citrate and yeast extract) | <u>21</u> |
| 5. Microcosm 4 (γ irradiated soil -vitamin B12, titanium citrate and glucose) | <u>24</u> |
| 6. Microcosm 5 (glucose) | <u>27</u> |
| 7. Microcosm 6 (vitamin B12, titanium citrate and glucose, yeast extract added after 2 days) | <u>30</u> |
| 8. Microcosm 7 (vitamin B12, new titanium chelate) | <u>32</u> |
| 9. Microcosm 8 (Titanium citrate, yeast extract, vitamin B12) | <u>35</u> |
| 10. Post-microcosm reactions | <u>38</u> |
| <i>IV. Conclusions</i> | <u>39</u> |
| <i>V. References</i> | <u>40</u> |
| <i>VI. Appendix A</i> | <u>41</u> |

i. List of Figures

| | <i>Page</i> |
|-----------|---|
| Figure 1 | Reactions pathways for the reaction of the site contaminants with vitamin B12 and titanium citrate _____ 7 |
| Figure 2 | Summary of results, Microcosm 1a _____ 12-13 |
| Figure 3 | Average glucose and citrate concentrations for the Q52 and Q14 microcosms _____ 14 |
| Figure 4 | Volatile fatty acids (averages, full data sets in Appendix 1) _____ 16 |
| Figure 5 | Microcosms 1a. Beginning and after 28 days. The small bottle is the supernatant of F that exploded. _____ 17 |
| Figure 6 | Summary of results, Microcosm 2 _____ 18-19 |
| Figure 7 | Summary of results, Microcosm 3 _____ 22-23 |
| Figure 8 | Summary of results, Microcosm 4 _____ 25-26 |
| Figure 9 | Summary of results, Microcosm 5 _____ 28-29 |
| Figure 10 | Summary of results, Microcosm 6 _____ 31 |
| Figure 11 | Summary of results, Microcosm 7 _____ 33-34 |
| Figure 12 | Summary of results, Microcosm 8 _____ 36-37 |

ii. List of Tables

| | |
|---------|--|
| Table 1 | Experimental Design – Microcosms _____ 9 |
|---------|--|

I. Introduction

This report summarizes the results that were obtained by conducting a series of microcosms for the treatment of groundwater from the Graces Quarter area at Aberdeen Proving Grounds. The use of vitamin B12 as part of an integrated chemical/biological treatment train was being envisaged because of the suitability of the contaminants at the site for the vitamin B12- catalyzed reaction.

In this project, combinations of chemical and biological treatments were conducted in microcosms containing contaminated water from two areas at the site, representing high (Q14) and low (Q52) concentrations of contaminants, in the presence of aquifer material from a single source at the site.

The experimental design was developed in collaboration with the supervising group. The purpose of the different treatments was to establish the rate of reaction of the different contaminants with titanium citrate with and without vitamin B12 and in combination with carbon sources to determine the possibility of natural attenuation after the chemical treatment.

In the vitamin B12/titanium citrate process, vitamin B12 is a catalyst, meaning that it enhances the reaction, but is not consumed by it. The titanium (III) citrate, on the other hand, provides the electrons for the reduction of the chlorinated compounds. It is therefore consumed and must be replenished. We have developed an alternative technique for field use based on titanium metal (Lesage et al., 1997). This method was used throughout the experiment.

The principal contaminants in the site water are: 1,1,2,2-tetrachloroethane (TeCA), carbon tetrachloride, chloroform, tetrachloroethene (PERC) and trichloroethene (TCE). The vitamin B12 reaction produces sequential reductive dechlorination of all of these compounds. A schematic diagram of the degradation pathways is shown on Figure 1.

Reductive dechlorination may be caused by titanium citrate alone or be catalysed by vitamin B12. The ease of dechlorination is governed by the oxidation state of the carbon. Therefore carbon tetrachloride is the most reactive, followed by chloroform and TeCA. All of these compounds can be dechlorinated by reduced titanium only, but the rate of reaction is enhanced by the presence of B12. The ethenes are much less reactive and require the presence of vitamin B12. The rate of reaction also decreases with the degree of chlorination. Because the reaction medium is buffered to pH 8, some base-catalyzed elimination of HCl may occur with the chloroethanes.

This unique combination of contaminants makes the site an ideal candidate for remediation using an integrated chemical/biological approach based on the use of vitamin B12. In the proposed scenario, the vitamin B12/titanium citrate would be injected in the zones of high contamination to reduce the concentration of the chlorinated solvents to a level that could be tolerated by bacteria. A carbon source is included in the treatment such that bacteria will be able to proliferate down gradient. The vitamin B12 treatment has been shown to be non-toxic and compatible with the growth of anaerobic bacteria (Lesage et al., 1996). This integrated approach is expected to reduce the cost of treating a plume.

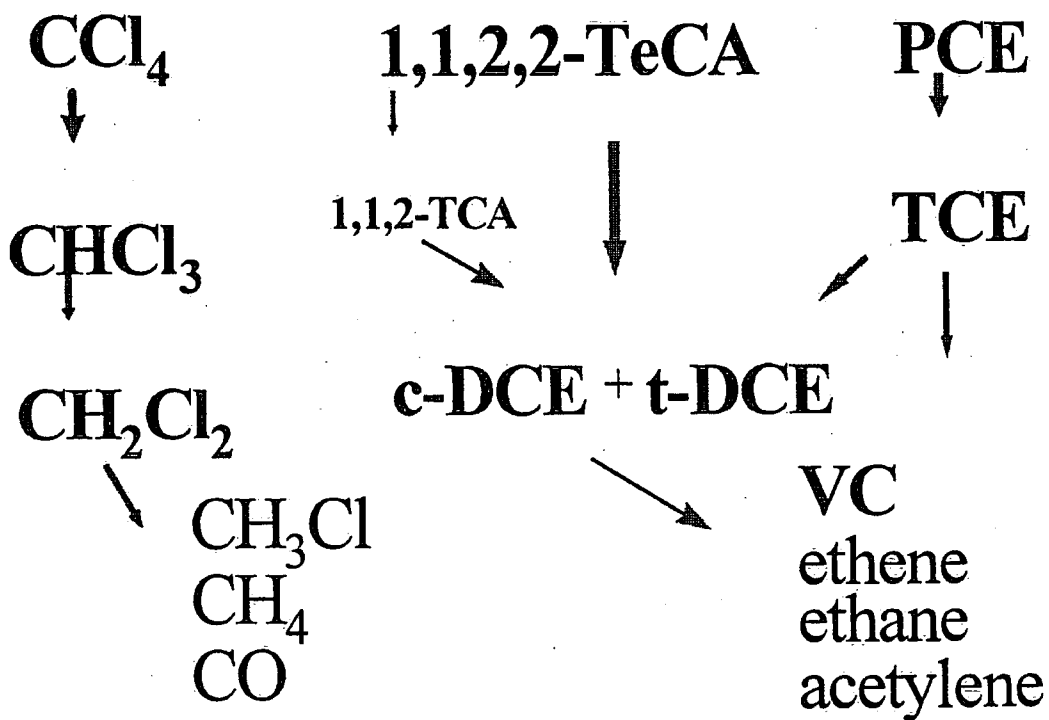


Figure 1: Reactions pathways for the reaction of the site contaminants with vitamin B12 and titanium citrate.

II. Methods

The experimental methods are described in details in the Work Plan and will only be summarized here.

The microcosms were conducted in sealed 160 mL serum bottles containing 100 g of aquifer material from the site and 100 mL of site water. The bottles were incubated inverted at room temperature. Each treatment was carried out in triplicate with Q52 (low) and Q14 (high) site water, except for M7 and M8 where only low site water was used. In addition, because the site water samples had not been composited and there was potential for variability between bottles, two references containing water from the same bottles as the treatment, but no reagents, were setup as on-going controls.

The eight treatments used, their purpose and the monitoring schedule are listed in Table I.

All the microcosms were monitored using an SRI GC equipped with dual columns leading to an electron capture detector (ECD) and a TCD/FID in series. This instrument was used to monitor the headspace of the microcosms for all the chlorinated volatile compounds and their known degradation products, except CO. Small liquid samples were withdrawn to analyze for citrate and the volatile fatty acids that are known biological degradation products of glucose and citrate. Glucose itself was tested using an EncoreTM glucometer .

Table 1: Experimental design- Microcosms.

| <i>Treatment (Each at 2 substrate concentrations)</i> | <i>Purpose</i> | <i>Chemical Analyses and Schedule</i> |
|---|---|---|
| 1. vitamin B12 (10 mg/L) with titanium citrate and glucose | optimal treatment | Days: 0, 1, 3, 7, 15, 30 |
| 2. titanium citrate and glucose | control for reducing conditions | Days: 0, 1, 7, 19, 29 |
| 3. titanium citrate and yeast extract | to promote methanogens | Days: 0, 1, 4, 16, 28 |
| 4. γ -irradiated with vitamin B12, titanium citrate and glucose | abiotic control | Days: 0, 1, 3, 7, 16, 28 |
| 5. glucose | Biologically active control | Days: 0, 5, 14, 27, 48 |
| 6. vitamin B12 (10 mg/L) with titanium citrate and glucose. Yeast extract after 2 days. | to establish a methanogenic population after TeCA has been reduced by B12 | Days: 0, 1, 2, 6, 14, 29 |
| 7. Alternate chelator with vitamin B12 | - hopefully not as easily biodegraded as citrate | Days: 0, 1, 3, 7, 15, 28 |
| 8. vitamin B12 (10 mg/L) with titanium citrate and yeast extract | similar to 3, but with vitamin B12 - again to promote methanogens | Days: 0, 1, 3, 7, 15, 28 |

III. Results

1. Preliminary experiments

Before beginning the actual experiments, it was necessary to establish the amount of titanium necessary for the reduction of the aquifer solids. Based on experience elsewhere, three rinses with a concentrated titanium citrate solution had been sufficient. Also, no data existed on the effect of titanium citrate alone on carbon tetrachloride and TeCA. It was therefore necessary to establish the effect of the treatment on site water alone.

It was found that only measuring the Eh was not sufficient to evaluate the long term viability of the treatment. Instead, the amount of iron (as Fe^{+2}) was found to be a more useful indicator of the redox status of the aquifer material. The measurement was done using colorimetric test strips. The aquifer material was judged as being sufficiently reduced when the supernatant contained less than 50 ppm Fe. This required six rinses with 30 mM Titanium (III) citrate on average. Based on the very high reducible Fe content of the aquifer material, a full scale system where all the aquifer material would have to be reduced would be cost prohibitive. Nonetheless, the experiments were still carried out with the planned amounts of solids, because combined chemical/biological treatment scenarios were to be evaluated.

2. Microcosm 1 (titanium citrate, 10 mg/L vitamin B12 and glucose)

The purpose of microcosm 1 was to see the effect on the site water of the vitamin B12/titanium citrate using conditions that had been found optimal for the degradation of PERC. The results obtained are to be used in the design of the column experiments. The results are summarized in graph in Figure 2, and the complete results are shown in Appendix A. Within one day, all the tetrachloroethane (TeCA), carbon tetrachloride and tetrachloroethylene (PERC), were below detection limit. Some chloroform, dichloromethane and chloromethane were apparent on day 0, but had disappeared by

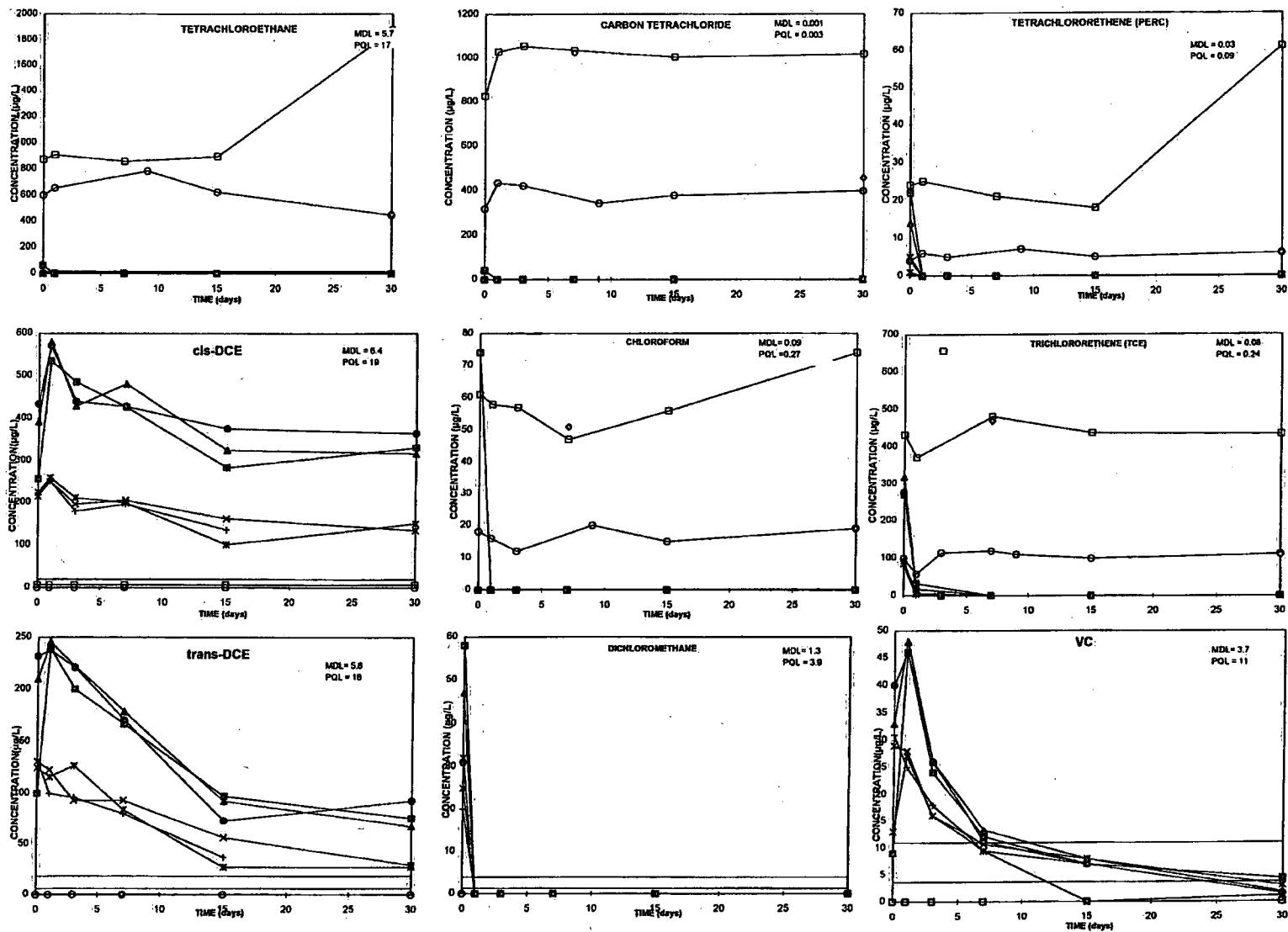
day 1. Trichloroethylene also decreased significantly by day 1 and was below detection limit by day 3. The products of transformation appearing within the first 3 days, *cis* and *trans*-DCE, vinyl chloride (VC), ethane and ethene can account for all the TeCA and PERC and TCE transformed within experimental error (400 from 450 nmoles for the low site water). For the high site water, approximately 1000 nmoles of product were obtained from 1400 nmoles (sum of TeCA, PCE and TCE). No significant amounts of 1,1,2-trichloroethane and chloroethane were found.

The half-lives for TeCA and carbon tetrachloride cannot be calculated because more than 90% was degraded at the first measurement. It is therefore estimated to be less than 5 minutes, the time usually taken for mixing and equilibration after the addition of vitamin B12. The half-lives for PERC was calculated using first order kinetics to be 33 min for the low and 57 min for the high level water. For TCE it was 5.6 hours and 6.2 hours for the low and high water respectively. This implies that in an *in-situ* well application most of the initial reaction would occur in the well, before recirculation into the surrounding aquifer.

After seven days, the gas pressure increased significantly within the microcosms because of the formation of CO₂, a product of glucose biodegradation. It was necessary to vent the microcosms to take accurate headspace measurements. The amount of gas removed was recorded and used in the calculations. The graphs show the results in total number of moles per bottle. The results were calculated using the ideal gas law and the measured volume. Because the amount of gas removed was often as large as the volume of headspace remaining, this caused a depletion of the gases, most of which could not be detected after 15 days. This also had an effect on the remaining chlorinated compounds.

A predicted value could be calculated based on the volume of gas removed and the Ostwald coefficient. Most of the reduction in contaminant concentration could be accounted for by these calculations. Confirmation of this was expected from the sterile control (see Microcosm 4 below).

Summary M1a



M1HA
 M1HB
 M1HC
 M1LD
 M1LE
 M1LF
 M1aHR
 M1aLR
 MDL
 PQL
 DUP

Figure 2

Summary M1a

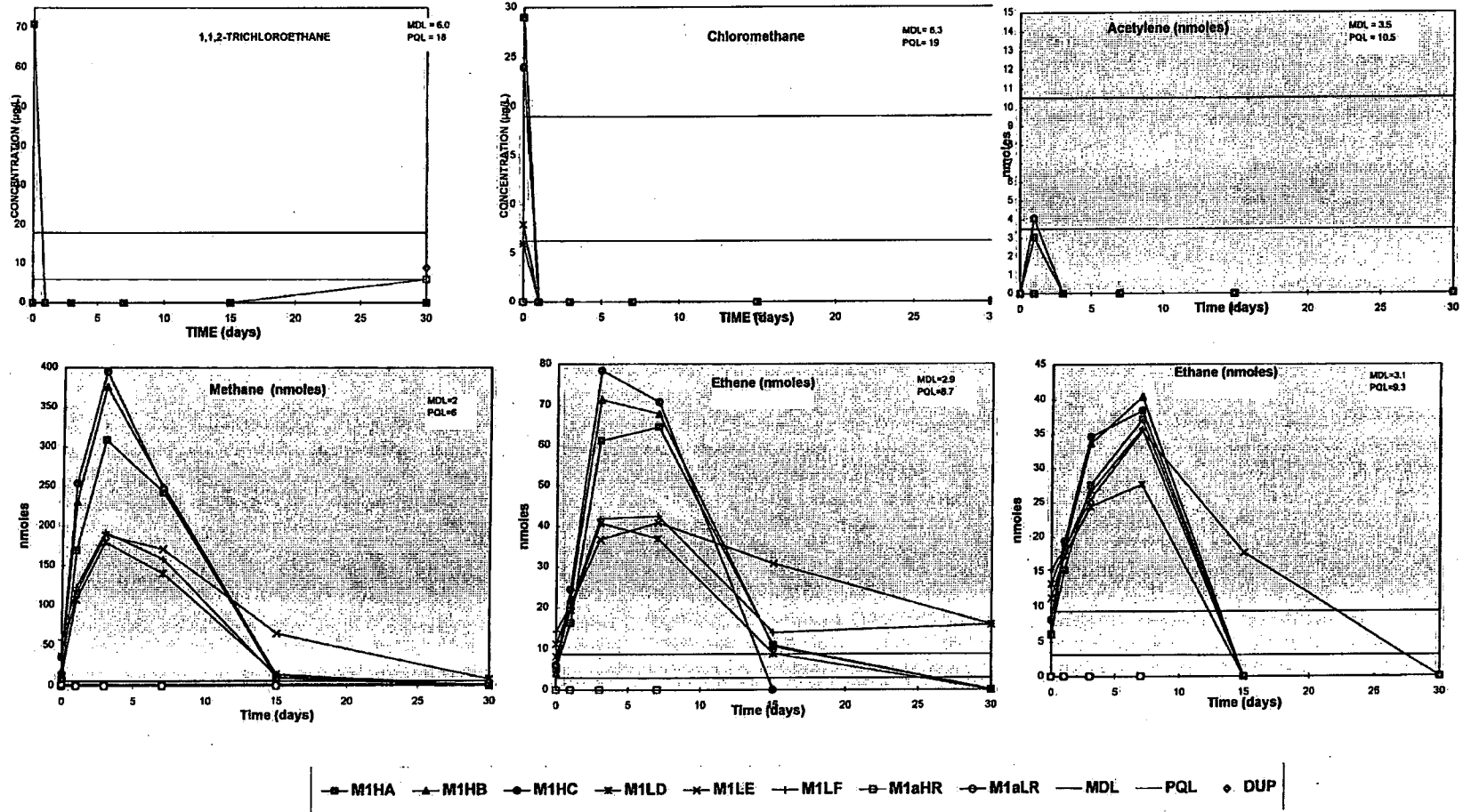


Figure 2

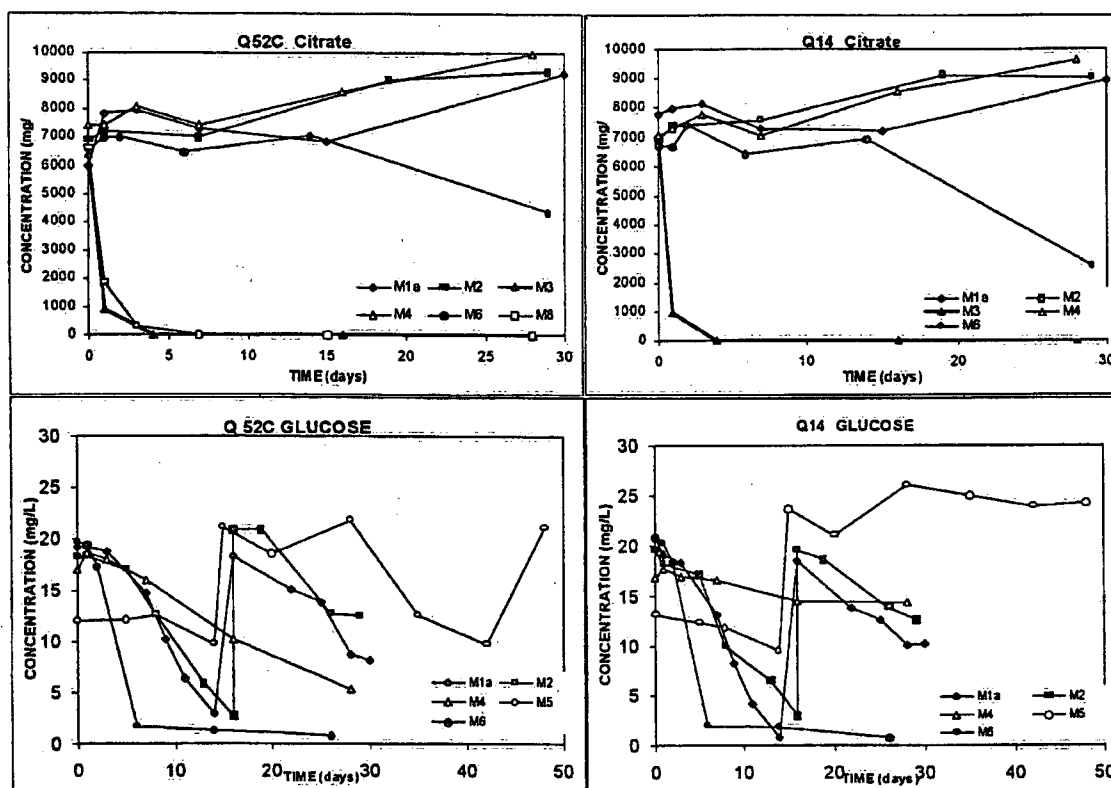


Figure 3. Average glucose and citrate concentrations for the Q52 and Q14 microcosms

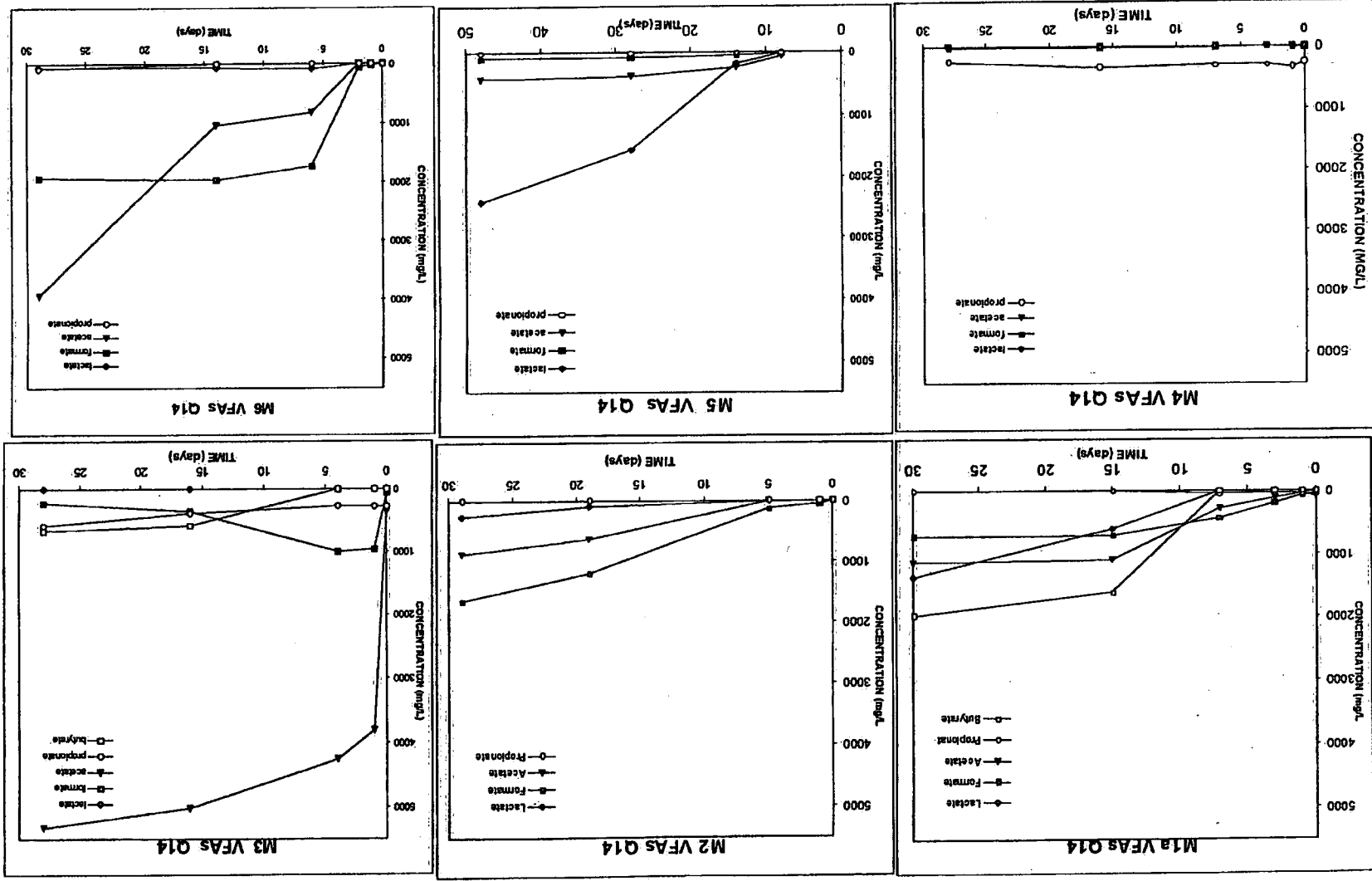
The biological activity in the microcosms was monitored by analyzing for glucose, citrate (Figure 3) and the volatile fatty acids (Figure 4). Formate and acetate were produced immediately, however glucose levels did not decrease significantly until day 7. These products probably arise from the other sugars present in the commercial glucose used. Invertose is actually a high fructose corn syrup that contains glucose, fructose, and other higher saccharides, which are not measurable by the glucose meter. Lactate appeared after the supplementation at day 15, when glucose became seriously depleted (from 20 to <1 mM). Microcosm M1aLF exploded due to a hairline crack in the neck of the vial and pressure build up sometime between days 18 and 21, however some of the

supernatant was saved and transferred to a 60-mL serum vial for continuous monitoring. Lactate, a common glucose metabolite, was measurable by day 19 and, along with formate and acetate, continued to increase over the 30 day monitoring period. Small amounts of propionate and butyrate, generally less than PQL, were measurable by the end of the analyses. The production of acid metabolites was accompanied by a drop in pH from 8.0 to a final pH of 5.5 on day 30. Because the methane concentrations were low and could be accounted for by the abiotic degradation of carbon tetrachloride during the first two days, it was concluded that no methanogenic bacteria were present. A picture of the microcosms (Figure 5) at the beginning and after 28 days of incubation show the color changes that accompany the redox status of the microcosms.

At the end of the experiment, a GC/MS analysis was conducted to confirm the absence of TeCA and the quantity of VC left. The TeCA was below 1 µg/L whereas the concentration of vinyl chloride varied from 1.1 to 4.3 µg/L (full results in Appendix A). The GC/MS analysis also revealed the presence of several metabolites of glucose (heterofermentative pathways): acetaldehyde, ethanol, acetone, butanol and three esters of butanoic acid (ethyl, propyl and butyl). These peaks were probably the cause of the large unknown peaks seen in the FID. There was also some butene, a compound that has been observed as a product of the B12 reduction of PERC and TCE.

In summary then, the vitamin B12 with titanium citrate converted carbon tetrachloride completely to non-chlorinated products. TeCA, PERC and TCE were mostly converted to *cis* and *trans*-DCE, with some VC, ethene and ethane.

Figure 4. Volatile fatty acids (averages, full data sets in Appendix 1)



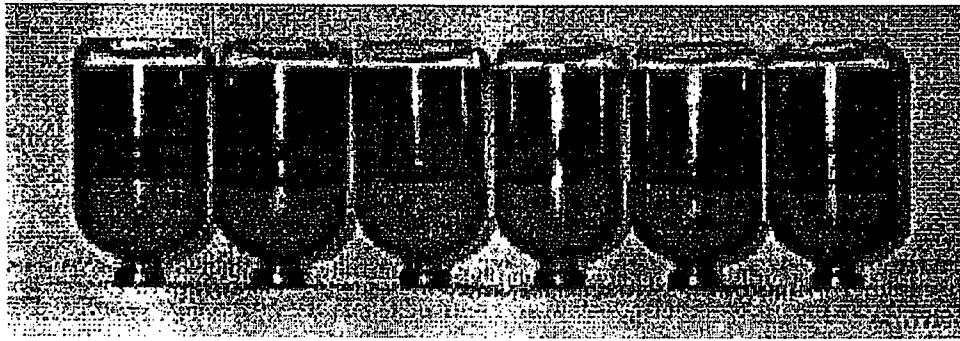


Figure 5. Microcosms 1a. Beginning and after 28 days. The small bottle is the supernatant of F that exploded.

3. *Microcosm 2 (titanium citrate and glucose)*

The purpose of microcosm 2 was to measure the effect of the reducing solution alone, without vitamin B12 being present. Glucose was added to prevent citrate utilization by bacteria.

A summary of the results is shown on Figure 6. Carbon tetrachloride was degraded within one day to chloroform which in turn was dechlorinated to DCM. However, in the absence of B12, DCM persisted for the 28 days of the study. TeCA was also degraded with the concurrent formation of *cis*- and *trans*-DCE, but at a much lower rate than in the

Summary Microcosm 2

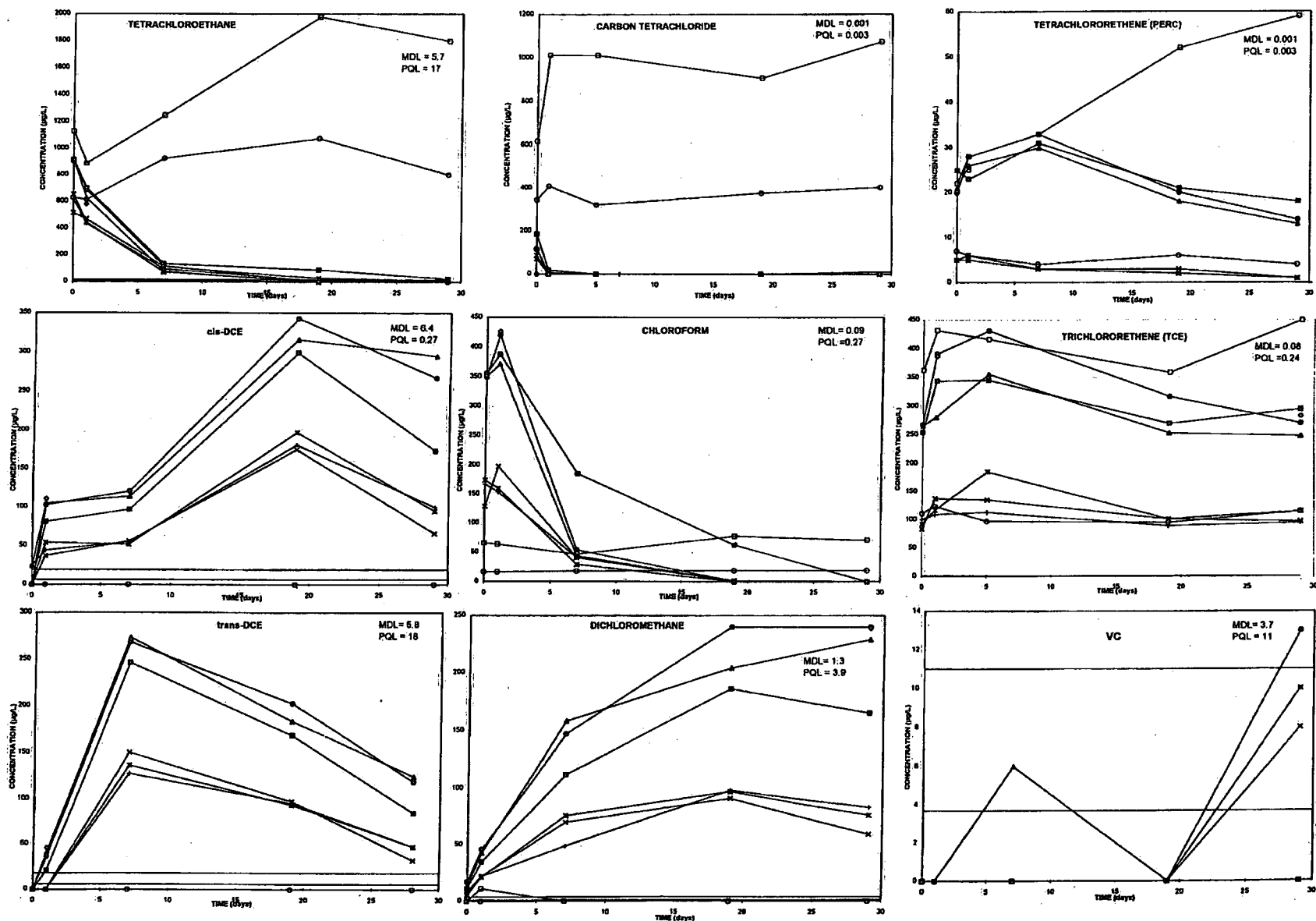


Figure 6

M2HA
 M2HB
 M2HC
 M2LD
 M2LE
 M2LF
 M2HR
 M2LR
 MDL
 PQL
 DUP

Summary Microcosm 2

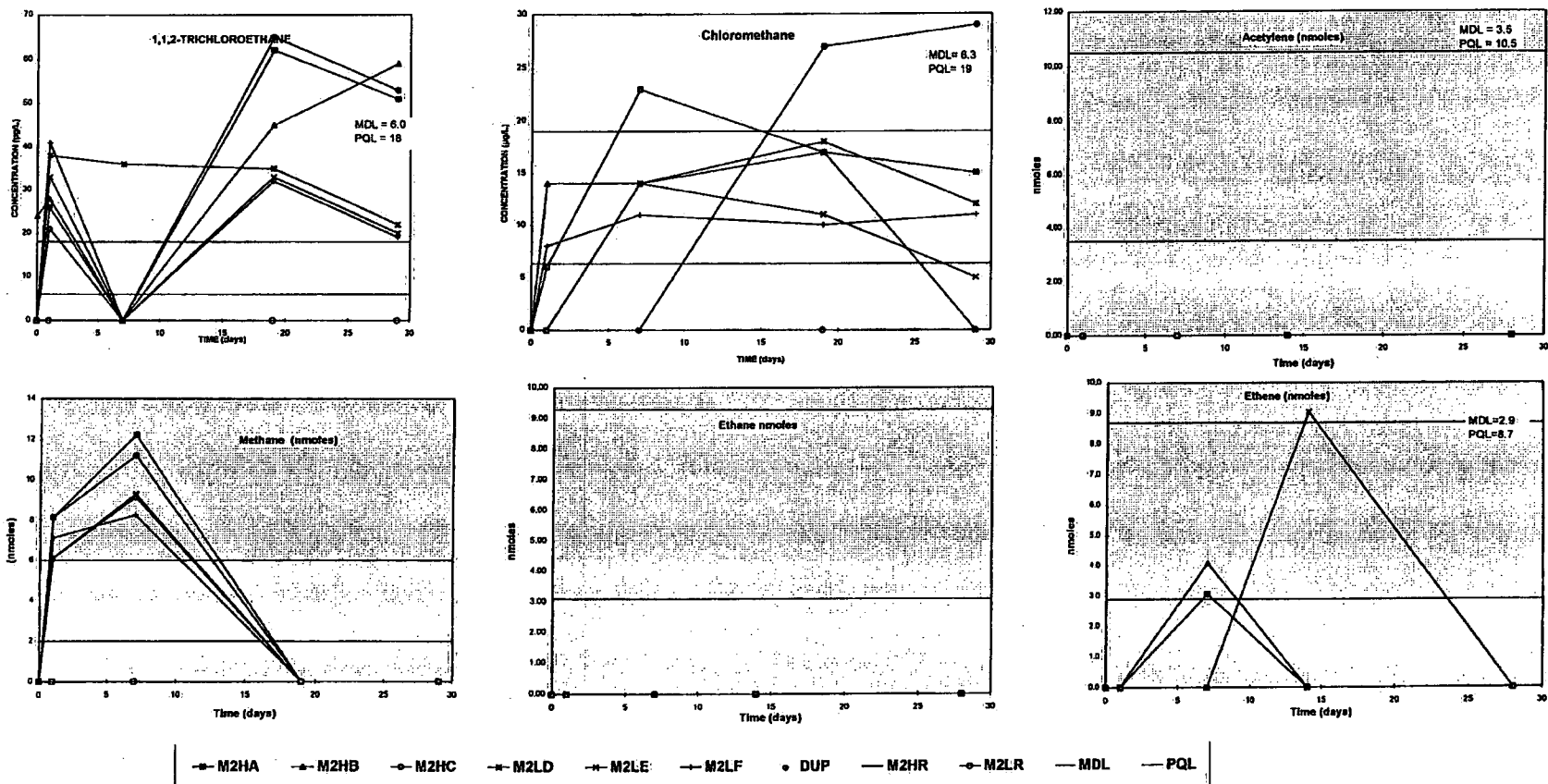


Figure 6

presence of B12 (half-life of 2.3 ± 0.3 days for the high level water and 2.6 ± 0.4 days for the low level). TeCA was transformed to *cis* and *trans*-DCE and 1,1,2-trichloroethane. Many analytical problems were experienced around day 7, which means that the accuracy of the data points on that day are less than for the rest. Nonetheless, the trends remained the same after that period.

There were very little observed losses of PERC and TCE throughout the incubation period and the losses that were, could potentially be attributed to venting. Indeed, similar to what had been observed for M1, glucose was degraded to CO₂ which had to be vented. This effect is very evident for methane, and somewhat less dramatic for the chlorinated compounds. There was very little vinyl chloride, ethene or ethane produced, supporting the fact that there was in fact no degradation of the chlorinated ethenes.

Glucose consumption (Figure 3) was comparable to M1a however there were generally lower amounts of VFAs produced, except for formate which was generated in concentrations double those found in the M1a series (Figure 4). Acetate production, on the other hand, was delayed and not detected until day 19. Lactate concentrations were lower than in M1a, and propionate and butyrate were not detected in the 29 day incubation period. As a result of the lower concentration of acids produced, the pH decreased a final pH of 6.4 as compared to 5.5 in M1a. CO₂ production was much lower in M2 microcosms with an average volume of 55 mL of gas vented from M2 vials as compared to 293 mL vented from M1a microcosms. One significant difference between M1a and M2 microcosms, apart from the vitamin B₁₂ addition, was the amount of titanium added. M1a microcosms contained an average of 20 mM titanium, while M2 and all subsequent microcosms had 30 mM titanium added. Although the reducing solution was prepared in the same manner for all of the microcosms, it is possible that in the particular batch used for microcosm M1a, the oxalate had a shorter reaction time with the titanium metal. To prevent further inconsistencies in future batches, the exact concentration of titanium oxalate was measured prior to preparing the microcosms. Increased concentrations of titanium in M2 microcosms may have had an inhibitory effect on the

indigenous microorganisms hence lower CO₂ production and decreased concentrations of acid metabolites.

In summary then, the presence of vitamin B12 was essential to achieve the degradation of chlorinated ethenes and to the complete degradation of carbon tetrachloride.

4. *Microcosm 3 (titanium citrate and yeast extract)*

The purpose of microcosm 3 was to create favorable conditions for anaerobic methanogenic bacteria which are known to be able to dechlorinate ethenes and ethanes. It was also useful as a comparison to M2, because there was no glucose added, and therefore no large amount of CO₂ present to create a sparging effect. However, because there was no glucose, titanium citrate was rapidly used by the bacteria present. Therefore any reduction reaction had to occur within the first four days. Afterward, any further transformation would be more likely the product of biological activity.

The results are summarized on Figure 7. The rate of degradation of TeCA was similar to M2 (half-lives 2.4 ± 0.6 days for the high concentration and 1.7 ± 0.8 , for the low concentration site water). Similarly all the carbon tetrachloride was transformed to chloroform within 1 day and to dichloromethane within the following sampling. Dichloromethane was not degraded further. Cis and trans-DCE appeared concurrently and this coincided almost exactly with the disappearance of TeCA. The production of vinyl chloride was only evident after 15 days. There was no degradation of TCE, but some PERC was lost in the high level microcosms after 15 days.

As predicted, without glucose amendments, citrate was degraded rapidly to less than PQL by day 1 (Figure 3). CO₂ generation was strong until citrate was completely utilized and then was negligible for the remainder of the monitoring period. The pH was relatively stable, between 7 and 8, over the course of monitoring.

Summary Microcosm 3

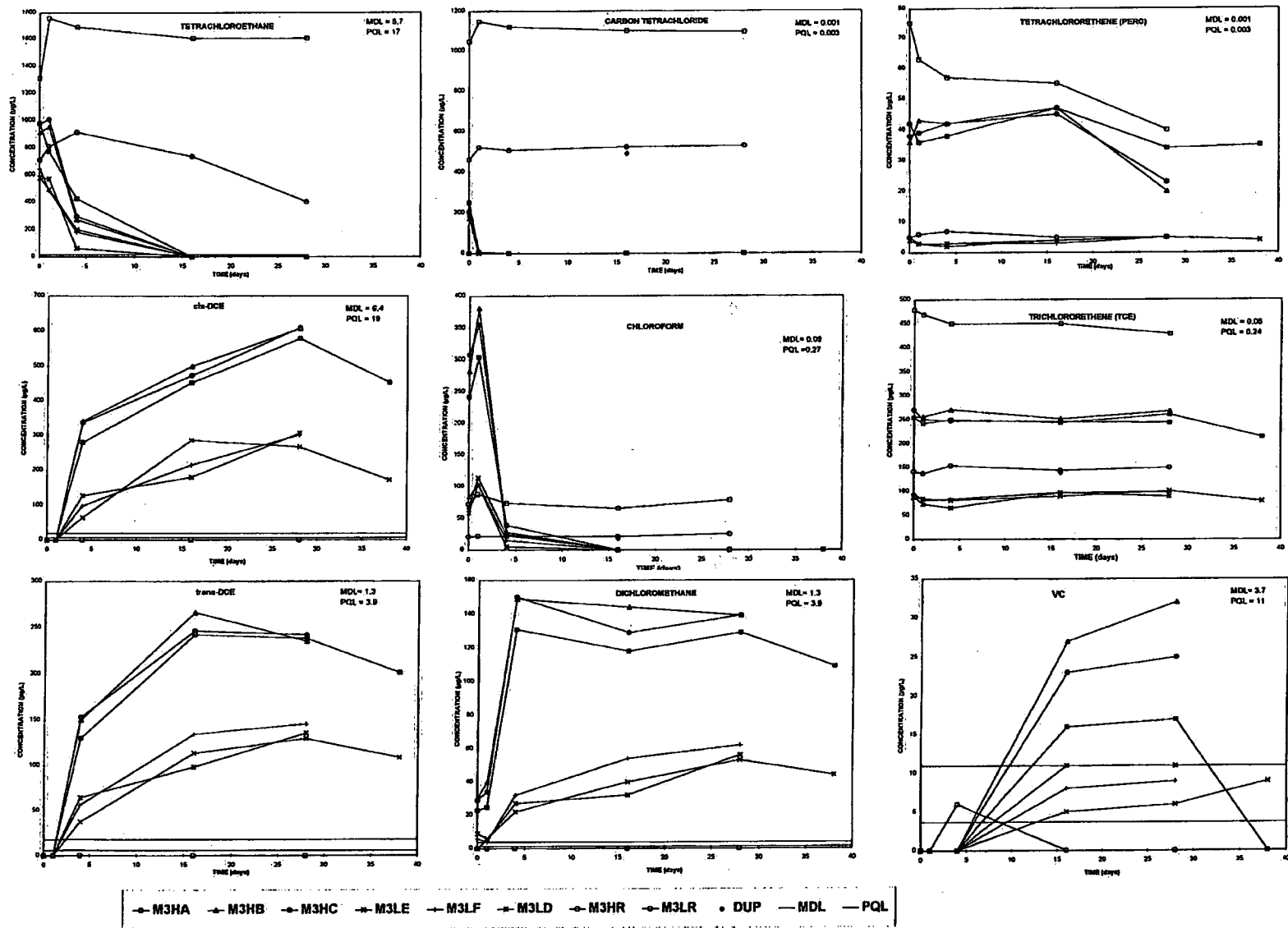


Figure 7

Summary Microcosm 3

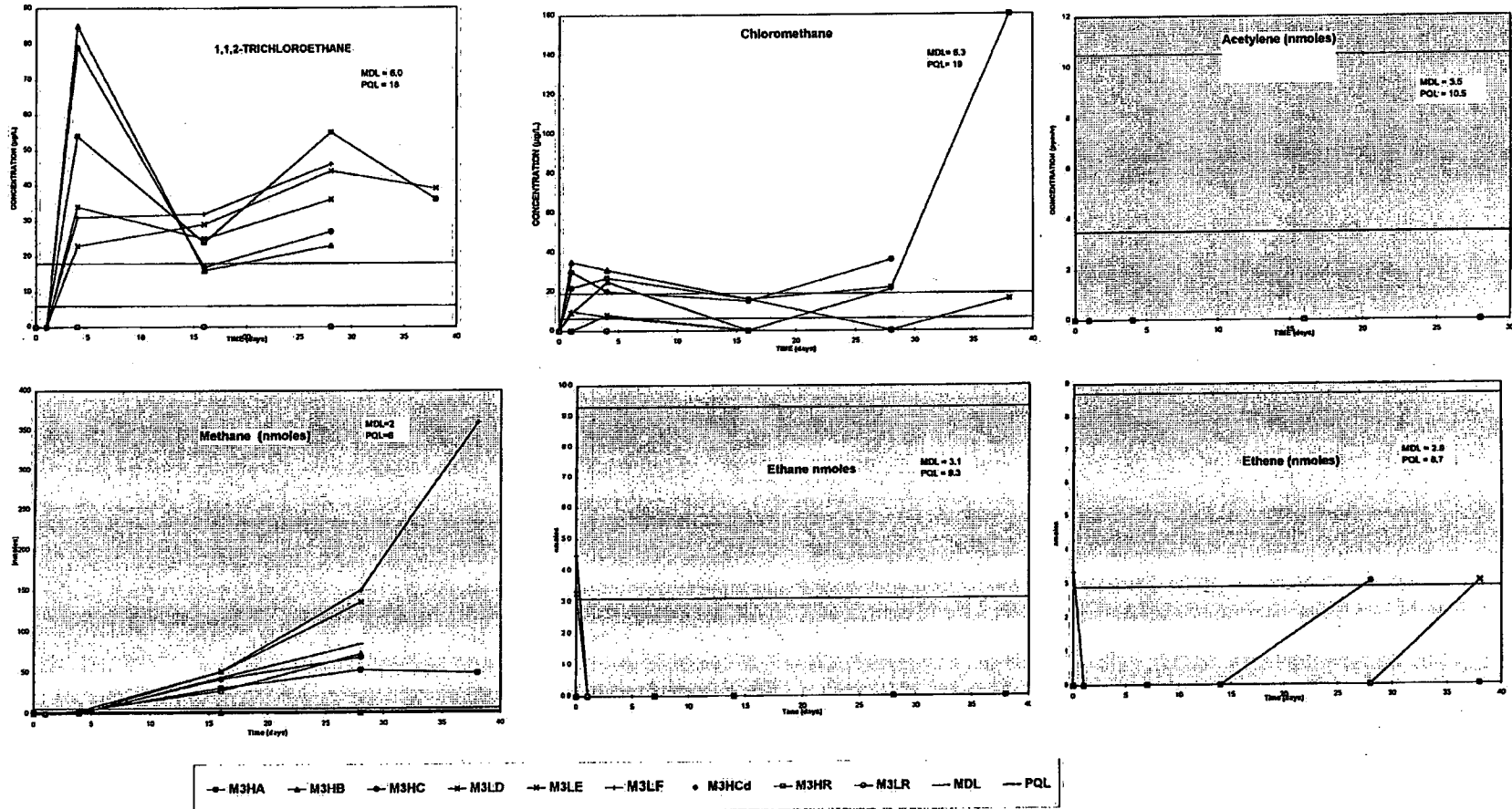


Figure 7

Because the amount of methane was still increasing after 28 days, the microcosms were kept intact and monitored after 38 days. More methane was present than in any of the other treatments. It is therefore apparent that a methanogenic population was being established. Although the concentration of *cis* and *trans*-DCE started to decrease, the rate of dechlorination was not very rapid.

In summary, methanogenic conditions were not obtained as rapidly as hoped and therefore biological degradation did not occur even within an extended period. This led to residual concentrations of dichloromethane, PCE, TCE and vinyl chloride.

5. *Microcosm 4 (γ- irradiated soil -vitamin B12, titanium citrate and glucose)*

The purpose of microcosm 4 was to be able to differentiate between the chemical and biological components of the dechlorination reaction and to provide an environment where no CO₂ would be produced and therefore where losses to the gas phase would be minimal. A preliminary sample of soil, site water and reagents was sent to the irradiation facility at McMaster University (Hamilton, Ontario) to see the effect of irradiation alone. While there were no visible changes in the soil and in any of the dry chemicals, the irradiation completely destroyed all the contaminants in site water. Alternatives such as sterilizing the site water and respiking contaminants, or irradiating the soil but filtering the site water were considered. The latter method was retained.

The soil was rinsed four times with titanium citrate in Milli-Q water before it was sent to the facility. When the sterilized reagents returned, they were used to make up titanium citrate in site water that was filtered through a 0.2 μm filter. All transfers were made in a laminar flow hood. In addition, to prevent contamination during sampling and GC analyses, the syringe needles were rinsed in pure ethanol.

The results (Figure 8) in the first few days were very similar to those in M1a, that is the degradation of TeCA, carbon tetrachloride and PERC all occurred within the first

Summary M4

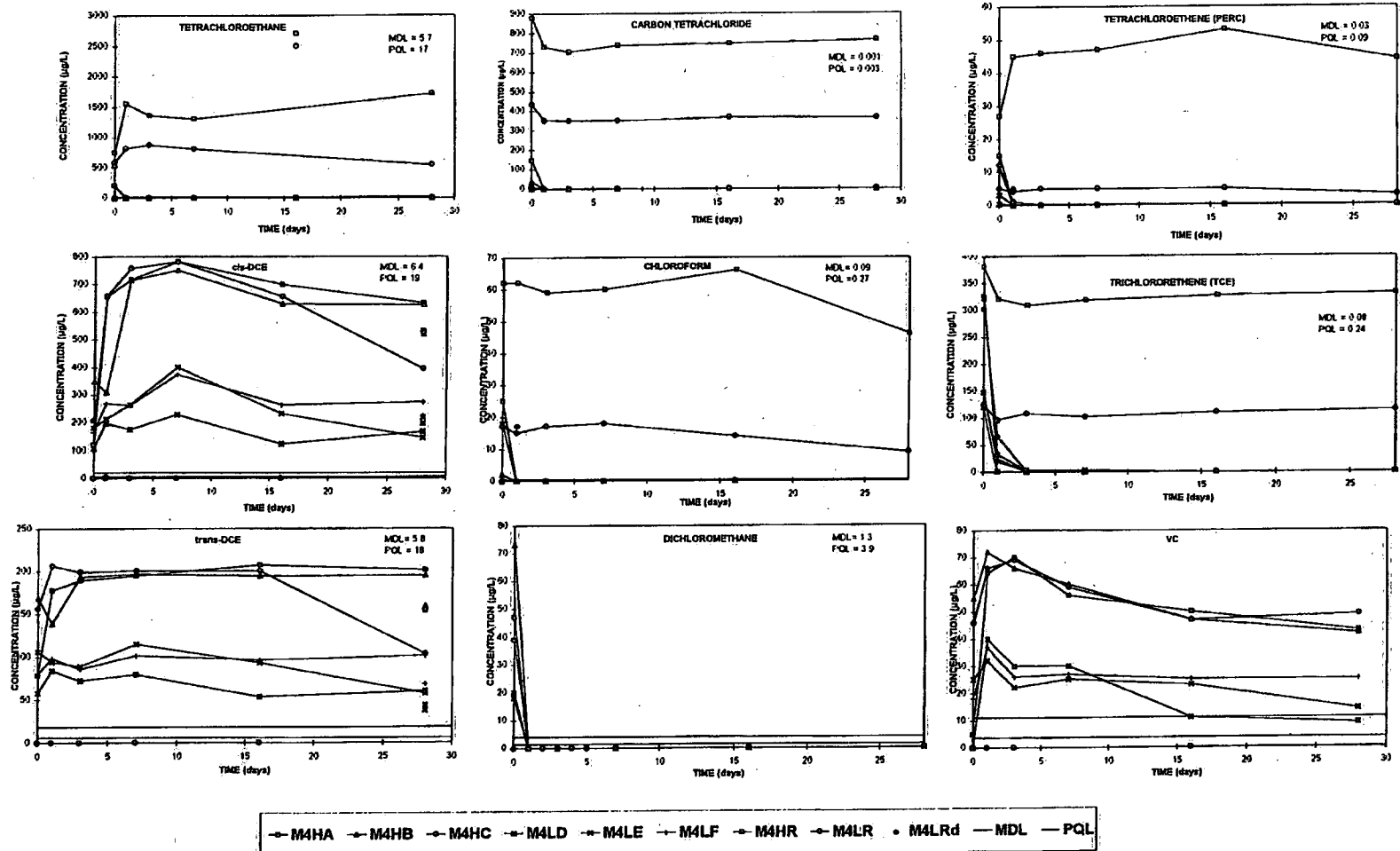


Figure 8

Summary M4

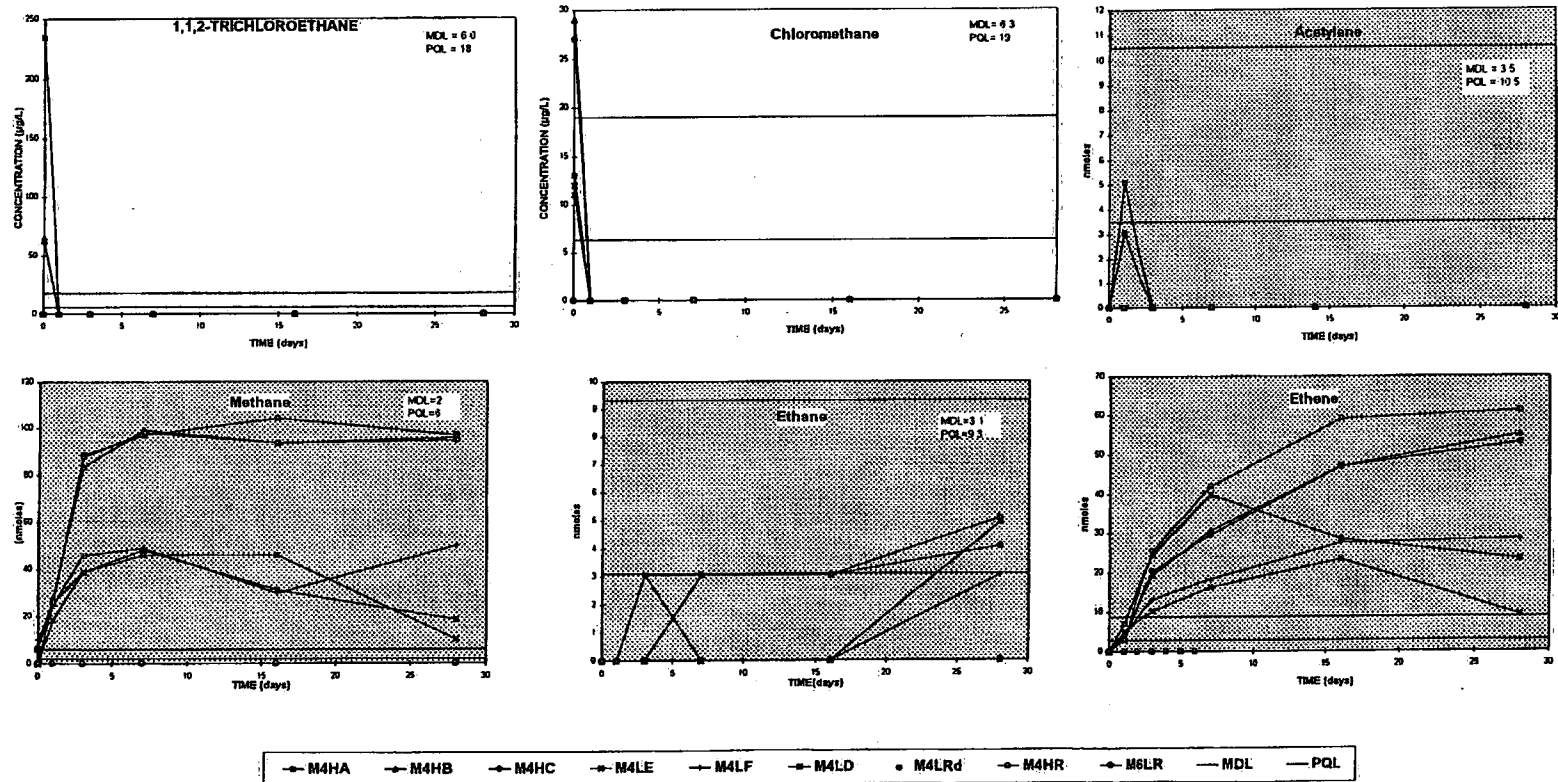


Figure 8

few hours. Measurable half-lives were obtained for carbon tetrachloride high only (21 min), PERC (high, 4 hours; low, 15 min) and TCE (high, 8 hours; low, 11.5 hours). After two days, the only chlorinated products present were *cis* and *trans*-DCE and vinyl chloride. During the whole period, there was no degradation of *trans*-DCE, a little of *cis* and slightly more of vinyl chloride. The apparent low results for *cis*-DCE of the M4HA bottle seems to be an analytical anomaly, because, as can be seen in the tabulated results, the concentrations calculated on the FID were closer to those of the two other high replicates. Similarly, the methane and ethene formed remained stable once formed, except for D and E after 7 and 15 days respectively.

No losses in citrate were observed over a 28 day period, however by day 7 the microcosms had lost their reducing conditions as was apparent by a colorless, as opposed to brown, supernatant. To restore reducing conditions, 10 mL of the supernatant was removed and replaced with a fresh sterile concentrate of Ti(III)-citrate, hence the increase in citrate noted on day 16. Sterility appeared to be maintained in all but two of the microcosms, M4LD and M4LE, in which glucose degradation products, acetate and formate, were detected by days 7 and 28, respectively. Lactate was not detected despite the complete removal of glucose. In addition, CO₂ was measurable solely in M4LD and M4LE.

This confirms that the reductive dechlorination reaction that was observed in the other vitamin B12 containing microcosms was indeed abiotic and that the later losses when glucose was present were attributable to venting to the gas phase. This also means that the vitamin B12 catalyzed reaction is not very effective in degrading *cis* and *trans*-DCE in this situation.

6. *Microcosm 5 (glucose)*

The purpose of this microcosm was to find out if the bacteria occurring naturally at the site could induce reductive dechlorination when given a carbon source.

Summary Microcosm 5

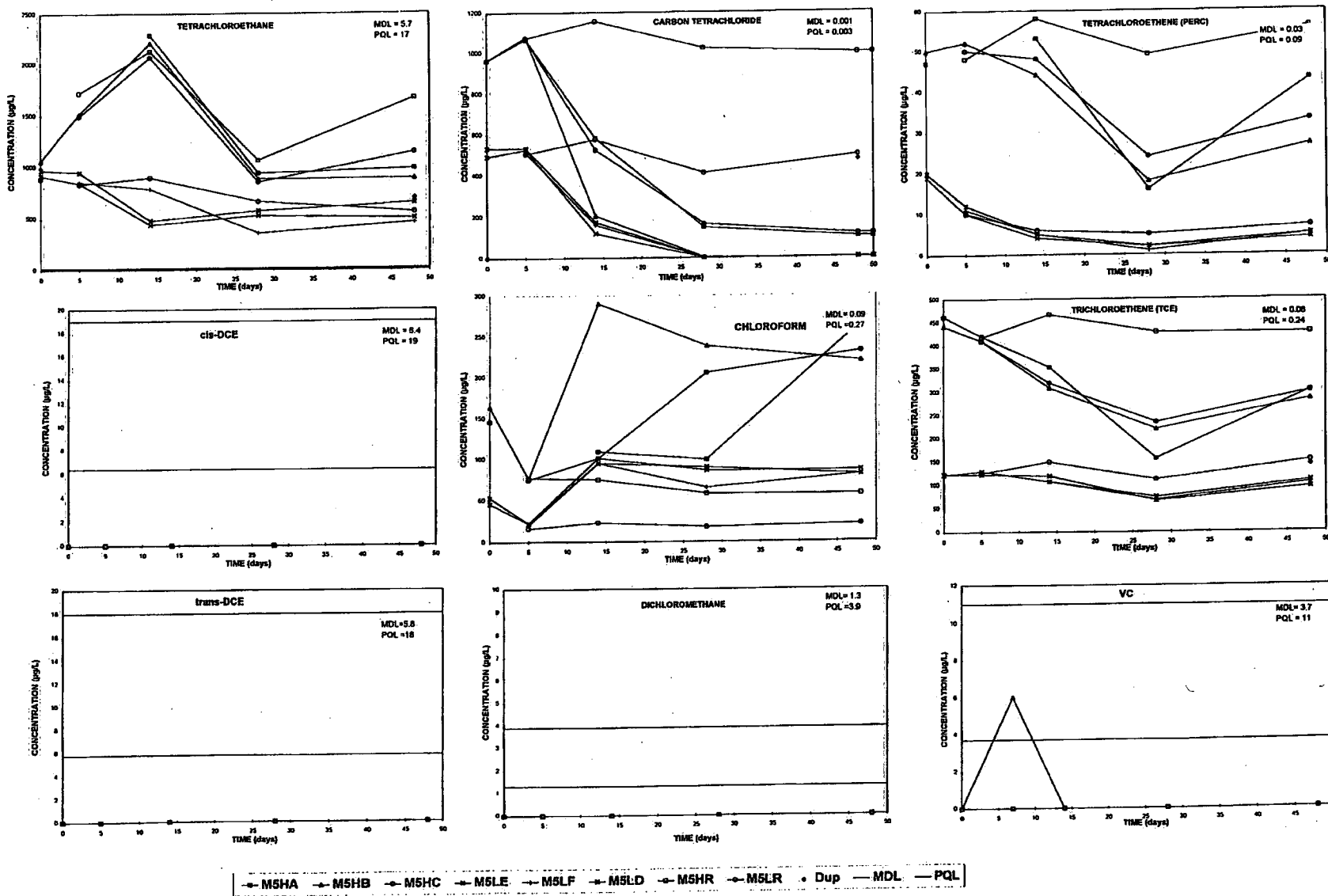


Figure 9

Summary Microcosm 5

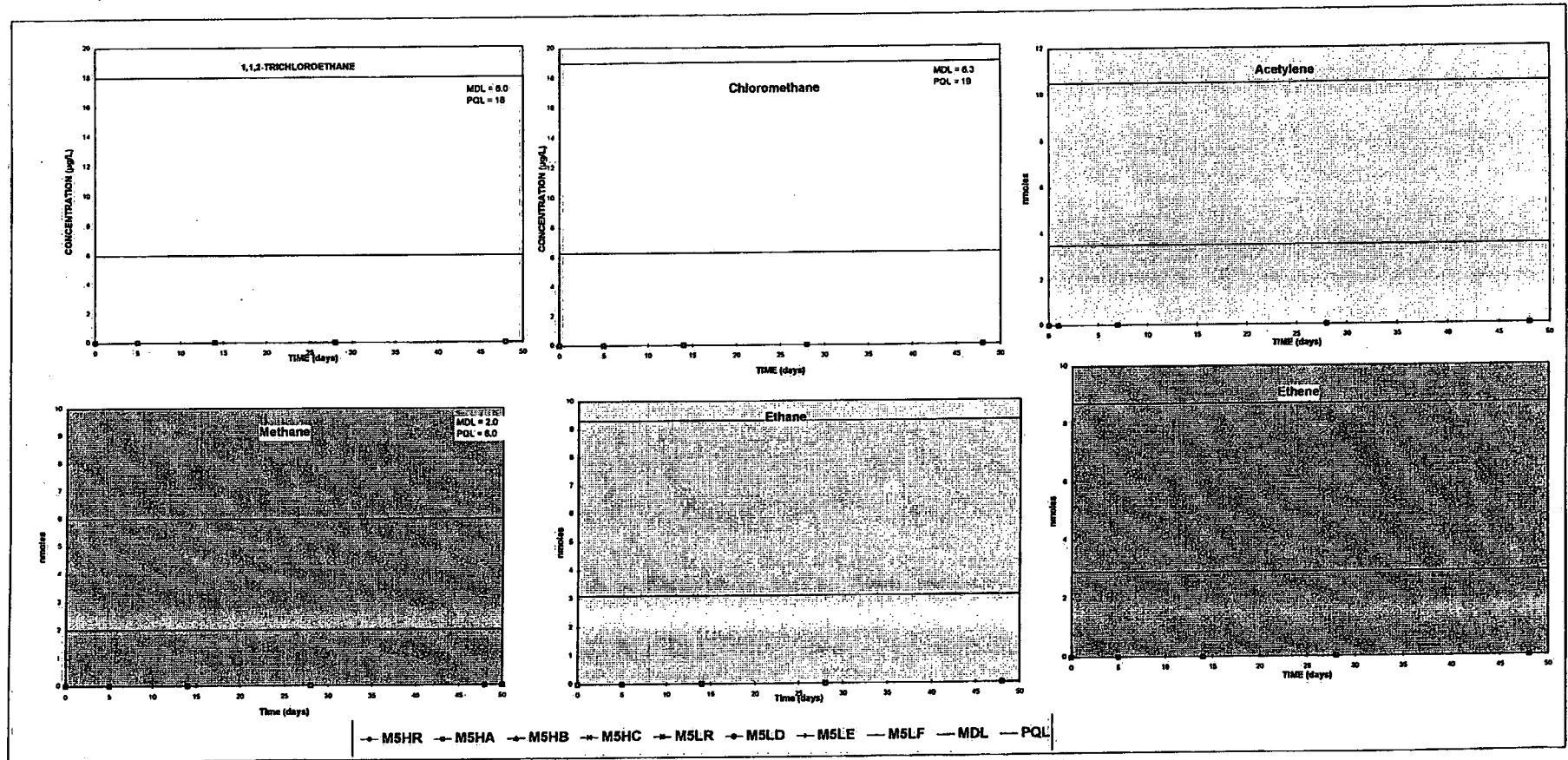


Figure 9

The results are shown on Figure 9. As expected, for the first few days, no reaction occurred. After 14 days a flocculate type growth was visible in the microcosms while glucose concentrations were beginning to decrease, with consequent production of lactate, acetate, formate and CO₂. As these microcosms were meant as a control for chloride, glucose was re-spiked at day 15 in accordance to its addition to M1a and M2 microcosms. It appeared however, that bacterial activity was lost shortly after this time as glucose concentrations remained stable over the next 28 days. This cease in growth was likely a result of inhibitory low pH values as the initial pH of the site water, 5, dropped to 4 by day 14, and between 3-4 by day 28.

After 14 days, the concentration of carbon tetrachloride started to drop with a concurrent increase in chloroform. There was no effect on any of the other chlorinated compounds and the chloroform was not degraded further, even after 48 days. Therefore, it is unlikely that the sole addition of a carbon source will be sufficient to produce conditions where the site contaminants will be biodegraded.

7. *Microcosm 6 (vitamin B12, titanium citrate and glucose, yeast extract added after 2 days)*

The purpose of this treatment was to try to induce methanogenic conditions after most of the chlorinated ethanes had been removed by the vitamin B12 treatment. This was to verify if the lack of intrinsic remediation at the site was due to the toxicity of some of the compounds to bacteria. The toxicity of trichloroethane and of chloroform to methanogens has been well documented.

Therefore for the first two days, the conditions were virtually identical to those in M1a, except that the titanium was higher due to some variability in the strength of the titanium oxalate solution. The results (Figure 10) were virtually identical as well. TeCA, carbon tetrachloride, chloroform and PERC disappeared within hours, and the TCE within one day. The measured half-lives were 15 and 48 min for PERC in the low and high site

Summary M6

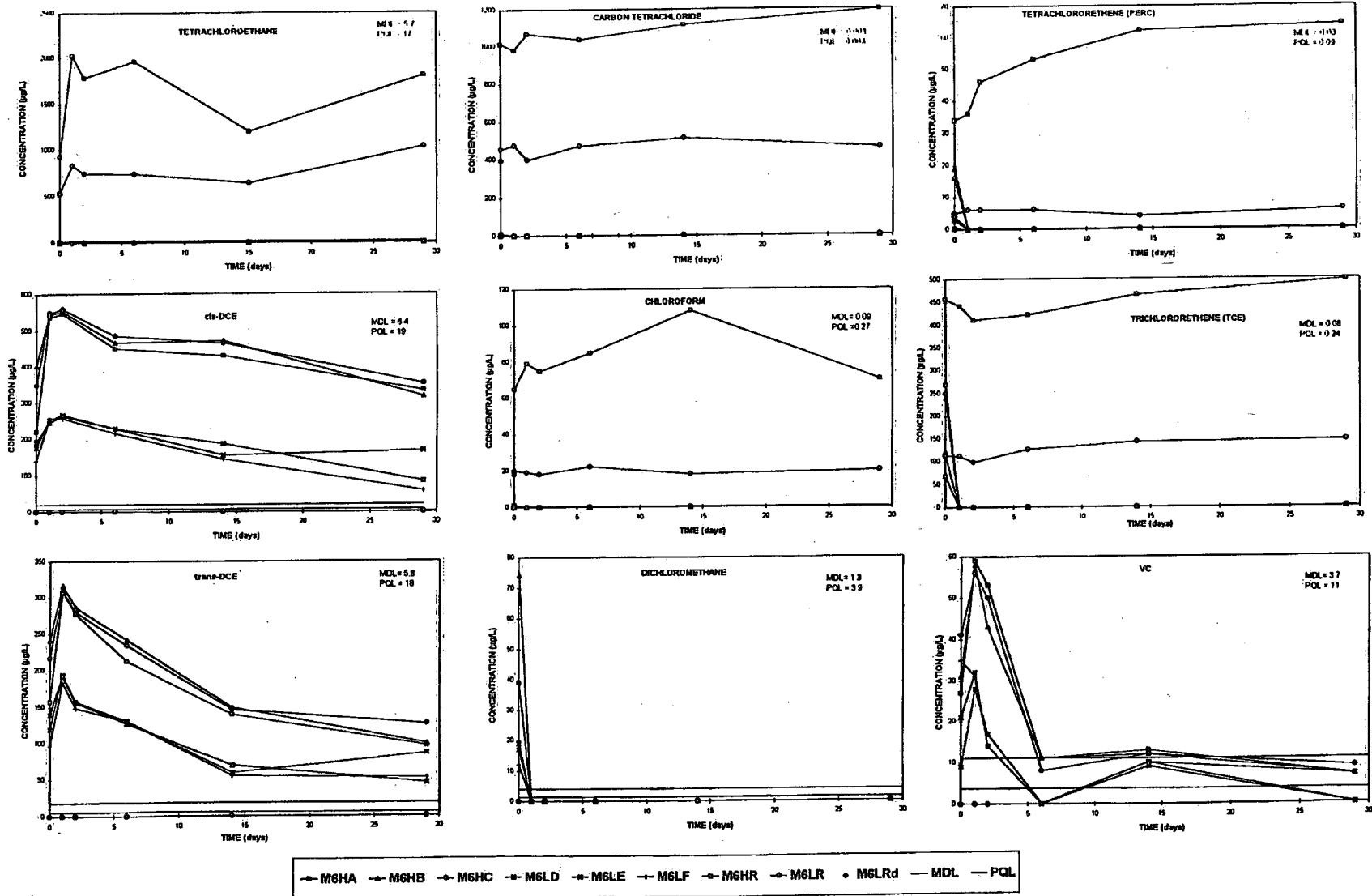


Figure 10

Summary M6

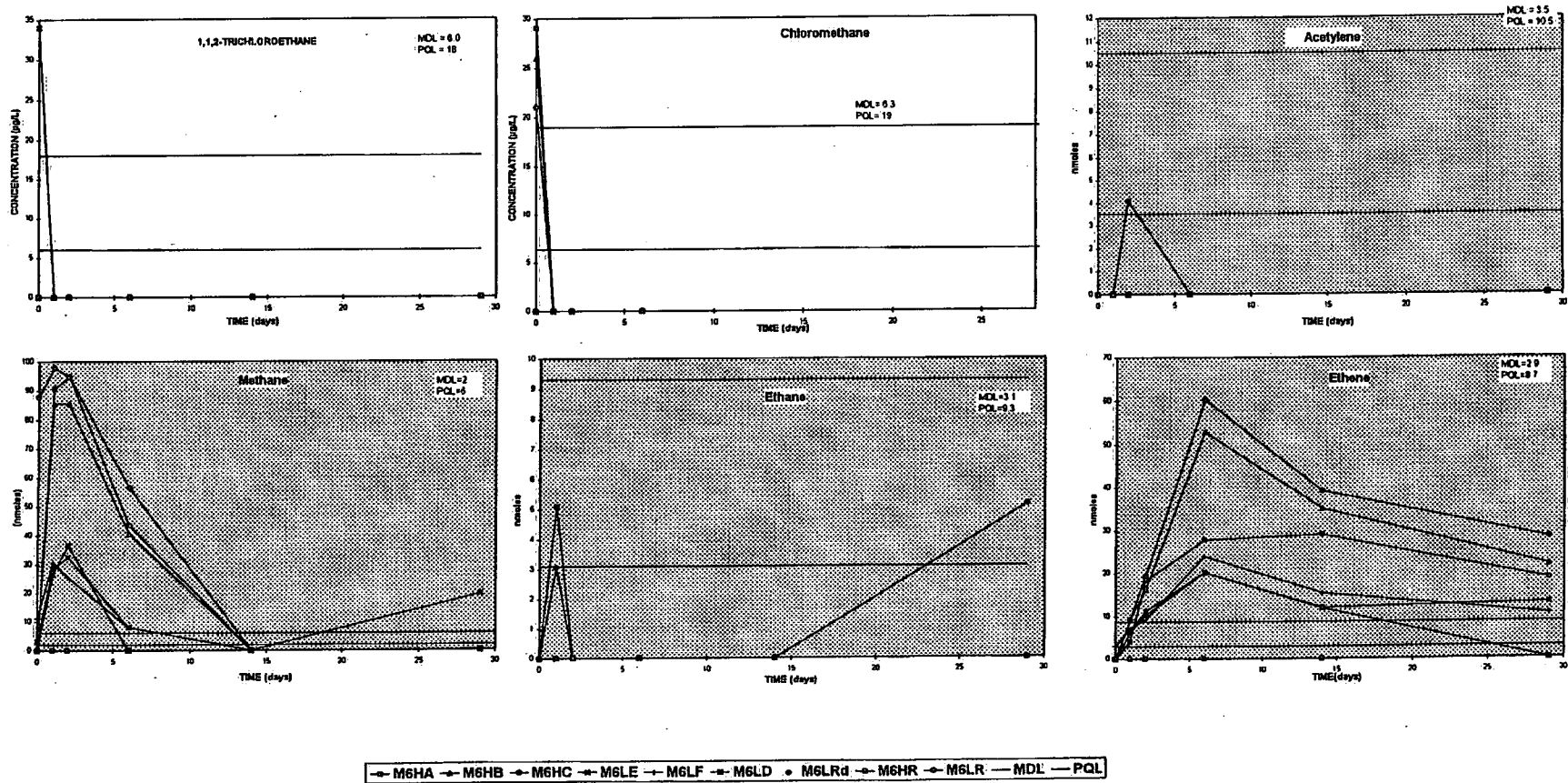


Figure 10

water and 2.4 (low) and 3.5 (high) hours for TCE. Therefore, when yeast extract was added, there were only *cis* and *trans*-DCE and vinyl chloride left to degrade.

The added glucose in M6 was sufficient to prevent citrate utilization for more than 14 days with losses notable in four of six of the microcosms on day 29. Degradation of glucose however, resulted in the generation of much more CO₂ than in the microcosms lacking glucose. Glucose metabolism is generally to lactate with smaller amounts of acetate and ethanol produced, while acetate is the major product of citrate degradation with no lactate being formed. High acetate concentrations coincided with those microcosms in M6 that exhibited significant citrate degradation by day 29. As formate is also generated from glucose, concentrations in M6 were double that of M3 and M8. The pH of the microcosms was relatively stable, between 7 and 8, over the course of monitoring.

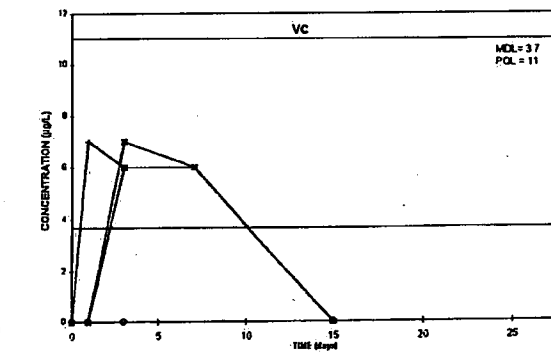
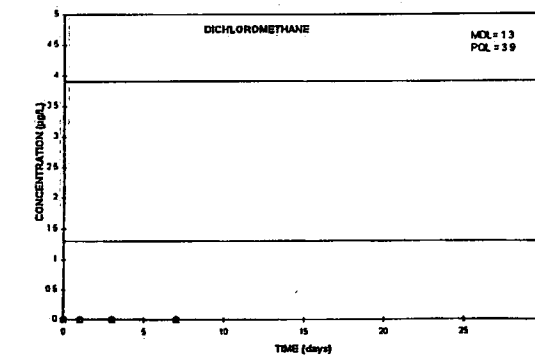
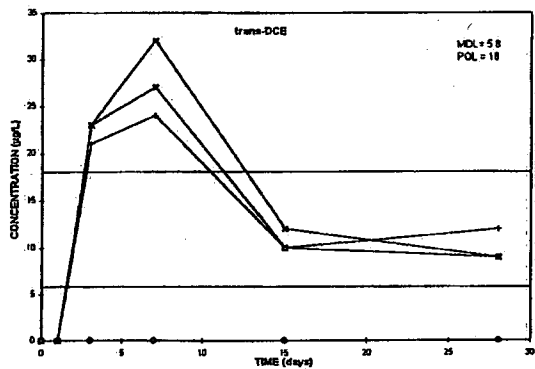
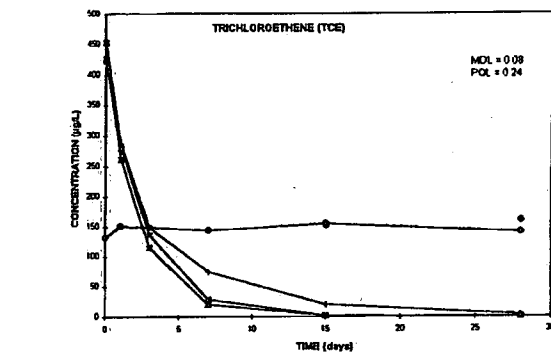
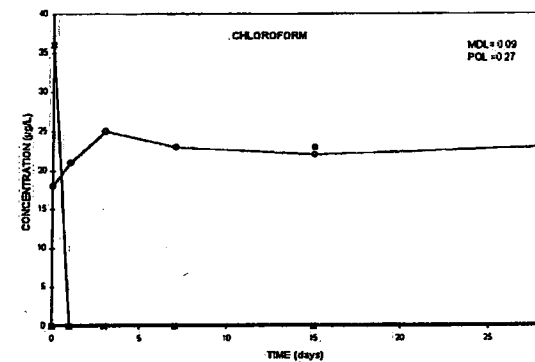
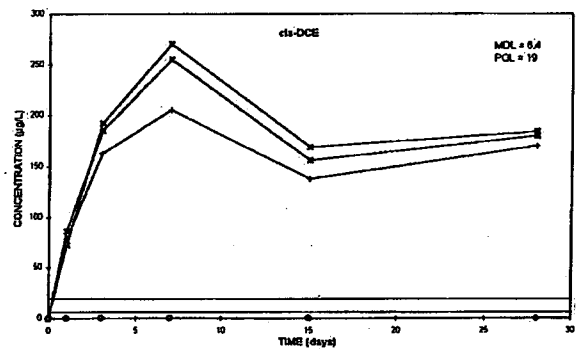
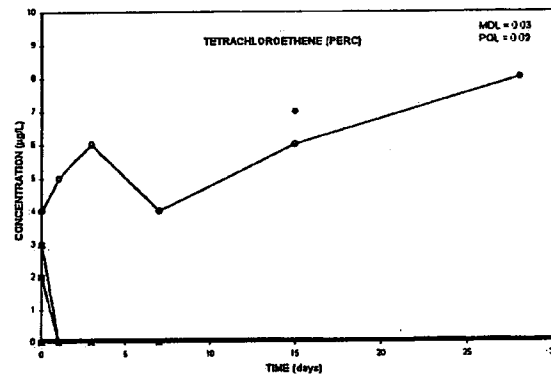
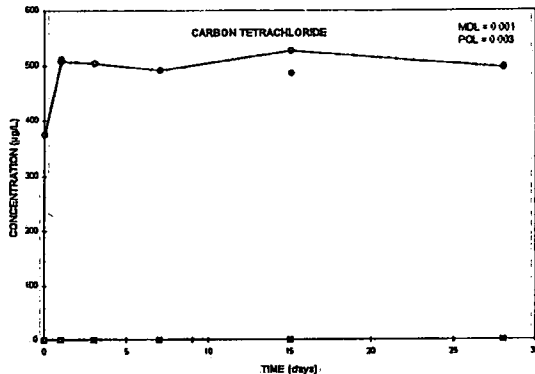
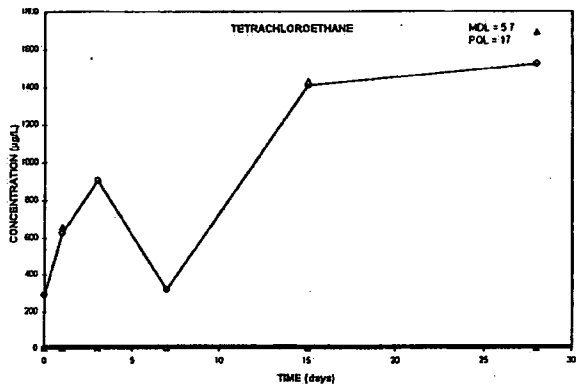
Methanogenic conditions were not established within the 29 days of the study in this system. Since M6LE did exhibit a slight increase in methane on the last day, it was monitored again on day 50. The concentration of methane had doubled, without any significant effect on the concentration of the chlorinated compounds.

8. *Microcosm 7 (vitamin B12, new titanium chelate)*

The purpose of this set was to evaluate the reactivity and stability of a new product that was developed by a chemical company. The premise was that this would be just as reactive as titanium citrate, but hopefully not degraded as rapidly, hence it could dispense us of adding the glucose. Because there was a lot of uncertainty associated with this treatment it was only tried with Q52 water.

The method that was used to make up this solution was different than with citrate and resulted in the site water being brought to pH 12 for a few minutes. As can be seen on Figure 11, this was sufficient to transform all the TeCA to TCE. The half-life of TCE was 2.3 ± 0.8 days. The disappearance of carbon tetrachloride was too rapid to be measured (half-life < 5 min.) and that of PERC was 70 min. Very little vinyl chloride was formed and

Summary M7



→ M7LD → M7LE → M7LF • M7LRd — MDL — POL → M7LR

Figure 11

Summary M7

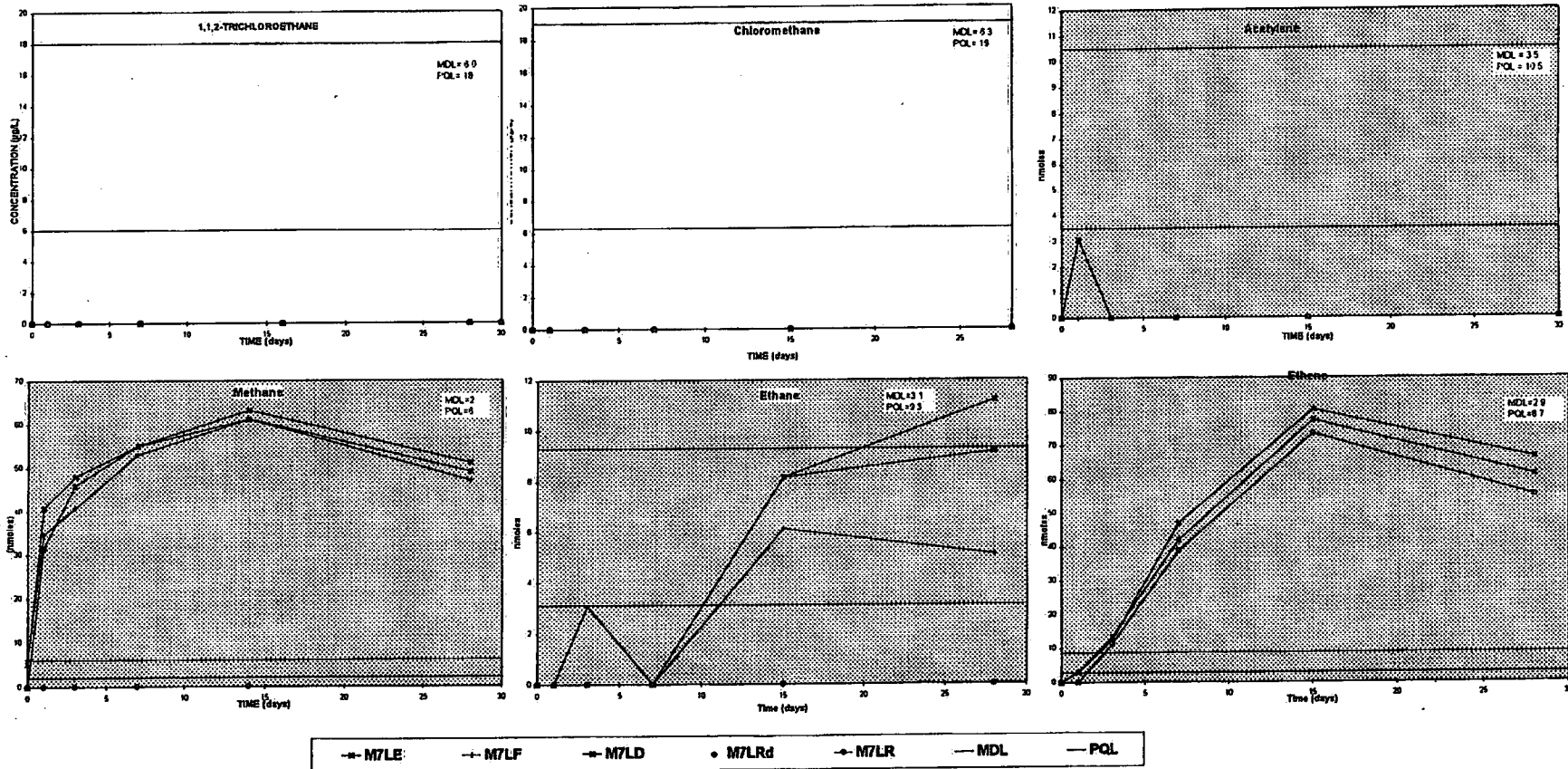


Figure 11

it was a transient species. The rate of formation of ethene was almost identical to the rate of degradation of TCE ($k = 0.2$ compared to -0.3 days^{-1}).

This chelate was chosen because it was anticipated to be less biodegradable than citrate, such that glucose amendments to the remedial solution would be unnecessary. As this compound was not measurable using the ion chromatography methods, its degradation could only be assessed by the appearance of degradation products that were measurable. Reducing conditions were maintained throughout the 28 day analysis period with the supernatant remaining dark and clear with visually no evidence of bacterial growth, such as clouding of the supernatant due to cell proliferation. On day 28 very small amounts of formate were measured, yet no other VFAs were detected. No gas generated in any of these microcosms.

Based on information for compounds with a similar chemical structure, this product is expected to have low toxicity and would be an excellent alternative to citrate. Because it is a metal chelating agent, however, there may be restrictions in its use in some areas because it could mobilize toxic metals. However, it is not yet commercially available. Therefore it was decided to continue to use citrate and glucose for the column study.

9. *Microcosm 8 (Titanium citrate, yeast extract, vitamin B12)*

This microcosm was added at the end, when it was noted that microcosm 3 had generated some methane but that the dechlorination was relatively slow. Since it is known that the anaerobic bacteria responsible for dechlorination require vitamin B12 as a growth factor, it was decided to try this combination. In addition, the presence of vitamin B12 would cause a rapid abiotic dechlorination of chloroform, carbon tetrachloride and tetrachloroethane that may have inhibitory effects on this microbial population. Only low site water was used for this set.

The results (Figure 12) were very similar to those of M4, the sterile control, in that there was no loss of cis and trans-DCE to the gas phase. However, since glucose had not been added, not all the TCE was degraded because citrate was lost after one day. The half-

Summary M8

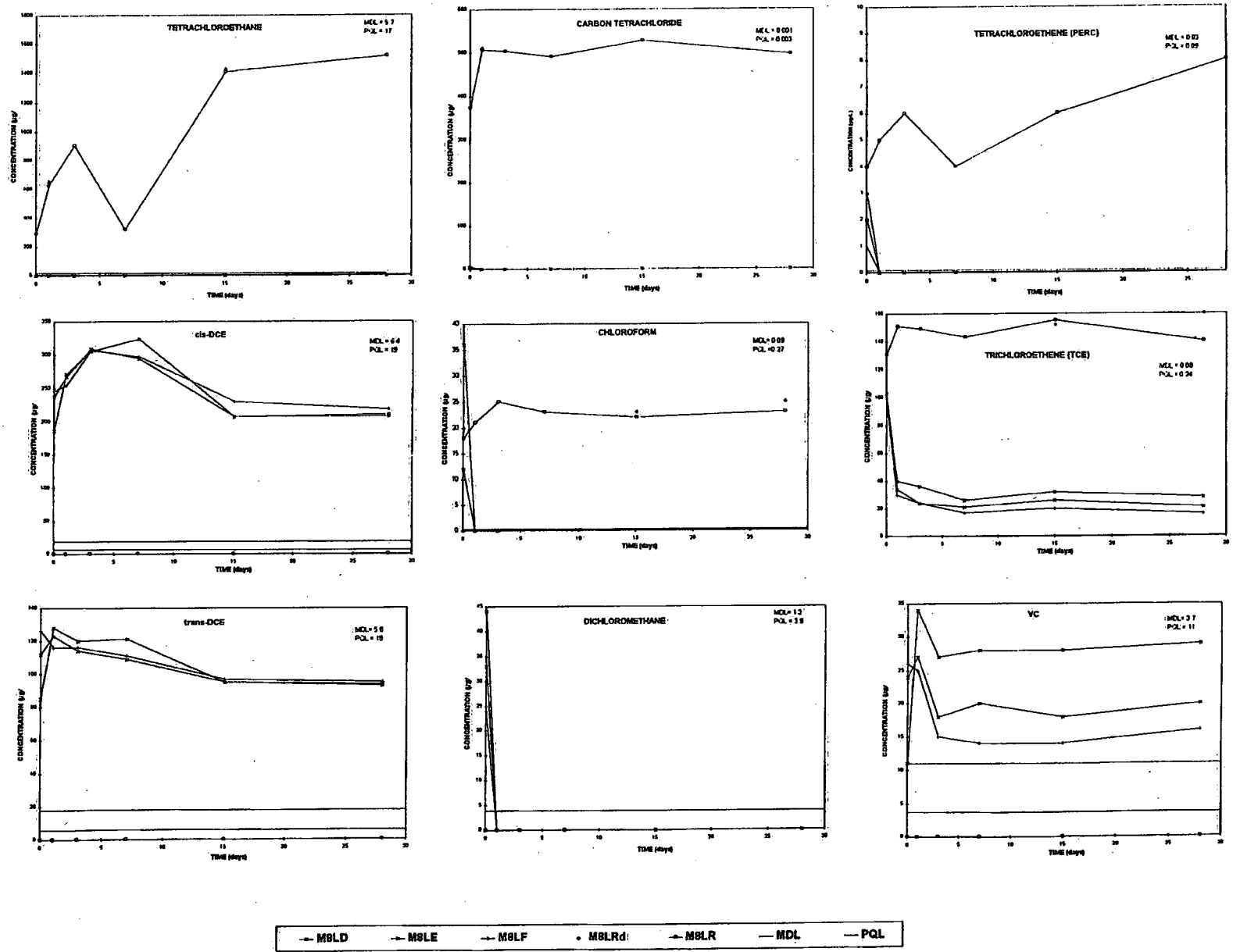


Figure 12

Summary M8

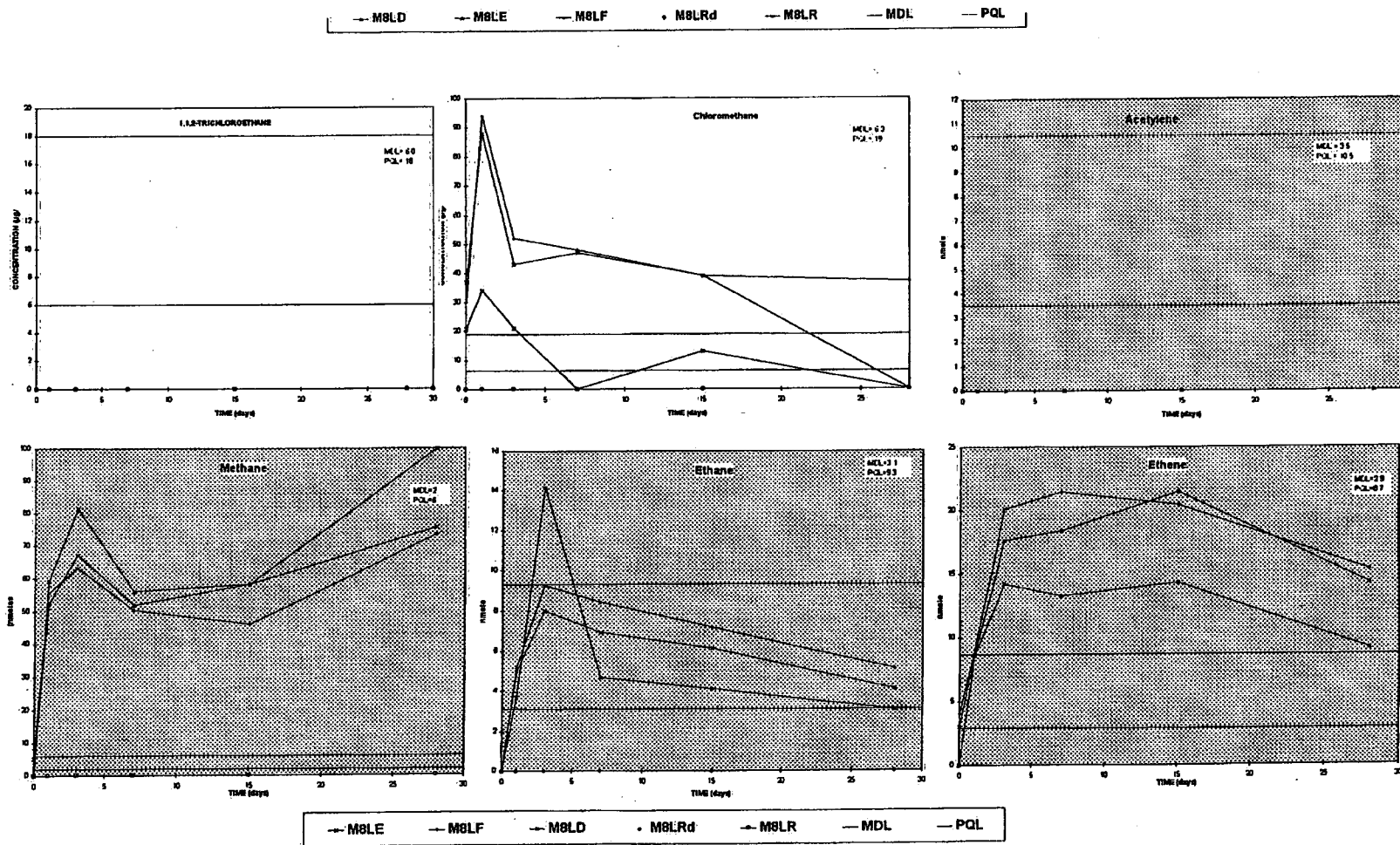


Figure 12

life for PERC was 65 min, whereas for TCE it was 1.4 ± 0.25 days, if the first 3 days of data only are considered. The results were also comparable to the first few days of M6, save the TCE. When compared to M3 (yeast extract and titanium citrate), the difference was in the B12-catalyzed degradation of PCE, TCE and TeCA. The amount of methane formed after 28 days was not higher than in M3 and could still all be attributed to the degradation of carbon tetrachloride (maximum potential of 320 nmoles/bottle) and not to methanogenesis.

As predicted, without glucose amendments, citrate was degraded rapidly to less than PQL by day 3 (Figure 3). CO₂ generation was strong until citrate was completely utilized and then was negligible for the remainder of the monitoring period. Acetate concentrations were in excess of 4 g/L upon complete degradation of citrate. Formate was generated also from citrate but it appeared that the bacteria were utilizing it as concentrations were decreasing over the last 2-3 weeks of analyses. The pH remained between 7 and 8, over the whole incubation period.

In summary, the presence of vitamin B12 did not significantly impact the methanogenesis or the rate of dechlorination in these microcosms. The absence of glucose resulted in the rapid disappearance of citrate, stopping the degradation of TCE.

10. *Post-microcosm reactions*

After the end of the incubation period, the supernatant was transferred to 40 mL volatile vials and 10 mLs were sampled with an autosampler for purge-and-trap GC/MS analysis to confirm the presence/absence of target compounds for which the MDL was below the MAC using the SRI GC, and to identify other compounds formed.

After a 11 days, the headspace was sampled again and analysed for *cis* and *trans*-DCE using the Photovac. The results are therefore semi-quantitative. In many of the bottles of M1a, the ratio of *trans/cis* DCE had decreased significantly and some new early eluting peaks, having the same retention time as acetone and ethanol were formed. After

three more days, *trans*-DCE was at the detection limit in one of the bottles. A month later, *cis* and *trans*-DCE had disappeared completely. This did not happen at the same rate in all the microcosms. The same phenomenon was observed in M4. After 45 days, *cis* and *trans*-DCE had decreased in M4LF and M4HB. Two weeks later their concentration was low in all but one (M4HC). The glucose was gone in all of them, but not the citrate. Acetate and formate was high in all, and propionate disappeared. The Eh was negative in all of them. There was no correlation between the concentration of the fatty acids and the disappearance of *cis* and *trans*-DCE. It is therefore possible that bioremediation will occur in the aquifer after the vitamin B12 treatment. The column experiments will provide a better indication of this possibility.

IV. Conclusions

This series of experiments showed that the vitamin-B12/titanium citrate reaction was effective in treating all the compounds present in the Graces Quarter site. The rate of degradation of the chlorinated ethanes and of tetrachloroethene was very rapid. The final products were *cis* and *trans*-DCE which remained above their maximum acceptable concentration after 28 days. There was no difference in reaction rates between high and low site water. The addition of titanium citrate alone, without vitamin B12, did degrade some of the contaminants, but at a much reduced rate. The γ -irradiated control showed that most of the dechlorination observed was abiotic. The degradation of glucose under these anaerobic conditions produced large amounts of CO₂, which contributed to the removal of some of the chlorinated products.

V. References

Lesage, S., Brown, S. and K. Millar. 1996. "Vitamin B₁₂-Catalyzed Dechlorination of Perchloroethylene present as Residual DNAPL" *Groundwater Monitoring and Remediation*, Fall issue, 76-85.

Lesage, S., Brown, S. and K. Millar 1997. "Method for Dehalogenating Contaminated Water and Soil" U.S. Patent 5,645,374. Date of issue: July 8, 1997.

Millar, K. and S. Lesage. 1997. "Bio-compatibility of the Vitamin B₁₂-catalyzed Reductive Dechlorination of Tetrachloroethylene". In: *In Situ and On-Site Bioremediation*, B. Alleman and A. Leeson, chairs. *Proceedings of the Fourth International Symposium on In Situ and On Site Bioremediation*, New Orleans, Louisiana, April 28-May 1, 1997. Vol 4(4), 471-477.

VI. Appendix A

Microcosm 1a

Vitamin B12, titanium citrate, glucose

Microcosm 2

Titanium citrate, glucose

Microcosm 3

Titanium citrate, yeast extract

Microcosm 4

Irradiated, vitamin B12, titanium citrate,
glucose

Microcosm 5

Glucose

Microcosm 6

Vitamin B12, titanium citrate, glucose,
yeast at day 3

Microcosm 7

Alternate chelator and titanium, vitamin
B12

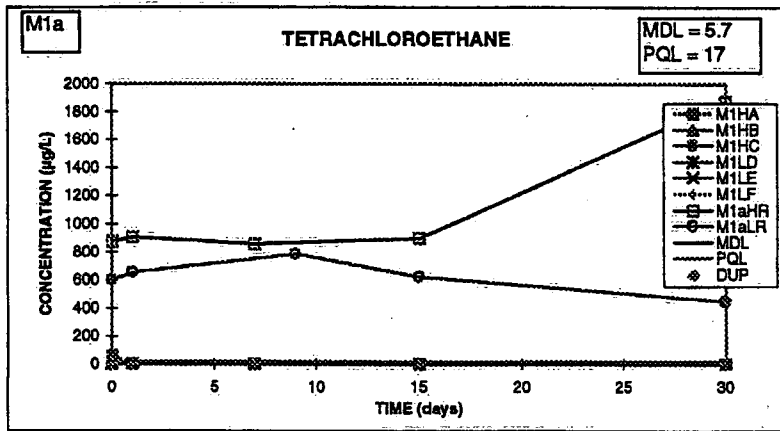
Microcosm 8

Vitamin B12, titanium citrate, yeast extract

Tetrachloroethane (TeCA) Microcosm 1a

Result concentration in µg/L

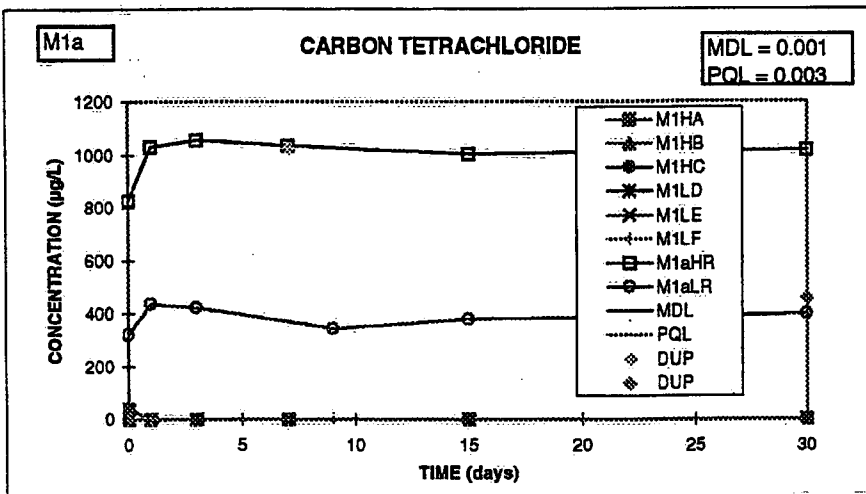
| Time | M1aHR | M1HA | M1HB | M1HC | M1aLR | M1LD | M1LE | M1LF | M1aLRd | r^2 (if < 0.99) |
|------|-------|------|------|------|-------|------|------|------|--------|--|
| 0 | 875 | 61 | | | 601 | n.d. | | | | 0.92 (when including initial calibration data) |
| 0.04 | | | n.d. | | | | n.d. | | | |
| 0.08 | | | | n.d. | | | n.d. | n.d. | | |
| 1 | 908 | n.d. | n.d. | n.d. | 656 | n.d. | n.d. | n.d. | | |
| 7 | 858 | n.d. | n.d. | n.d. | | n.d. | n.d. | n.d. | | |
| 9 | | | | | 786 | | | | | |
| 15 | 895 | n.d. | n.d. | n.d. | 524 | n.d. | n.d. | n.d. | | 0.951 |
| 30 | 1875 | n.d. | n.d. | n.d. | 449 | n.d. | n.d. | n.d. | 456 | |



Carbon tetrachloride Microcosm 1a

Result concentration in µg/L

| SITE WATER | | 904 | | | | | 364 | | | |
|------------|-------|------|------|------|-------|------|------|------|-------|--------|
| Time | M1aHR | M1HA | M1HB | M1HC | M1aLR | M1LD | M1LE | M1LF | M1HRd | M1aLRd |
| 0 | 825 | 41 | | | 319 | n.d. | | | | |
| 0.04 | | | n.d. | | | | n.d. | | | |
| 0.08 | | | | n.d. | | | | n.d. | | |
| 1 | 1029 | n.d. | n.d. | n.d. | 435 | n.d. | n.d. | n.d. | | |
| 3 | 1055 | n.d. | n.d. | n.d. | 421 | n.d. | n.d. | n.d. | | |
| 7 | 1035 | n.d. | n.d. | n.d. | | n.d. | n.d. | n.d. | 1024 | |
| 9 | | | | | 344 | | | | | |
| 15 | 1004 | n.d. | n.d. | n.d. | 378 | n.d. | n.d. | n.d. | | |
| 30 | 1017 | n.d. | n.d. | n.d. | 398 | n.d. | n.d. | n.d. | | 390 |

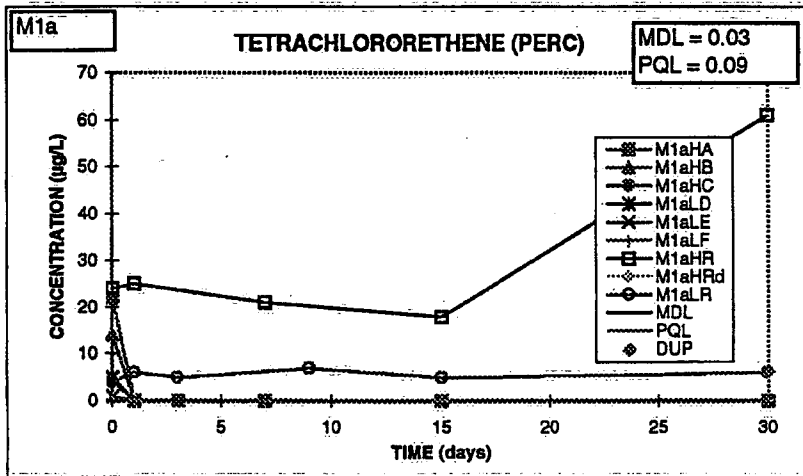


TETRACHLOROETHENE (PERC)

Microcosm 1a

Result concentration in µg/L

| Time | 35 | | | | | 6 | | | | r ² (f < 0.99) |
|------|-------|-------|-------|-------|-------|-------|-------|-------|--------|---------------------------|
| | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aHD | M1aHE | M1aHF | M1aLRd | |
| 0 | 24 | 22 | | | 4 | 5 | | | | 0.97 |
| 0.04 | | | 14 | | | | 1 | | | 0.93 (lowest range) |
| 0.08 | | | | 4 | | | | 0.4 | | |
| 1 | 25 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | | 0.96 |
| 3 | | n.d. | n.d. | n.d. | 5 | n.d. | n.d. | n.d. | | |
| 7 | 21 | n.d. | n.d. | n.d. | | n.d. | n.d. | n.d. | | 0.96 |
| 15 | 18 | n.d. | n.d. | n.d. | 7 | | | | | 0.902; 0.982 |
| 30 | 61 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | 6 | 0.938 |

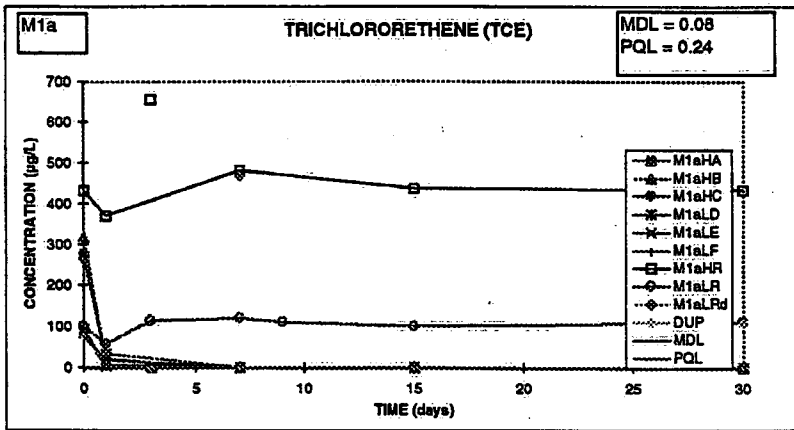


TRICHLOROETHENE (TCE)

Microcosm 1a

Result concentration in µg/L

| SITE WATER | | 86 | | | | | | | | | |
|------------|-------|-------|-------|-------|-------|-------|-------|-------|--------|--------|---------------------------|
| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aHRd | M1aLRd | (if not 0.00) |
| 0 | 432 | 269 | | | 99 | 85 | | | | | |
| 0.04 | | | 319 | | | | 93 | | | | |
| 0.08 | | | | 276 | | | | 80 | | | |
| 1 | 371 | 32 | 17 | 18 | 57 | 6 | 4 | 3 | | | 88 in calibration range B |
| 3 | 656 | n.d. | n.d. | n.d. | 114 | n.d. | n.d. | n.d. | | | |
| 7 | 479 | n.d. | n.d. | n.d. | 119 | n.d. | n.d. | n.d. | 469 | | |
| 9 | | | | | 110 | | | | | | |
| 15 | 437 | n.d. | n.d. | n.d. | 99 | n.d. | n.d. | n.d. | | | |
| 30 | 434 | n.d. | n.d. | n.d. | 111 | n.d. | n.d. | n.d. | | | 109 |



CHLOROFORM

Microcosm 1a

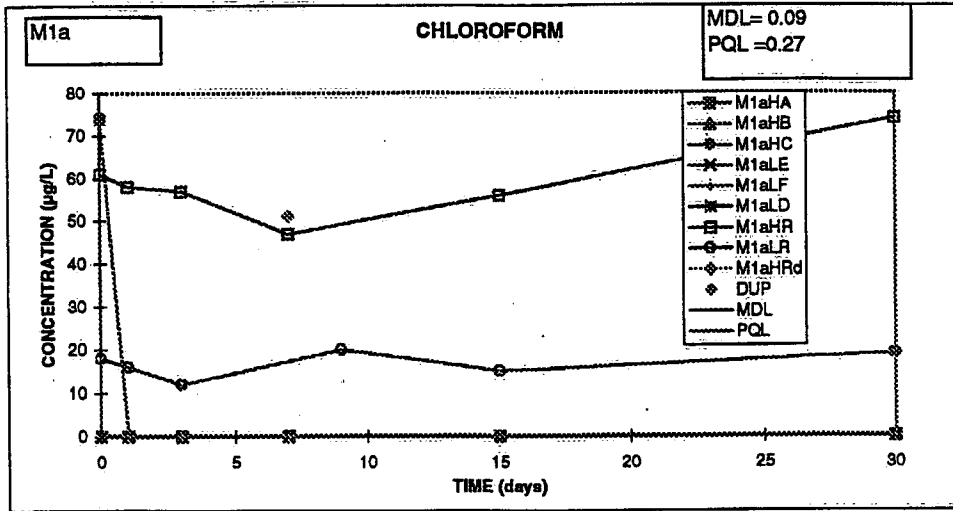
Result concentration in µg/L

SITE WATER

71

20

| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aHRd | M1aLRd | (I<0.99) |
|------|-------|-------|-------|-------|-------|-------|-------|-------|--------|--------|----------|
| 0 | 61 | 74 | | | 18 | n.d. | | | | | 0.97 |
| 0.04 | | | 0.1 | | | | n.d. | | | | |
| 0.08 | | | | n.d. | | | | n.d. | | | |
| 1 | 58 | n.d. | n.d. | n.d. | 16 | n.d. | n.d. | n.d. | | | 0.987 |
| 3 | 57 | n.d. | n.d. | n.d. | 12 | n.d. | n.d. | n.d. | | | |
| 7 | 47 | n.d. | n.d. | n.d. | | n.d. | n.d. | n.d. | 51 | | |
| 9 | | | | | 20 | | | | | | |
| 15 | 56 | n.d. | n.d. | n.d. | 15 | n.d. | n.d. | n.d. | | | 0.974 |
| 30 | 74 | n.d. | n.d. | n.d. | 19 | n.d. | n.d. | n.d. | | 19 | 0.978 |



cis-DICHLOROETHENE

Microcosm 1a

MAC = 70 µg/L

Result concentration in µg/L

SITE WATER

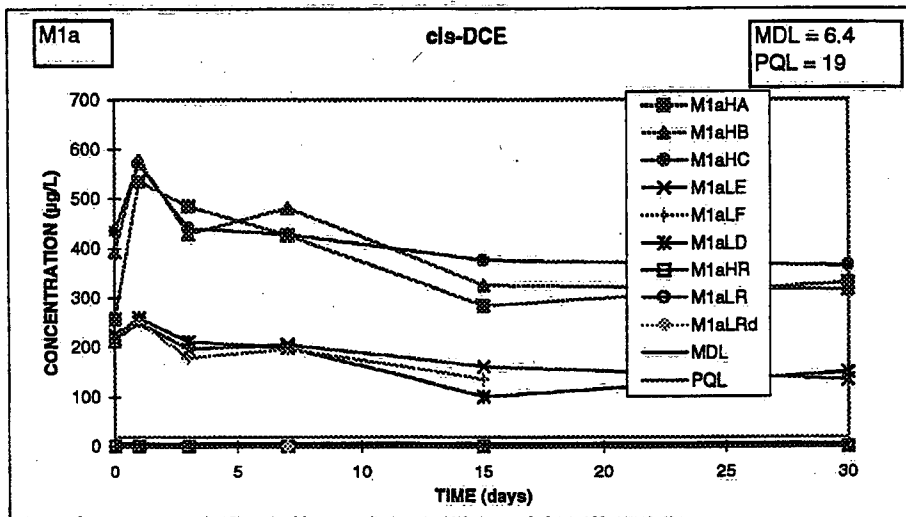
<PQL

<PQL

| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aHRd | M1aLRd |
|------|-------|-------|-------|-------|-------|-------|----------------|-------|--------|--------|
| 0 | n.d. | 257 | | | n.d. | 224 | | | | |
| 0.04 | | | 392 | | | | 215 | | | |
| 0.08 | | | | 434 | | | | 223 | | |
| 1 | n.d. | 535 | 580 | 571 | n.d. | 260 | 251 | 251 | | |
| 3 | n.d. | 485 | 429 | 440 | n.d. | 211 | 196 | 179 | | |
| 7 | n.d. | 427 | 481 | 428 | | 199 | 205 | 196 | n.d. | |
| 9 | | | | | n.d. | | | | | |
| 15 | n.d. | 284 | 325 | 376 | n.d. | 101 | 161 | 135 | | |
| 30 | n.d. | 331 | 317 | 365 | n.d. | 149 | 134 *exploded* | | | n.d. |

Predicted for day 15
Based on venting
day 30

304 333 300 147 198 139



Trans-DICHLOROETHENE

Microcosm 1a

MAC = 100 µg/L

Result concentration in µg/L

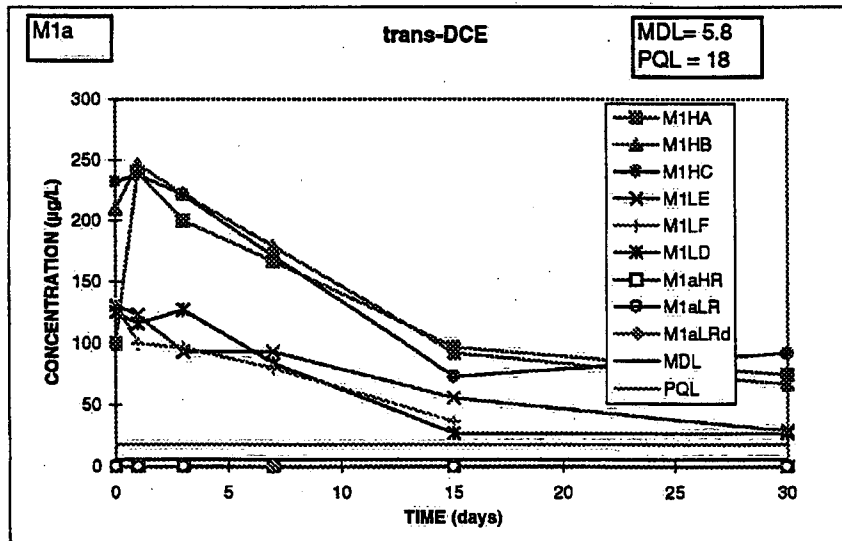
SITE WATER <PQL

<PQL

| Time | M1aHR | M1HA | M1HB | M1HC | M1aLR | M1LD | M1LE | M1LF | M1aHRd |
|------|-------|------|------|------|-------|------|------|------|--------|
| 0 | n.d. | 100 | | | n.d. | 125 | | | |
| 0.04 | | | 210 | | | | 131 | | |
| 0.08 | | | | 232 | | | | 133 | |
| 1 | <PQL | 240 | 246 | 238 | n.d. | 116 | 123 | 100 | |
| 3 | n.d. | 200 | 222 | 221 | n.d. | 127 | 93 | 96 | |
| 7 | n.d. | 167 | 179 | 171 | | 84 | 93 | 80 | n.d. |
| 9 | | | | | n.d. | | | | |
| 15 | n.d. | 97 | 92 | 73 | n.d. | 27 | 56 | 37 | |
| 30 | n.d. | 75 | 67 | 92 | n.d. | 27 | 29 | | |

Predicted for day 15

| | | | | | | |
|------------------|----|----|----|----|----|----|
| Based on venting | 70 | 68 | 69 | 40 | 87 | 33 |
|------------------|----|----|----|----|----|----|



VINYL CHLORIDE (VC)

Microcosm 1a

PQL = 11 µg/L

Result concentration in µg/L

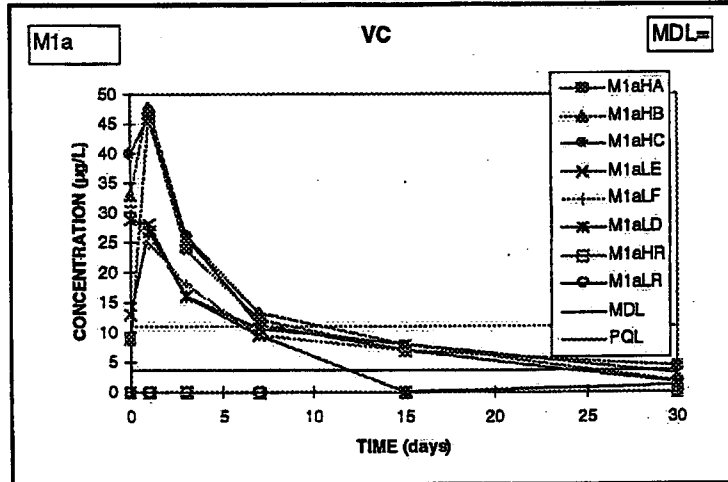
SITE WATER <PQL

<PQL

| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aHRd |
|------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| 0 | n.d. | 9 | | | n.d. | 13 | | | |
| 0.04 | | | 33 | | | | 29 | | |
| 0.08 | | | | 40 | | | | 31 | |
| 1 | n.d. | 46 | 48 | 46 | n.d. | 27 | 28 | 25 | |
| 3 | n.d. | 24 | 26 | 26 | n.d. | 16 | 16 | 18 | |
| 7 | n.d. | 12.1 | 13.3 | 11.2 | | 9.5j | 10.7j | 9.5j | n.d. |
| 9 | | | | | n.d. | | | | |
| 15 | n.d. | 7j | 8j | 7j | n.d. | n.d. | 8j | 7j | |

Predicted value

| | | | | | | | | |
|--------|------|------|------|------|------|------|------|------|
| Day 15 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
|--------|------|------|------|------|------|------|------|------|



ETHANE

Microcosm 1a

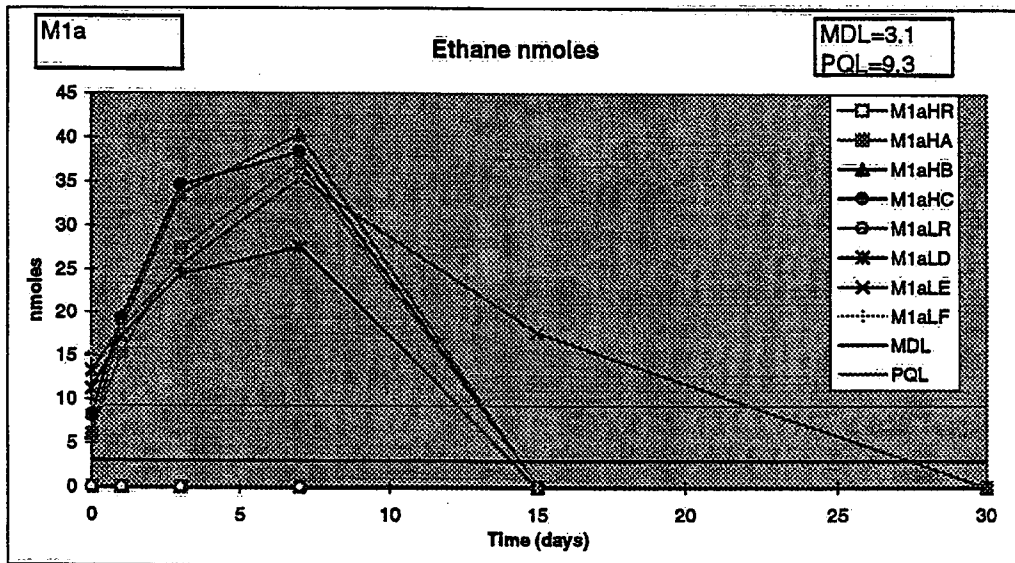
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aLRd |
|------|-------|-------|-------|-------|-------|-------|-----------------|-------|--------|
| 0 | n.d. | 6 | | | n.d. | 11 | | | |
| 0.04 | | | 6 | | | | 13 | | |
| 0.08 | | | | 8 | | | | 15 | |
| 1 | n.d. | 15 | 18 | 19 | n.d. | 17 | 18 | 18 | |
| 3 | n.d. | 27 | 33 | 34 | n.d. | 24 | 25 | 26 | |
| 7 | n.d. | 23 | 31 | 19 | n.d. | 15 | 26 | 21 | n.d. |
| 15 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 15 | n.d. | |
| 30 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. *exploded* | | |



ETHENE

Microcosm 1a

SITE WATER

<PQL

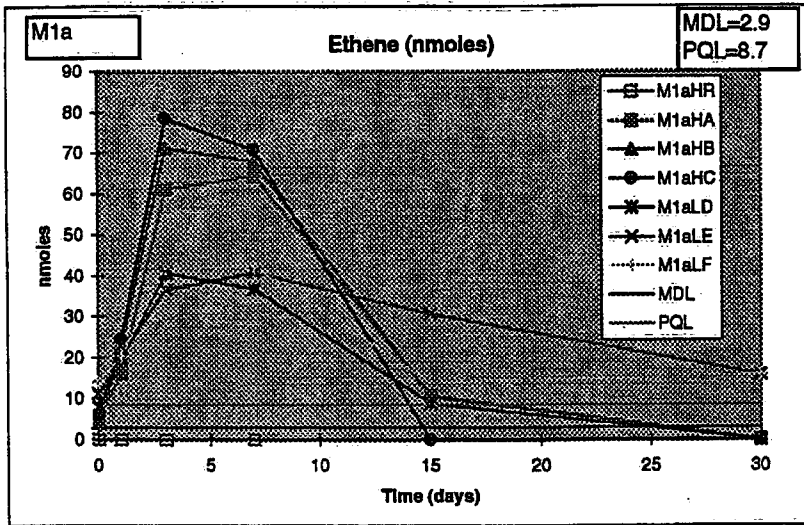
<PQL

Results in ppmv

| Time | M1aHR | M1aHA | M1aHB | M1aHC | M1aLR | M1aLD | M1aLE | M1aLF | M1aLRd |
|------|-------|-------|-------|-------|-------|-------|--------------|-------|--------|
| 0 | n.d. | 5 | | | n.d. | 8 | | | |
| 0.04 | | | 4 | | | | 11 | | |
| 0.08 | | | | 6 | | | | 14 | |
| 1 | n.d. | 16 | 22 | 24 | n.d. | 19 | 20 | 20 | |
| 3 | n.d. | 60 | 70 | 77 | n.d. | 40 | 36 | 41 | |
| 7 | n.d. | 52 | 52 | 52 | n.d. | 23 | 36 | 36 | n.d. |
| 15 | n.d. | 5j | 4j | n.d. | n.d. | 5j | 28 | 5j | |
| 30 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 6J*exploded* | | |

note volume has changed because of degassing
 Predicted for day 15
 Based on varying

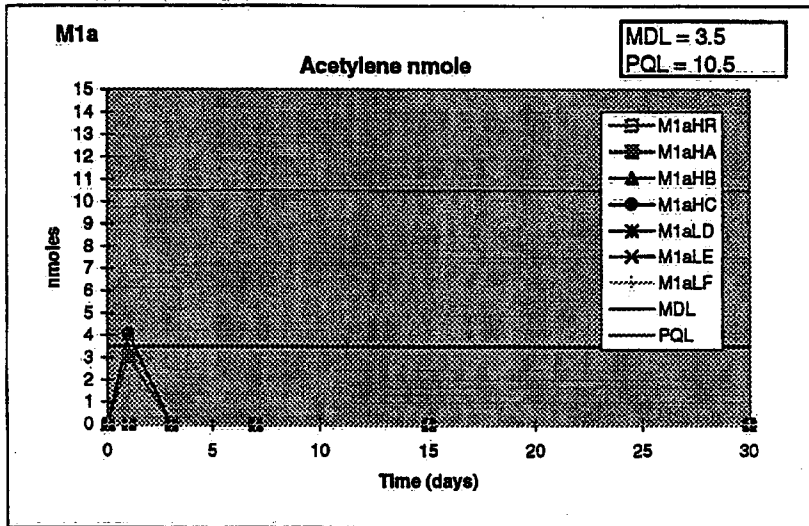
n.d. n.d. n.d. n.d. n.d. n.d. n.d.



ACETYLENE

Microcosm 1a

| Time | SITE WATER | | Results in ppmv | | | | | | |
|------|------------|------|-----------------|------|-------|------|------|------|------------|
| | M1aHR | M1HA | M1HB | M1HC | M1aLR | M1LD | M1LE | M1LF | M1aLRd |
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | 3 | 4 | 4 | n.d. | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 30 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | *exploded* |

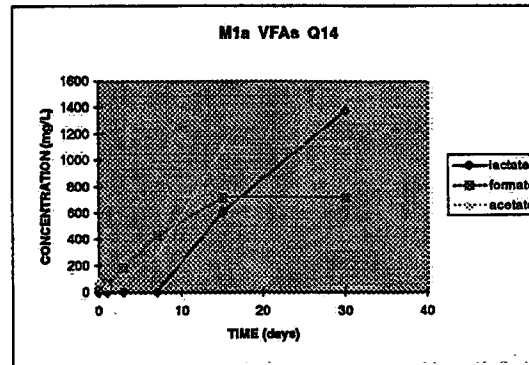
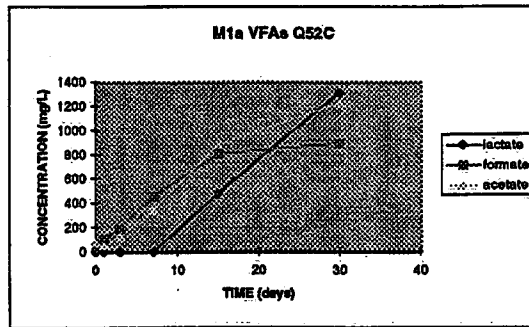


| Lactate | | conc. (mg/L) | | | | | |
|---------|-------|--------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 15 | 597 | 287 | 555 | 597 | 588 | 631 | |
| 30 | 1501 | 964 | 1475 | 1386 | 1153 | 1588 | |

| Formate | | conc. (mg/L) | | | | | |
|---------|-------|--------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | 36 | 40 | 39 | 41 | 37 | 37 | |
| 1 | 123 | 100 | 92 | 65 | 66 | 71 | |
| 3 | 190 | 192 | 208 | 171 | 182 | 212 | |
| 7 | 427 | 481 | 489 | 420 | 375 | 472 | |
| 15 | 649 | 1086 | 675 | 731 | 700 | 718 | |
| 30 | 758 | 1319 | 618 | 702 | 681 | 782 | |

| Acetate | | conc. (mg/L) | | | | | |
|---------|-------|--------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | 93 | n.d. | 62 | n.d. | 85 | 86 | |
| 1 | 60 | 53 | 50 | n.d. | 50 | 48 | |
| 3 | 128 | 114 | 134 | 118 | 116 | 101 | |
| 7 | 355 | 298 | 379 | 309 | 158 | 375 | |
| 15 | 1056 | 637 | 961 | 1096 | 1109 | 1062 | |
| 30 | 1365 | 759 | 1541 | 1100 | 1165 | 1139 | |

| Propionate | | conc. (mg/L) | | | | | |
|------------|-------|--------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 30 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |



| Titanium | | conc. (mM) | | | | | |
|----------|-------|------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | 14.9 | 15.6 | 15.3 | 14.8 | 15.2 | 15.0 | |
| 30 | 20.9 | 21.5 | - | 21.4 | 21.3 | 19.9 | |

| Chloride | | conc. (mg/L) | | | | | | | |
|----------|-------|--------------|-------|-------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | M1aLR | M1aHR | |
| 0 | 74.6 | 76.1 | 75.6 | 82.6 | 87.1 | 85.3 | 54.3 | 57.3 | |
| 30 | 31 | 85 | - | 118 | 76 | 78 | 5 | 33 | |

| Citrate M1a | | conc. (mg/L) | | | | | |
|-------------|-------|--------------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | |
| 0 | 6846 | 6682 | 4400 | 7774 | 7925 | 7670 | |
| 1 | 8219 | 7895 | 7399 | 7955 | 7785 | 8239 | |
| 3 | 7582 | 8120 | 8155 | 8068 | 8358 | 8023 | |
| 7 | 7007 | 7093 | 7817 | 7359 | 7300 | 7200 | |
| 15 | 6866 | 6928 | 6800 | 7597 | 7097 | 6932 | |
| 30 | 9743 | 9375 | 8618 | 9221 | 8382 | 9349 | |

| Glucose M1a | | conc. (mM) | | | | | | | |
|-------------|-------|------------|-------|-------|-------|-------|-------|-------|--|
| day | M1aLD | M1aLE | M1aLF | M1aHA | M1aHB | M1aHC | M1aLR | M1aHR | |
| 0 | 18.3 | 18.5 | 21.0 | 20.6 | 21.0 | 21.1 | 0.0 | 0.0 | |
| 1 | 18.9 | 19.0 | 19.9 | 18.2 | 19.7 | 19.4 | | | |
| 3 | 18.6 | 18.0 | 19.7 | 17.5 | 18.8 | 18.5 | | | |
| 7 | 14.6 | 15.0 | 14.5 | 12.6 | 13.7 | 12.8 | | | |
| 9 | 10.2 | 10.2 | 10.2 | 8.3 | 10.3 | 6.2 | | | |
| 11 | 6.2 | 7.7 | 5.2 | 4.2 | 5.8 | 2.5 | | | |
| 14 | 1.7 | 5.1 | 1.9 | 0.8 | 1.7 | 0.0 | | | |
| 16 | 16.5 | 21 | 17.5 | 19.2 | 17.6 | 18.9 | | | |
| 22 | 14.4 | 17.1 | 13.6 | 14.6 | 13.0 | 13.7 | | | |
| 25 | 13.4 | 14.8 | 13.4 | 14.0 | 11.4 | 12.5 | | | |
| 28 | 10.5 | 11.3 | 4.3 | 11.2 | 8.6 | 10.6 | | | |
| 30 | 12 | 12.5 | 0 | 10.7 | 9.7 | 10.3 | | | |

N.B. Microcosm M1aLF exploded over weekend, some of culture supernatant saved and transferred to a 60-mL serum vial (no soil present).

TETRACHLOROETHENE (PERC)

Microcosm 2

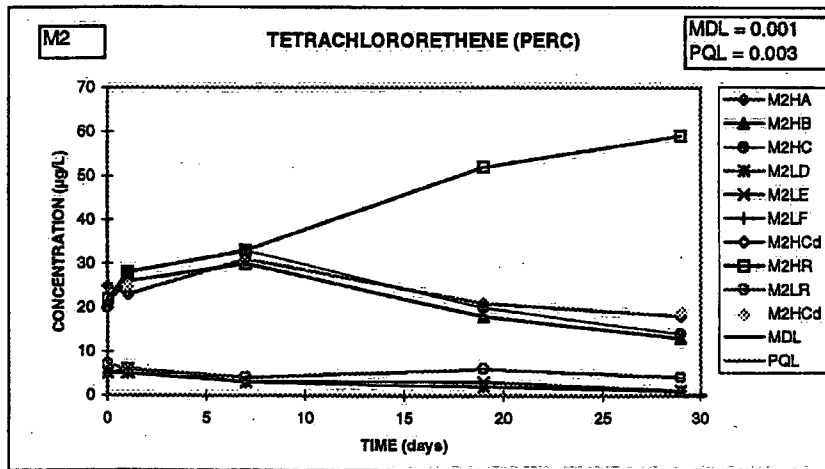
Result concentration in µg/L

SITE WATER

35

6

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd | |
|------|------|------|------|------|------|------|------|------|-------|---------------------------------|
| 0 | 22 | 25 | 21 | 20 | 7 | 5 | 5 | 5 | 5 | if less than 0.99 |
| 1 | 28 | 23 | 25 | 28 | 6 | 5 | 6 | 5 | 25 | 0.977 (D-F) and 0.963 (A-C) |
| 7 | 33 | 31 | 30 | 33 | 4 | 3 | 3 | 3 | | single point calibration |
| 19 | 52 | 21 | 18 | 20 | 6 | 2 | 3 | 2 | | B: 0.962; C: 0.938 |
| 29 | 59 | 18 | 13 | 14 | 4 | 1 | 1 | 1 | | 19 B: 0.976; C: 0.901; D: 0.979 |
| 36 | | | | | | | | | | |



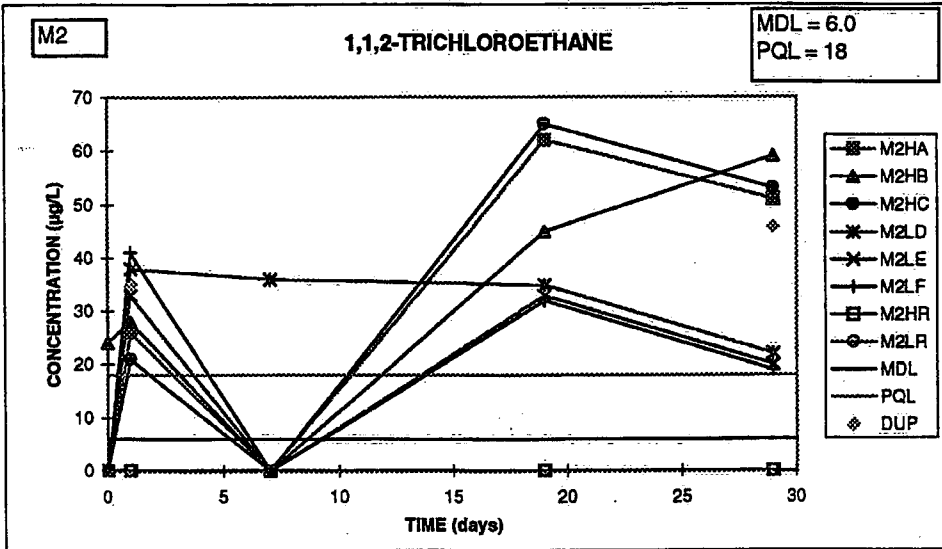
1,1,2-Trichloroethane

Microcosm 2

Result concentration in µg/L

SITE WATER

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | DUP |
|------|------|------|------|------|------|------|------|------|-----|
| 0 | n.d. | n.d. | 24 | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | 26 | 28 | 21 | n.d. | 38 | 33 | 41 | 35 |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | 36 | n.d. | n.d. | |
| 19 | n.d. | 62 | 45 | 65 | n.d. | 35 | 33 | 32 | |
| 29 | n.d. | 51 | 59 | 53 | n.d. | 22 | 20 | 19 | 46 |
| 30 | | | | | | | | | |

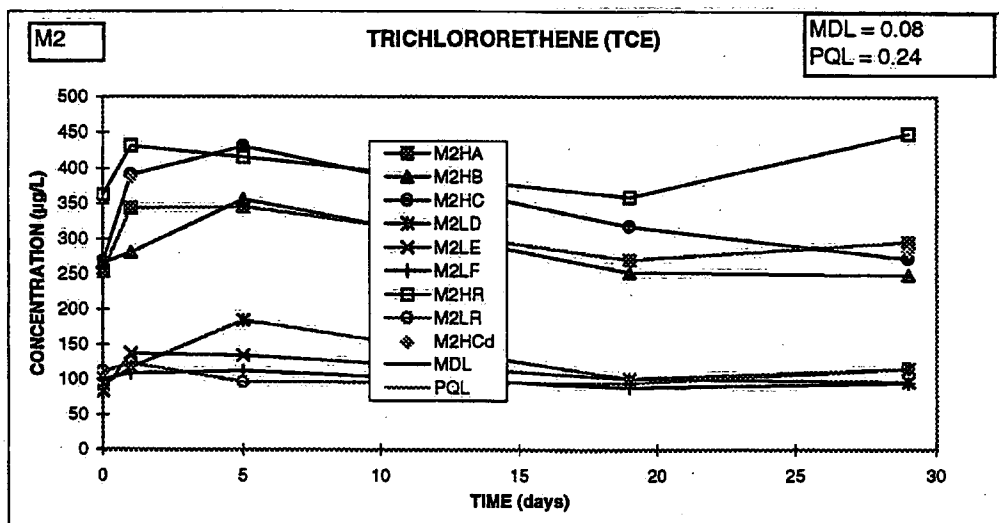


TRICHLOROETHENE (TCE)

Microcosm 2

Result concentration in µg/L

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | 363 | 254 | 266 | 268 | 111 | 92 | 83 | 100 | |
| 1 | 432 | 344 | 281 | 391 | 123 | 117 | 137 | 109 | 387 |
| 5 | 416 | 346 | 356 | 431 | 97 | 185 | 135 | 113 | |
| 19 | 359 | 270 | 253 | 317 | 95 | 101 | 101 | 89 | |
| 29 | 449 | 295 | 248 | 271 | 115 | 115 | 97 | 95 | 283 |
| 30 | | | | | | | | | |



CHLOROFORM

Microcosm 2

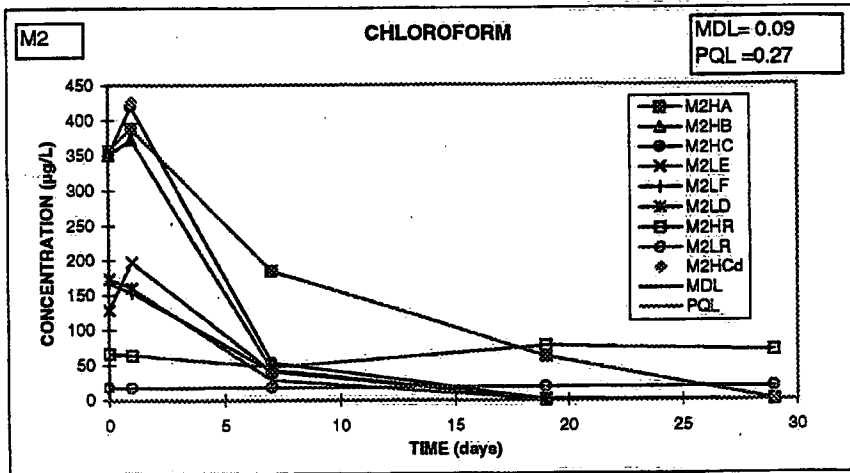
Result concentration in µg/L

SITE WATER

71

20

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd | if less than 0.99 |
|------|------|------|------|------|------|------|------|------|-------|--------------------------|
| 0 | 66 | 356 | 350 | 351 | 17 | 174 | 129 | 167 | | 0.977 |
| 1 | 64 | 388 | 372 | 420 | 17 | 160 | 198 | 154 | 426 | 0.972 |
| 7 | 47 | 185 | 44 | 54 | 18 | 29 | 41 | 41 | | single point calibration |
| 19 | 78 | 63 | n.d. | 1 | 19 | n.d. | 2 | n.d. | | |
| 29 | 71 | n.d. | n.d. | n.d. | 19 | n.d. | n.d. | n.d. | n.d. | 0.975 |
| 30 | | | | | | | | | | |



DICHLOROMETHANE

Microcosm 2

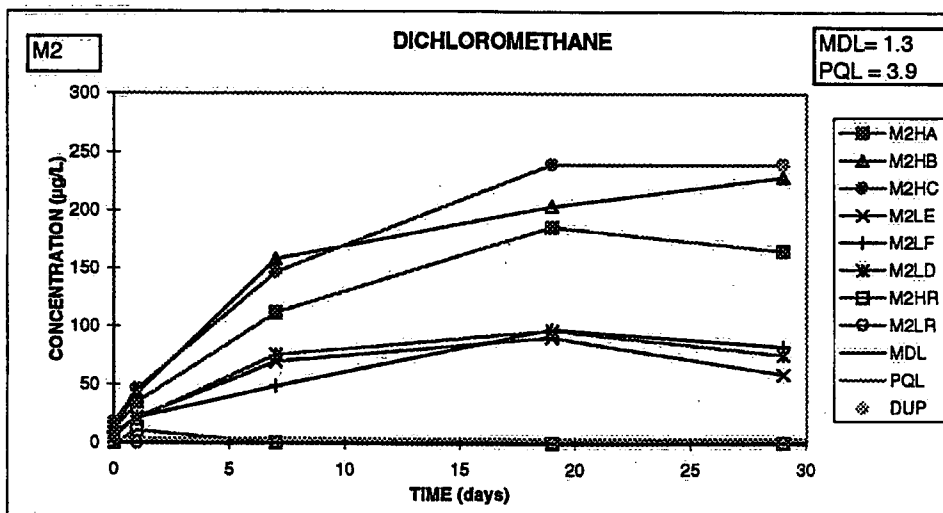
MAC = 5 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | DUP |
|------|------|------|------|------|------|------|------|------|-----|
| 0 | n.d. | 11 | 13 | 17 | n.d. | 6 | 5 | 8 | |
| 1 | 11 | 35 | 43 | 46 | n.d. | 21 | 22 | 22 | |
| 7 | n.d. | 112 | 158 | 147 | n.d. | 76 | 70 | 49 | |
| 19 | n.d. | 186 | 204 | 240 | n.d. | 97 | 91 | 98 | |
| 29 | n.d. | 165 | 229 | 240 | n.d. | 76 | 59 | 83 | 239 |
| 30 | | | | | | | | | |



Trans-DICHLOROETHENE

Microcosm 2

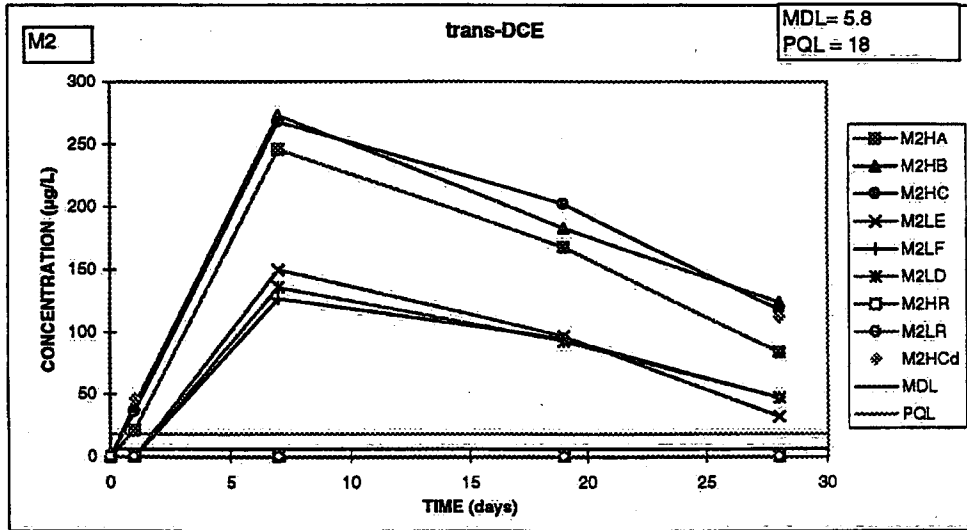
MAC = 100 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd |
|------|------|------|------|------|------|------|------|------|--------------------------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | 21 | 41 | 36 | n.d. | n.d. | n.d. | n.d. | 46 |
| 7 | n.d. | 246 | 273 | 269 | n.d. | 136 | 150 | 127 | single point calibration |
| 19 | n.d. | 168 | 183 | 202 | n.d. | 93 | 97 | 95 | |
| 28 | n.d. | 84 | 124 | 118 | n.d. | 47 | 32 | 47 | 112 |
| 30 | | | | | | | | | |



CHLOROMETHANE

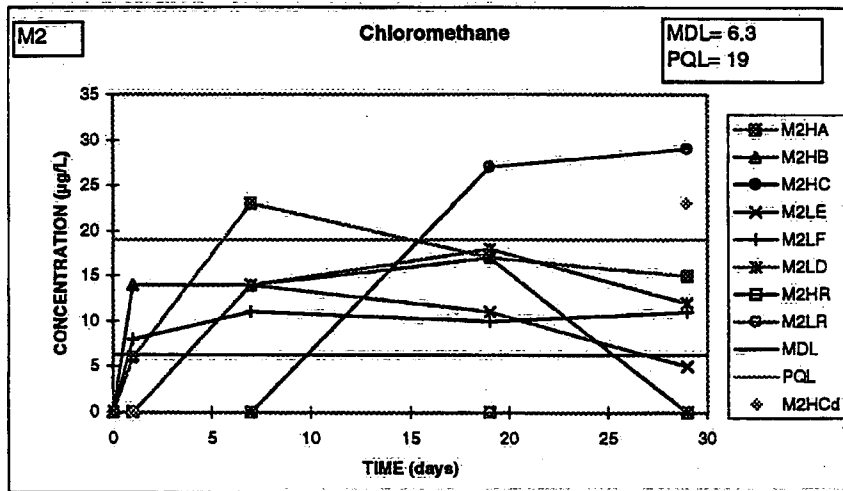
Microcosm 2

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd |
|------|------|------|------|------|------|------|------|------|--------------------------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | 6 | 14 | n.d. | n.d. | n.d. | n.d. | 8 | n.d. |
| 7 | n.d. | 23 | 14 | n.d. | n.d. | 14 | 14 | 11 | single point calibration |
| 19 | n.d. | 17 | 17 | 27 | n.d. | 18 | 11 | 10 | |
| 29 | n.d. | 15 | n.d. | 29 | n.d. | 12 | 5 | 11 | 23 |
| 30 | | | | | | | | | |



ETHENE

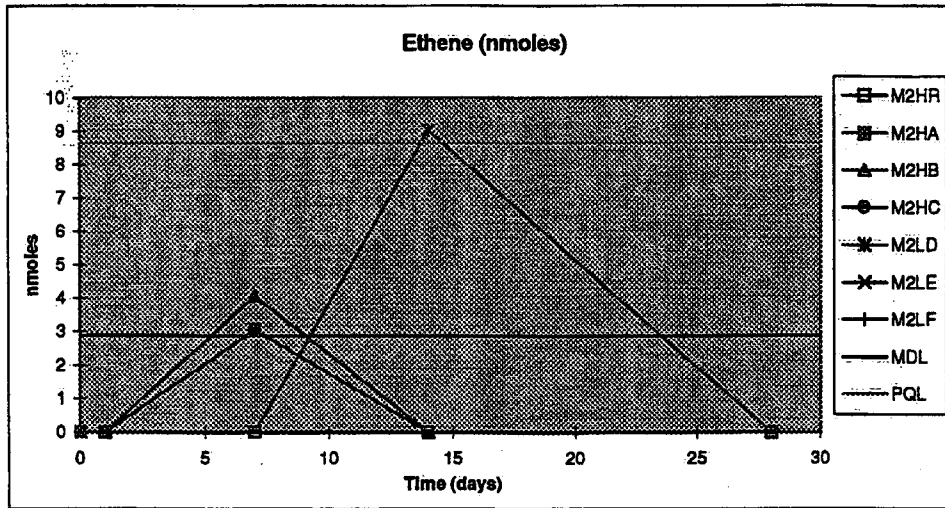
Microcosm 2

SITE WATER

<PQL

<PQL

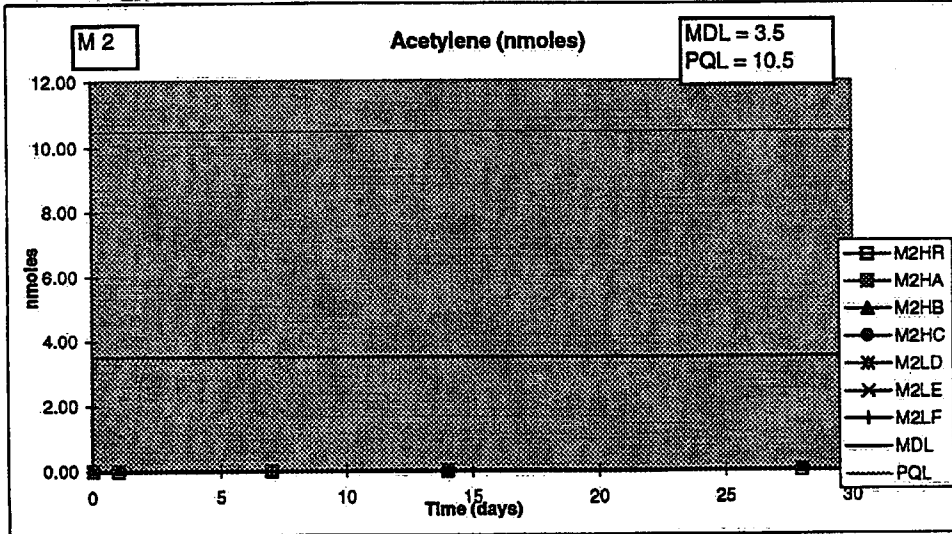
| Time | nmoles | | | | | | | | | |
|------|--------|------|------|------|------|------|------|------|-------|------|
| | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | 3 | 4 | 3 | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 19 | n.d. | n.d. | n.d. | n.d. | n.d. | 3 | n.d. | n.d. | n.d. | |
| 29 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 30 | | | | | | | | | | |



ACETYLENE

Microcosm 2

| Time | nmoles | | | | <PQL | | | | | |
|------|--------|------|------|------|------|------|------|------|-------|------|
| | M2HR | M2HA | M2HB | M2HC | M2LR | M2LD | M2LE | M2LF | M2HCd | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 19 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 29 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 30 | | | | | | | | | | |



Citrate 2

| day | M2LD | M2LE | conc. (mg/L) | | | |
|-----|------|------|--------------|------|------|------|
| | | | M2LF | M2HA | M2HB | M2HC |
| 0 | 6799 | 6919 | 6905 | 7082 | 6851 | 6802 |
| 1 | 7334 | 7092 | 7182 | 7522 | 7098 | 7531 |
| 5 | 7019 | 7126 | 6931 | 7710 | 7683 | 7389 |
| 19 | 9301 | 9821 | 8076 | 8879 | 9789 | 8934 |
| 29 | 9565 | 9575 | 8874 | 9376 | 8992 | 8730 |

Note: Microcosms were amended with Ti(III)citrate-reduced invertose on day 16, hence the increase in glucose and citrate concentrations.

Glucose 2

| day | M2LD | M2LE | conc. (mM) | | | |
|-----|------|------|------------|------|------|------|
| | | | M2LF | M2HA | M2HB | M2HC |
| 0 | 18.5 | 18.9 | 17.3 | 21.3 | 18.5 | 19.0 |
| 1 | 18.1 | 19.1 | 17.8 | 18.0 | 17.1 | 19.6 |
| 5 | 17.6 | 16.2 | 17.0 | 17.7 | 16.4 | 17.5 |
| 8 | 14.0 | 11.7 | 12.0 | 11.0 | 7.6 | 11.4 |
| 13 | 7.8 | 4.2 | 5.8 | 7.4 | 5.1 | 6.8 |
| 16 | 4.6 | 1.3 | 2.2 | 4.7 | 1.6 | 2.8 |
| 16 | 24.9 | 16.7 | 21.3 | 20.0 | 19.0 | 19.7 |
| 19 | 22.7 | 21.6 | 18.7 | 20.1 | 17.6 | 18.1 |
| 26 | 13.9 | 11.5 | 12.8 | 14.1 | 13.2 | 14.2 |
| 29 | 15.2 | 11.2 | 11.1 | 14.0 | 11.8 | 12.0 |

Volatile Fatty Acids-M2

Lactate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 5 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 19 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 29 | 320 | 872 | 289 | 184 | 289 | 296 |

Formate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | .47 | n.d. | 44 | 31 | 41 | 46 |
| 5 | 288 | 126 | 123 | 96 | 144 | 118 |
| 19 | 1229 | 1452 | 1097 | 1091 | 1404 | 1223 |
| 29 | 1745 | 1895 | 1632 | 1659 | 1884 | 1578 |

Acetate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 5 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 19 | 584 | 790 | 516 | 559 | 717 | 617 |
| 29 | 894 | 1004 | 841 | 881 | 875 | 852 |

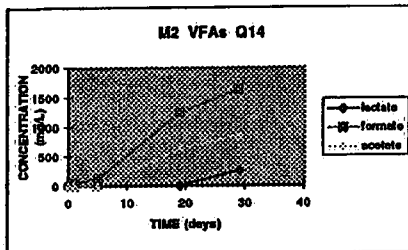
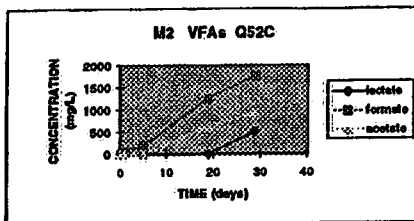
Propionate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 5 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 19 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 29 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

Notes: Problems with M2LD5 VFA analysis required a re-analysis of this sample on day 7. Results shown for day 5 are day 7 results.

| Q52C day | lactate | formate | acetate |
|-------------|---------|---------|---------|
| | lactate | formate | acetate |
| 0 | 0 | 0 | 0 |
| 1 | 0 | 46 | 0 |
| 5 | 0 | 179 | 0 |
| 19 | 0 | 1260 | 630 |
| 29 | 494 | 1757 | 813 |

| Q14 day | lactate | formate | acetate |
|------------|---------|---------|---------|
| | lactate | formate | acetate |
| 0 | 0 | 0 | 0 |
| 1 | 0 | 40 | 0 |
| 5 | 0 | 120 | 0 |
| 19 | 0 | 1239 | 631 |
| 29 | 256 | 1640 | 869 |



Titanium

| day | conc. (mM) | | | | | |
|-----|------------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC |
| 0 | 26.9 | 27.4 | 26.9 | 25.4 | 29.1 | 28.9 |
| 30 | 33.2 | 33.6 | 32.5 | 33.9 | 32.9 | 34.8 |

Chloride

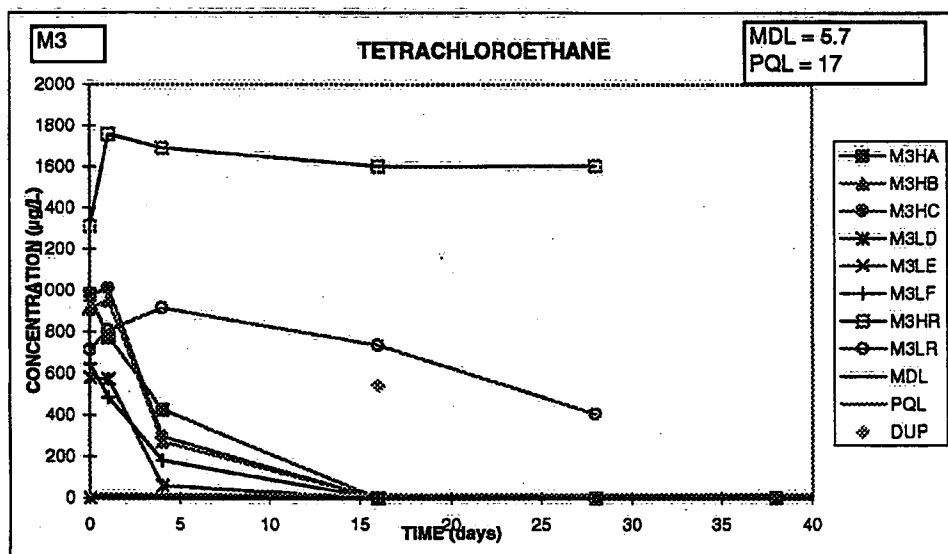
| day | conc. (mg/L) | | | | | | | |
|-----|--------------|------|------|------|------|------|------|------|
| | M2LD | M2LE | M2LF | M2HA | M2HB | M2HC | M2LR | M2HR |
| 0 | 74.2 | 75.5 | 75.1 | 81.7 | 85.9 | 84.2 | 22.1 | 53.6 |

Note: Microcosms were amended with Ti(II) citrate-reduced invertose on day 16, hence the increase in glucose and citrate concentrations.

Tetrachloroethane (TeCA) Microcosm 3 (titanium citrate + yeast extract)

Result concentration in µg/L

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | DUP |
|------------|------|------|------|------|------|------|------|------|--------------------|
| SITE WATER | 2114 | | | | | 695 | | | |
| 0 | 1311 | 978 | 912 | 978 | 711 | 578 | 582 | 645 | |
| 1 | 1759 | 773 | 953 | 1008 | 810 | 573 | 495 | 486 | |
| 4 | 1692 | 426 | 273 | 296 | 914 | 62 | 201 | 183 | |
| 16 | 1604 | n.d. | n.d. | n.d. | 735 | n.d. | n.d. | 7j | 543 |
| 28 | 1607 | n.d. | n.d. | n.d. | 405 | n.d. | n.d. | n.d. | B: 0.974, C: 0.983 |
| 38 | | n.d. | | | | | | | |
| 40 | | | | | | | | | |



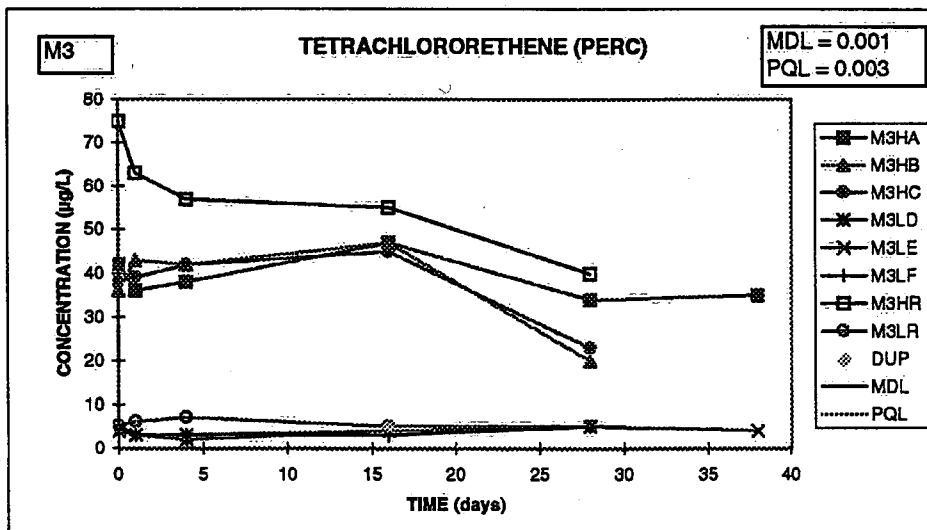
Tetrachloroethene (PERC) Microcosm 3 (titanium citrate + yeast extract)

Result concentration in µg/L

SITE WATER 35 6

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | DUP |
|------|------|------|------|------|------|------|------|------|-----|
| 0 | 75 | 42 | 36 | 38 | 5 | 4 | 4 | 5 | |
| 1 | 63 | 36 | 43 | 39 | 6 | 3 | 3 | 3 | |
| 4 | 57 | 38 | 42 | 42 | 7 | 2 | 3 | 3 | |
| 16 | 55 | 47 | 47 | 45 | 5 | 4 | 4 | 3 | |
| 28 | 40 | 34 | 20 | 23 | 5 | 5 | 5 | 5 | |
| 38 | | 35 | | | | | 4 | | |
| 40 | | | | | | | | | |

5
 (less than 0.99
 B: 0.969; D:0.939
 B: 0.976; D:0.939
 C: 0.901; D:0.979



1,1,2-Trichloroethane

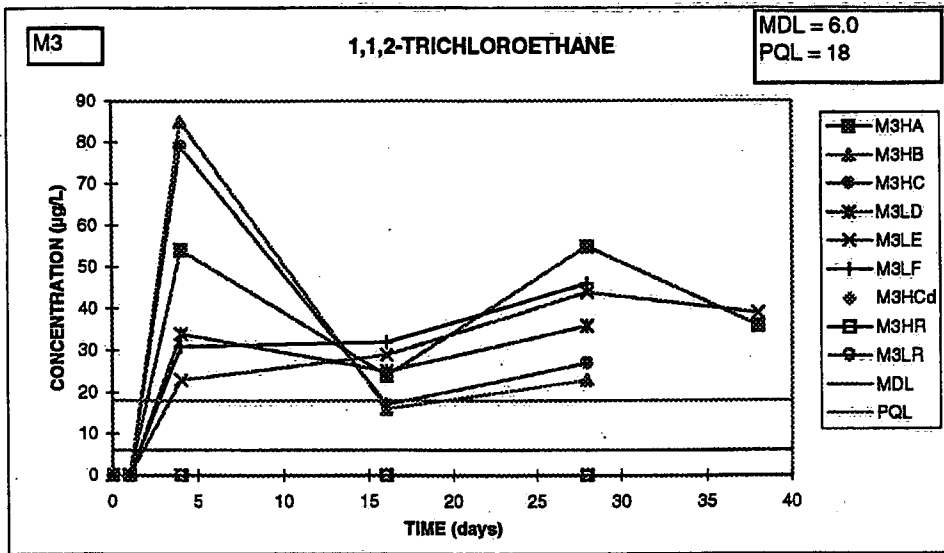
Microcosm 3

(titanium citrate + yeast extract)

Result concentration in µg/L

SITE WATER

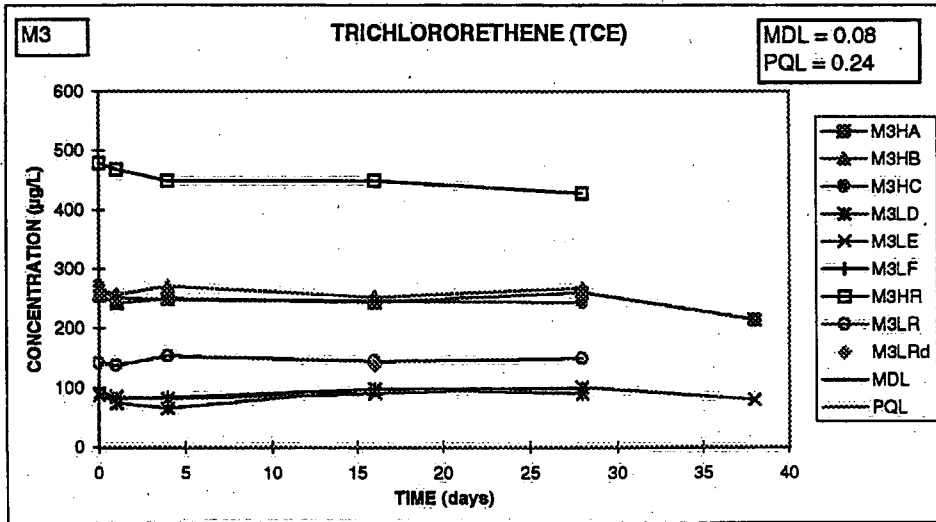
| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3HCd | |
|------|------|------|------|------|------|------|------|------|-------|-------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 4 | n.d. | 54 | 85 | 79 | n.d. | 34 | 29 | 31 | | 0.954 |
| 16 | n.d. | 24 | 16 | 17 | n.d. | 25 | 29 | 32 | | |
| 28 | n.d. | 55 | 23 | 27 | n.d. | 36 | 44 | 46 | | |
| 38 | | 36 | | | | | 39 | | | |
| 40 | | | | | | | | | | |



TRICHLOROETHENE (TCE) Microcosm 3 (titanium citrate + yeast extract)

Result concentration in µg/L

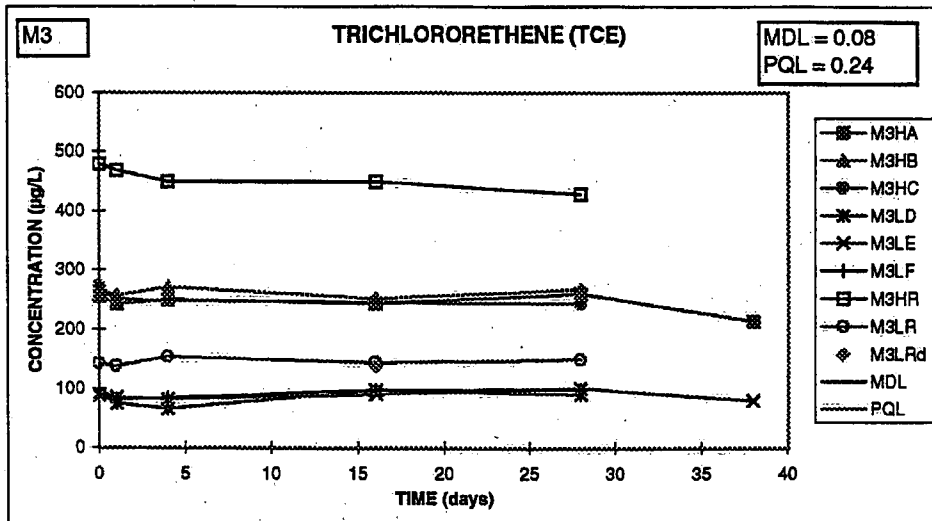
| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | 479 | 255 | 257 | 271 | 142 | 90 | 88 | 96 | |
| 1 | 469 | 243 | 257 | 251 | 138 | 74 | 85 | 82 | |
| 4 | 450 | 250 | 271 | 248 | 154 | 66 | 82 | 84 | |
| 16 | 449 | 244 | 252 | 246 | 144 | 97 | 90 | 97 | 139 |
| 28 | 428 | 261 | 268 | 244 | 150 | 90 | 101 | 99 | |
| 38 | | 214 | | | | | 80 | | |
| 40 | | | | | | | | | |



TRICHLOROETHENE (TCE) Microcosm 3 (titanium citrate + yeast extract)

Result concentration in µg/L

| SITE WATER | | 197 | | | | | | | | 86 | |
|------------|------|------|------|------|------|------|------|------|-------|-----|--|
| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3LRd | | |
| 0 | 479 | 255 | 257 | 271 | 142 | 90 | 88 | 96 | | | |
| 1 | 469 | 243 | 257 | 251 | 138 | 74 | 85 | 82 | | | |
| 4 | 450 | 250 | 271 | 248 | 154 | 66 | 82 | 84 | | | |
| 16 | 449 | 244 | 252 | 246 | 144 | 97 | 90 | 97 | | 139 | |
| 28 | 428 | 261 | 268 | 244 | 150 | 90 | 101 | 99 | | | |
| 38 | | 214 | | | | | 80 | | | | |
| 40 | | | | | | | | | | | |



DICHLOROMETHANE

Microcosm 3

(titanium citrate + yeast extract)

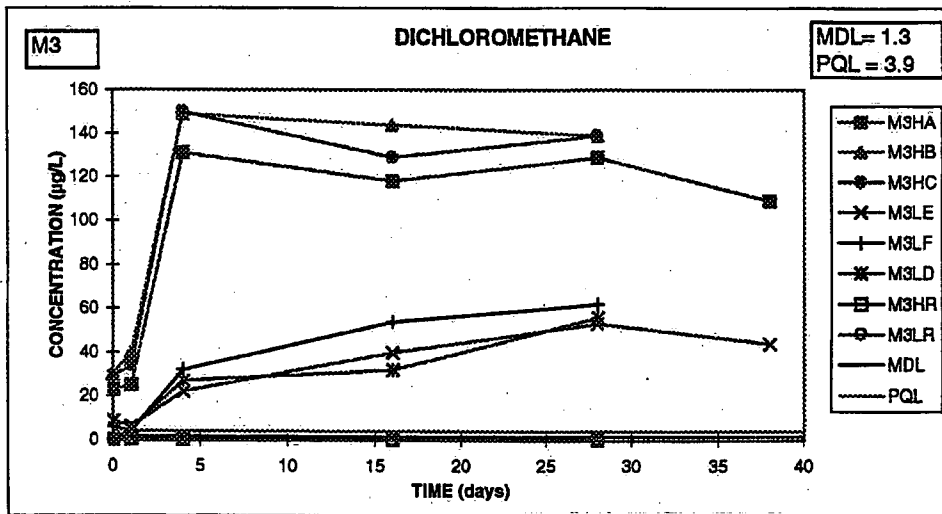
MAC = 5 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3LRd | r ² |
|------|------|------|------|------|------|------|------|------|-------|----------------|
| 0 | n.d. | 23 | 30 | 29 | n.d. | n.d. | 9 | 6 | | 0.93 |
| 1 | n.d. | 25 | 40 | 34 | n.d. | 5 | 6 | 4 | | 0.923 |
| 4 | n.d. | 131 | 149 | 150 | n.d. | 27 | 22 | 32 | | 0.975 |
| 16 | n.d. | 118 | 144 | 129 | n.d. | 32 | 40 | 54 | n.d. | |
| 28 | n.d. | 129 | 139 | 139 | n.d. | 56 | 53 | 62 | | 0.962 |
| 38 | | 109 | | | | | 44 | | | |
| 40 | | | | | | | | | | |



VINYL CHLORIDE (VC)

Microcosm 3

(titanium citrate + yeast extract)

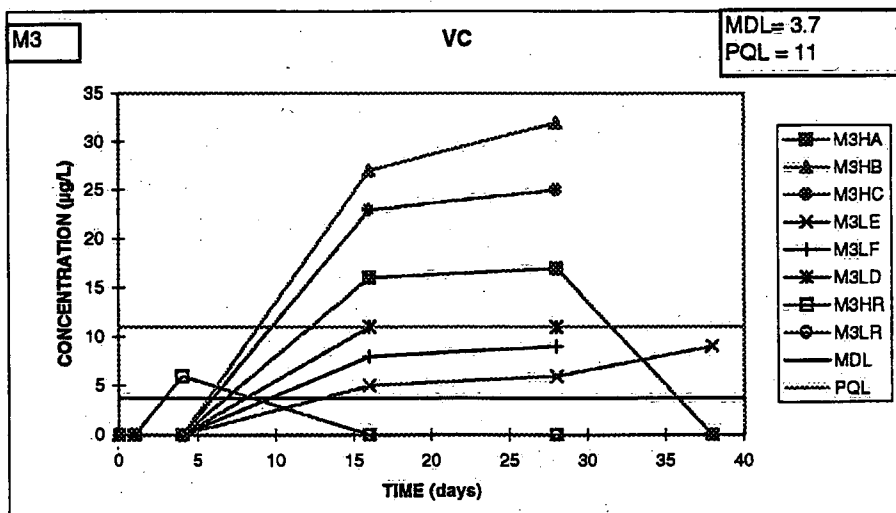
Result concentration in µg/L

SITE WATER

<PQL

<PQL

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3HCd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 4 | 6 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | n.d. | 16 | 27 | 23 | n.d. | 11 | 5 | 8 | n.d. |
| 28 | n.d. | 17 | 32 | 25 | n.d. | 11 | 6 | 9 | n.d. |
| 38 | | n.d. | | | | | 9 | | |
| 40 | | | | | | | | | |



CHLOROMETHANE

Microcosm 3

(titanium citrate + yeast extract)

Result concentration in µg/L

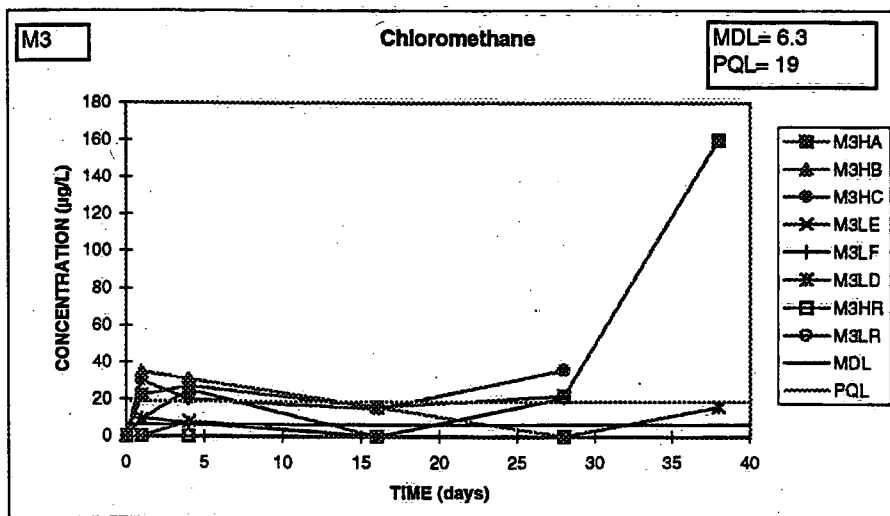
SITE WATER

<PQL

<PQL

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3HCd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | 22 | 35 | 30 | n.d. | 9 | n.d. | 10 | |
| 4 | n.d. | 27 | 31 | 20 | n.d. | 25 | 8 | 7 | |
| 16 | n.d. | 16 | 16 | 15 | n.d. | n.d. | n.d. | n.d. | |
| 28 | n.d. | 22 | n.d. | 36 | n.d. | n.d. | n.d. | 21 | |
| 38 | | 160 | | | | 16 | | | |
| 40 | | | | | | | | | |

need to confirm by GC/MS that it is indeed chloromethane



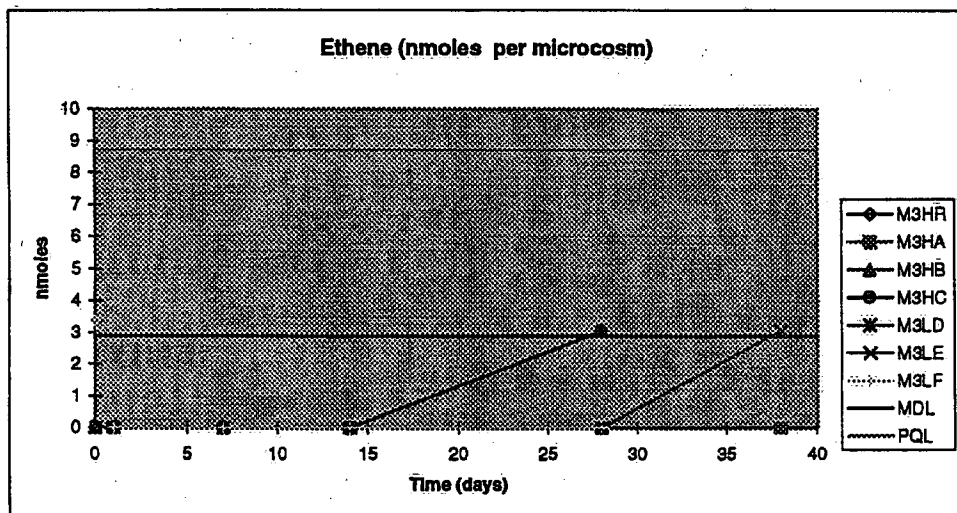
ETHENE

Microcosm 3 (titanium citrate + yeast extract)

SITE WATER <PQL

<PQL

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF | M3HCd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | n.d. | 3 | n.d. | n.d. | n.d. | | 3 |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | | n.d. |
| 4 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | | n.d. |
| 16 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | | n.d. |
| 28 | n.d. | n.d. | n.d. | 3 | n.d. | n.d. | n.d. | | n.d. |
| 38 | | n.d. | | | | | 3 | | |
| 40 | | | | | | | | | |



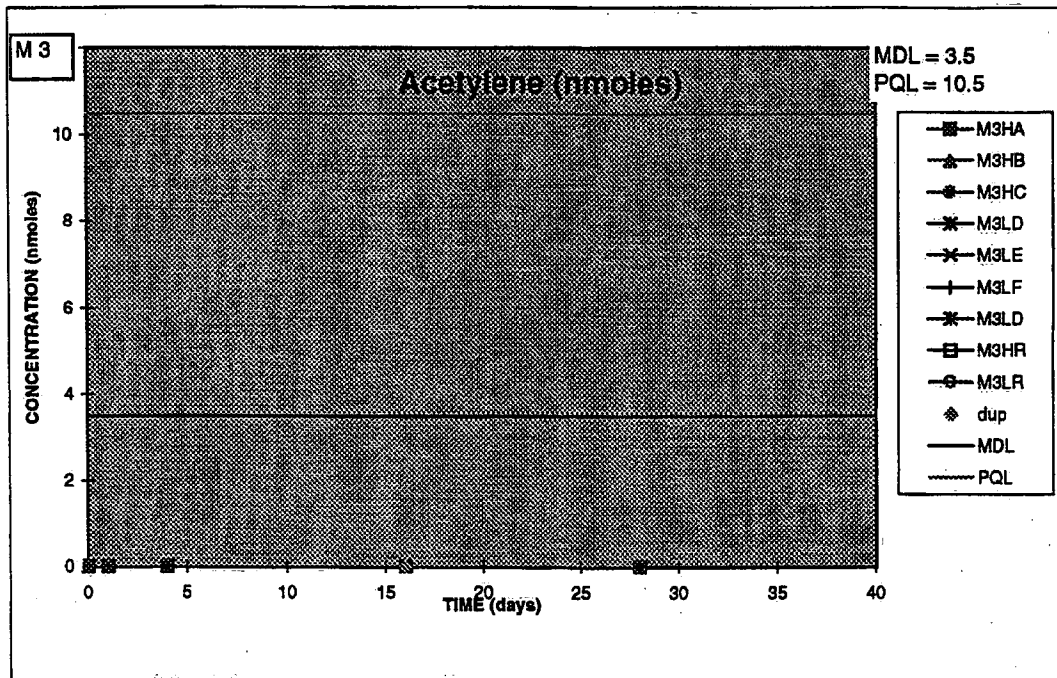
ACETYLENE

Microcosm 3 (titanium citrate + yeast extract)

SITE WATER <PQL

<PQL

| Time | M3HR | M3HA | M3HB | M3HC | M3LR | M3LD | M3LE | M3LF dup |
|------|------|------|------|------|------|------|------|-----------|
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 4 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 38 | | n.d. | | | | | n.d. | |
| 40 | | | | | | | | |



Citrate 3

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|-------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | 6722 | 6216 | 6229 | 6815 | 6920 | 7319 |
| 1 | 880j | 926j | 929j | 902j | 1018j | 937j |
| 4 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | 938j | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

Volatile Fatty Acids-M3

Lactate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 4 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

Formate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | 78 | 132 | 213 | 84 | 75 | 55 |
| 1 | 659 | 983 | 1025 | 698 | 1013 | 657 |
| 4 | 982 | 697 | 698 | 917 | 1056 | 1025 |
| 16 | n.d. | 650 | n.d. | n.d. | n.d. | 350 |
| 28 | n.d. | n.d. | 330 | n.d. | 228 | n.d. |

Acetate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | 307 | 439 | 654 | 381 | 397 | 325 |
| 1 | 3536 | 3949 | 4242 | 3640 | 3975 | 3801 |
| 4 | 4439 | 3945 | 4199 | 3952 | 4438 | 4381 |
| 16 | 5177 | 5094 | 5644 | 4353 | 5021 | 5660 |
| 28 | 4973 | 6210 | 5261 | 4793 | 5632 | 5540 |

Propionate

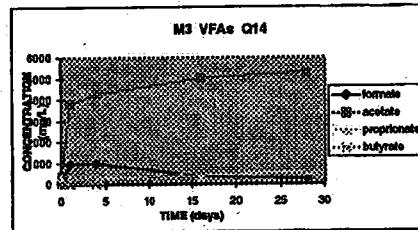
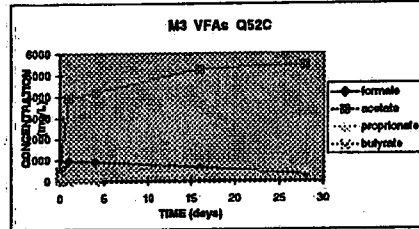
| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | 233 | 239 | 235 | 274 | 281 | n.d. |
| 1 | 263 | 194 | 240 | 263 | 302 | 266 |
| 4 | 212 | 168 | 226 | 254 | 282 | 279 |
| 16 | 414 | 489 | 374 | 223 | 421 | 518 |
| 28 | 425 | 603 | 558 | 429 | 722 | 600 |

Butyrate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 4 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | n.d. | 1733 | 1683 | n.d. | n.d. | 1740 |
| 28 | n.d. | 1991 | 1969 | n.d. | 1964 | n.d. |

Titanium

| day | M3LD | M3LE | M3LF | M3HA | M3HB | M3HC |
|-----|------|------|------|------|------|------|
| 0 | 29.0 | 26.5 | 29.0 | 31.4 | 30.6 | 28.7 |



| Q52C day | formate | acetate | propionate | butyrate | 0 | 1733 | 1693 |
|----------|---------|---------|------------|----------|---|------|------|
| | formate | acetate | propionate | butyrate | | | |
| 0 | 141 | 468 | 235 | 0 | 0 | 1733 | 1693 |
| 1 | 955 | 3909 | 239 | 0 | 0 | 1991 | 1969 |
| 4 | 928 | 4185 | 203 | 0 | | | |
| 16 | 650 | 5305 | 426 | 1142 | | | |
| 28 | 330 | 5481 | 529 | 1920 | | | |

| Q14 day | formate | acetate | propionate | butyrate | 0 | 266 | 279 | 518 | 600 |
|---------|---------|---------|------------|----------|-----|------|-----|-----|-----|
| | formate | acetate | propionate | butyrate | | | | | |
| 0 | 233 | 239 | 235 | 274 | 281 | n.d. | | | |
| 1 | 263 | 194 | 240 | 263 | 302 | 266 | | | |
| 4 | 212 | 168 | 226 | 254 | 282 | 279 | | | |
| 16 | 414 | 489 | 374 | 223 | 421 | 518 | | | |
| 28 | 425 | 603 | 558 | 429 | 722 | 600 | | | |

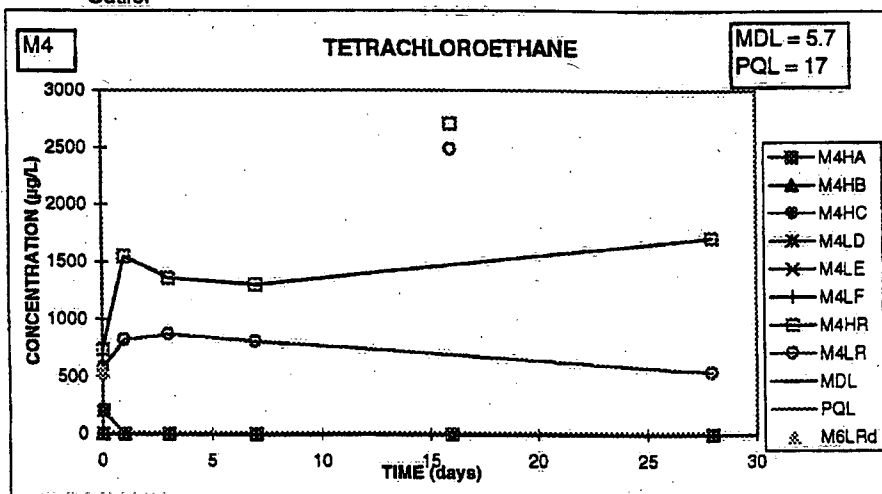
| Q14 day | formate | acetate | propionate | butyrate | 0 | 266 | 279 | 518 | 580 |
|---------|---------|---------|------------|----------|---|-----|-----|-----|-----|
| | formate | acetate | propionate | butyrate | | | | | |
| 0 | 65 | 368 | 277 | 0 | | | | | |
| 1 | 956 | 3805 | 277 | 0 | | | | | |
| 4 | 999 | 4256 | 275 | 0 | | | | | |
| 16 | 350 | 5011 | 387 | 580 | | | | | |
| 28 | 228 | 5322 | 584 | 681.3333 | | | | | |

Tetrachloroethane (TeCA) Microcosm 4 Sterile control

Result concentration in µg/L

| SITE WATER | 2114 | | 695 | | | | | | | |
|------------------------|-------|------|------|------|-------|------|------|------|-------|----------------------------|
| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | r ² (if < 0.95) |
| 0 | 740 | 205 | | | 579 | n.d. | | | 538 | 0.98 |
| 0.04 | | | n.d. | | | | | | | |
| 0.08 | | | | n.d. | | | | n.d. | | |
| 1 | 1549 | n.d. | n.d. | n.d. | 819 | n.d. | n.d. | n.d. | 881 | |
| 3 | 1358 | n.d. | n.d. | n.d. | 870 | n.d. | n.d. | n.d. | | 0.955 |
| 7 | 1302 | n.d. | n.d. | n.d. | 810 | n.d. | n.d. | n.d. | | 0.955 |
| 16 | 2711* | n.d. | n.d. | n.d. | 2492* | n.d. | n.d. | n.d. | | |
| 28 | 5128* | n.d. | n.d. | n.d. | 541 | n.d. | n.d. | n.d. | | |
| Repeat analy at day 36 | 1713 | | | | | | | | | 0.982 |

*Outlier



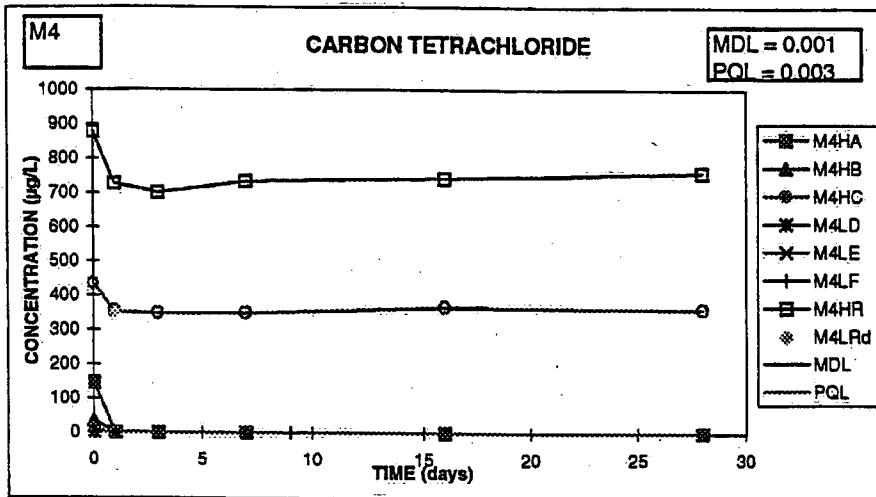
Carbon tetrachloride

Microcosm 4 Sterile control

MAC = 5 µg/L

Result concentration in µg/L

| SITE WATER | | 904 | | | | | 364 | | | |
|------------|------|------|------|------|------|------|------|------|-------|--|
| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | |
| 0 | 877 | 147 | | | 433 | n.d. | | | | |
| 0.04 | | | 36 | | | | | | | |
| 0.08 | | | | 9 | | | | | | |
| 1 | 727 | n.d. | n.d. | n.d. | 353 | n.d. | n.d. | n.d. | 351 | |
| 3 | 702 | n.d. | n.d. | n.d. | 349 | n.d. | n.d. | n.d. | | |
| 7 | 736 | n.d. | n.d. | n.d. | 350 | n.d. | n.d. | n.d. | | |
| 16 | 744 | n.d. | n.d. | n.d. | 368 | n.d. | n.d. | n.d. | | |
| 28 | 761 | n.d. | n.d. | n.d. | 363 | n.d. | n.d. | n.d. | | |



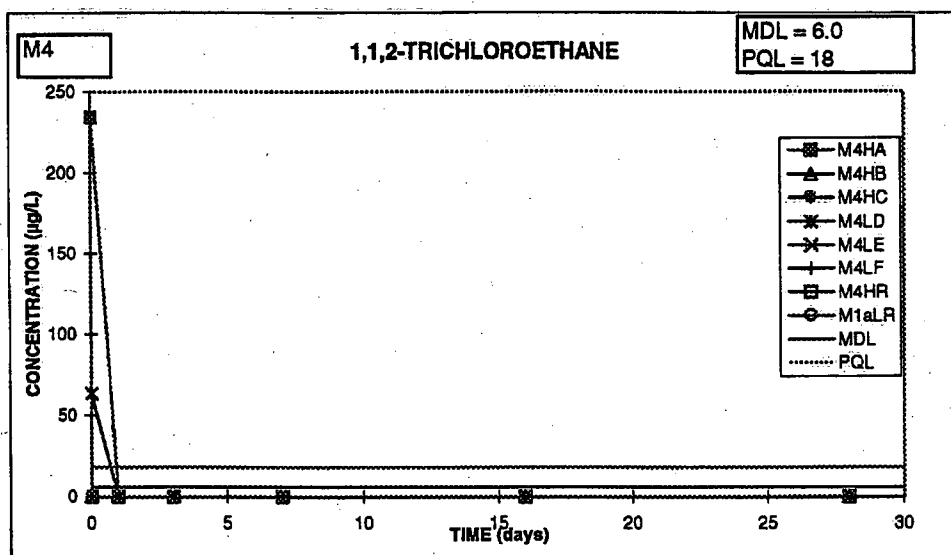
1,1,2-Trichloroethane

Microcosm 4 Sterile control

Result concentration in µg/L

SITE WATER

| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | 234 | | | n.d. | 63 | | | |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 16 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

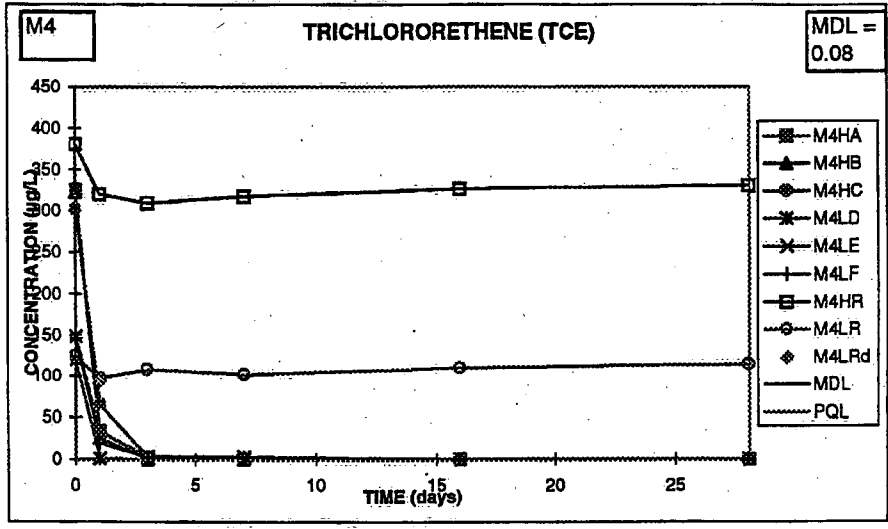


TRICHLOROETHENE (TCE)

Microcosm 4 Sterile control

Result concentration in µg/L

| SITE WATER | 197 | | | | | 86 | | | | |
|------------|------|------|------|------|------|------|------|------|-------|---------------------|
| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | r^2 (if not 0.99) |
| 0 | 381 | 326 | | | 124 | 120 | | | | |
| 0.04 | | | 322 | | | | 148 | | | |
| 0.08 | | | | 302 | | | | 146 | | |
| 1 | 320 | 34 | 25 | 65 | 97 | 1 | 33 | 19 | 95 | 0.971 |
| 3 | 309 | n.d. | n.d. | 2J | 108 | n.d. | 3 | 2j | | 0.969 |
| 7 | 318 | n.d. | n.d. | n.d. | 102 | n.d. | 3 | 2j | | |
| 16 | 327 | n.d. | n.d. | n.d. | 110 | n.d. | n.d. | n.d. | | |
| 28 | 331 | n.d. | n.d. | n.d. | 114 | n.d. | n.d. | n.d. | | |



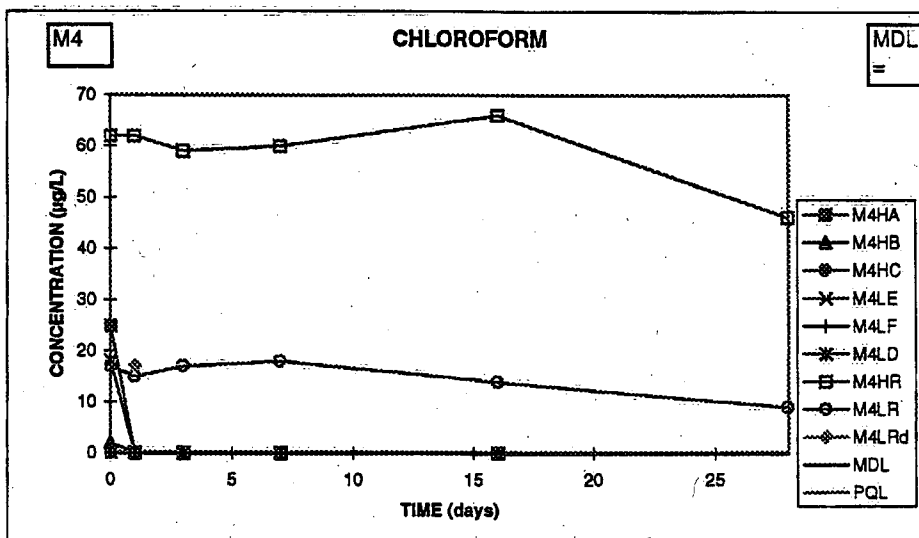
CHLOROFORM

Microcosm 4 Sterile control

Result concentration in µg/L

SITE WATER 71 20

| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | (if < 0.99) |
|------|------|------|------|------|------|------|------|------|-------|-------------|
| 0 | 62 | 25 | | | 17 | 18 | | | | 0.971 |
| 0.04 | | | 2 | | | | n.d. | | | |
| 0.08 | | | | n.d. | | | | | n.d. | |
| 1 | 62 | n.d. | n.d. | n.d. | 15 | n.d. | n.d. | n.d. | n.d. | 17 0.965 |
| 3 | 59 | n.d. | n.d. | n.d. | 17 | n.d. | n.d. | n.d. | n.d. | 0.965 |
| 7 | 60 | n.d. | n.d. | n.d. | 18 | n.d. | n.d. | n.d. | n.d. | |
| 16 | 66 | n.d. | n.d. | n.d. | 14 | n.d. | n.d. | n.d. | n.d. | |
| 28 | 46 | n.d. | n.d. | n.d. | 9 | n.d. | n.d. | n.d. | n.d. | 0.983 |



cis-DICHLOROETHENE

Microcosm 4 Sterile control

MAC = 70 µg/L

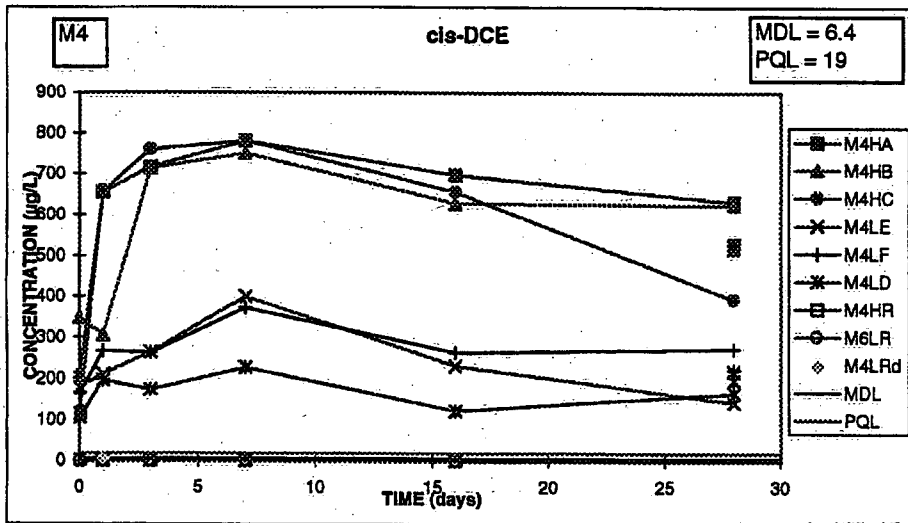
Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | P (t < 0.99) |
|-------------|------|------|------|------|------|------|------|------|-------|--------------|
| 0 | n.d. | 119 | | | n.d. | 107 | | | | |
| 0.04 | | | 348 | | | | 181 | | | |
| 0.08 | | | | 207 | | | | 165 | | |
| 1 | n.d. | 655 | 307 | 658 | n.d. | 197 | 212 | 268 | n.d. | 0.98 |
| 3 | n.d. | 716 | 714 | 759 | n.d. | 174 | 265 | 263 | | |
| 7 | n.d. | 782 | 750 | 782 | n.d. | 228 | 400 | 373 | | 0.978 |
| 16 | n.d. | 698 | 629 | 656 | n.d. | 124 | 232 | 265 | | |
| 28 | n.d. | 632 | 625 | 394 | n.d. | 167 | 144 | 274 | | |
| FID results | | 530 | 519 | 527 | | 197 | 219 | 224 | | |

* suspect poor injection on ECD - results calculated by FID are more consistent and shown for comparison



Trans-DICHLOROETHENE

Microcosm 4 Sterile control

MAC = 100 µg/L

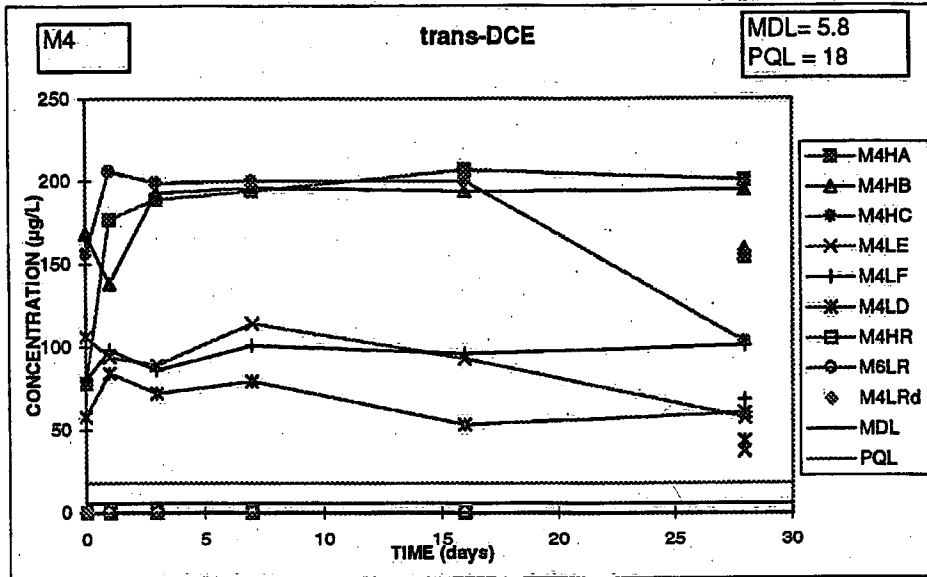
Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | r ² (if < 0.99) |
|------|------|------|------|------|------|------|------|------|-------|----------------------------|
| 0 | n.d. | 78 | | | n.d. | 58 | | | n.d. | |
| 0.04 | | | 168 | | | | 106 | | | |
| 0.08 | | | | 156 | | | | 80 | | |
| 1 | n.d. | 177 | 138 | 206 | n.d. | 84 | 94 | 98 | n.d. | |
| 3 | n.d. | 189 | 193 | 199 | n.d. | 72 | 89 | 86 | | |
| 7 | n.d. | 194 | 196 | 200 | n.d. | 79 | 114 | 101 | | |
| 16 | n.d. | 207 | 194 | 200 | n.d. | 53 | 93 | 96 | | 0.973 |
| 28 | n.d. | 201 | 195 | 103 | n.d. | 60 | 57 | 101 | | |
| FID | | 154 | 159 | 155 | | 43 | 37 | 68 | | |

* suspect poor injection on ECD - results calculated by FID are more consistent and shown for comparison



VINYL CHLORIDE (VC)

Microcosm 4 Sterile control

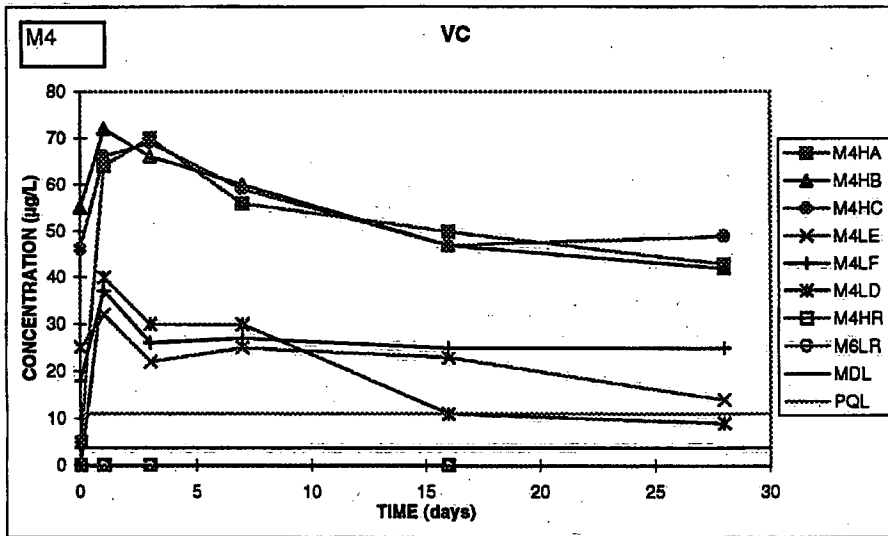
PQL = 11 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

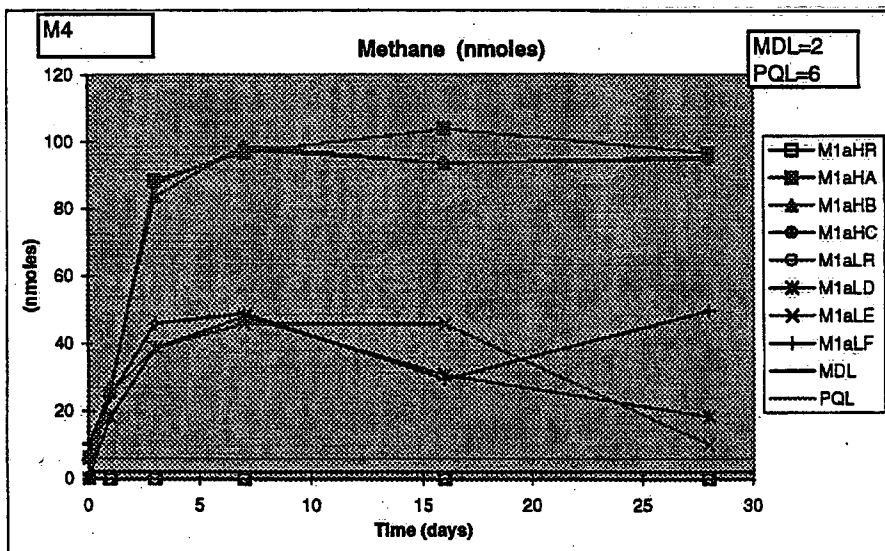
| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | 5j | | | n.d. | n.d. | | | n.d. |
| 0.04 | | | 55 | | | | 25 | | |
| 0.08 | | | | 46 | | | | 18 | |
| 1 | n.d. | 64 | 72 | 66 | n.d. | 40 | 32 | 37 | n.d. |
| 3 | n.d. | 70 | 66 | 69 | n.d. | 30 | 22 | 26 | |
| 7 | n.d. | 56 | 60 | 59 | n.d. | 30 | 25 | 27 | |
| 16 | n.d. | 50 | 47 | 47 | n.d. | 11 | 23 | 25 | |
| 28 | n.d. | 43 | 42 | 49 | n.d. | 9j | 14 | 25 | |



METHANE

Microcosm 4 Sterile control

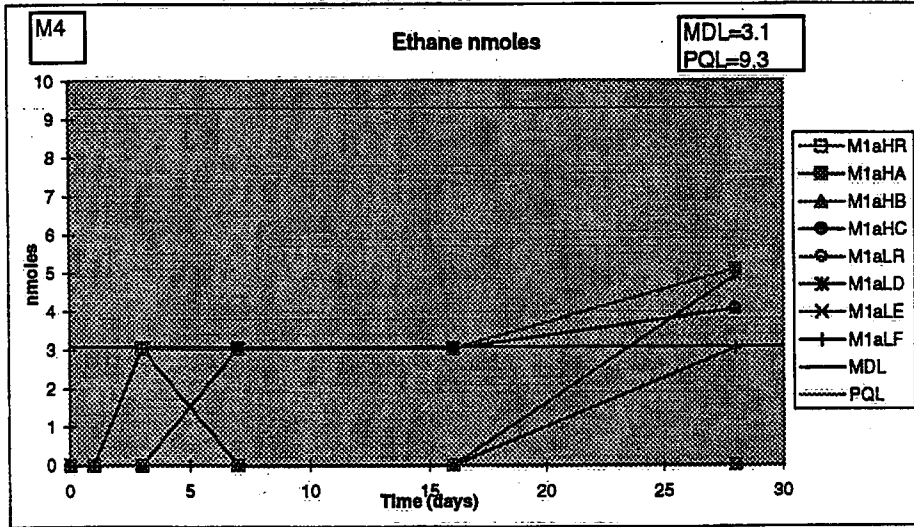
| Time | <PQL | | RESULTS in ppmv | | | | | | | <PQL | |
|------|------|------|-----------------|------|------|------|------|------|-------|------|--|
| | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd | | |
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | | | |
| 0.04 | | | 7 | | | | 6 | | | | |
| 0.08 | | | | 6 | | | | 10 | | | |
| 1 | n.d. | 25 | 27 | 25 | n.d. | 18 | 25 | 24 | n.d. | | |
| 3 | n.d. | 87 | 82 | 86 | n.d. | 38 | 38 | 45 | | | |
| 7 | n.d. | 95 | 97 | 96 | n.d. | 47 | 45 | 48 | | | |
| 16 | n.d. | 102 | 92 | 92 | n.d. | 25 | 45 | 29 | | | |
| 28 | n.d. | 95 | 94 | 93 | n.d. | 15 | 10 | 49 | | | |



ETHANE

Microcosm 4 Sterile control

| Time | <PQL | | Results in ppmv | | | | | <PQL | |
|------|------|------|-----------------|------|------|------|------|------|-------|
| | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd |
| 0 | n.d. | n.d. | | | | | | | |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | 3j | n.d. | n.d. | n.d. | n.d. | n.d. | 3j | |
| 7 | n.d. | 3j | 3j | 3j | n.d. | n.d. | n.d. | n.d. | |
| 16 | n.d. | 5j | 4j | 4j | n.d. | 4j | n.d. | 3j | |
| 28 | n.d. | 6j | 6j | 5j | n.d. | n.d. | n.d. | 3j | |



ETHENE

Microcosm 4 Sterile control

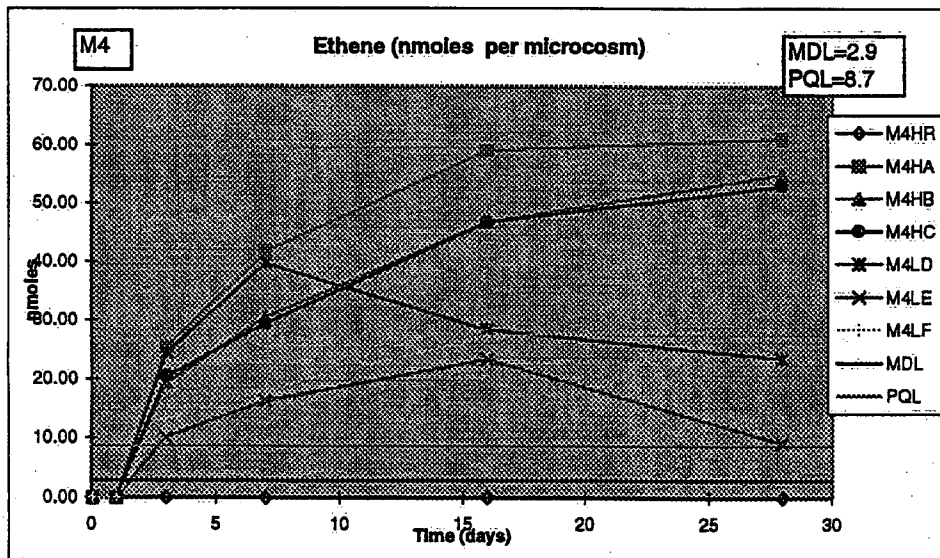
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M4HR | M4HA | M4HB | M4HC | M4LR | M4LD | M4LE | M4LF | M4LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | 3j | 3j | 3j | n.d. | 7j | 5j | 4j | n.d. |
| 3 | n.d. | 25 | 19 | 20 | n.d. | 24 | 10 | 13 | |
| 7 | n.d. | 41 | 30 | 29 | n.d. | 39 | 16 | 18 | |
| 16 | n.d. | 58 | 46 | 46 | n.d. | 23 | 23 | 27 | |
| 28 | n.d. | 60 | 54 | 52 | n.d. | 19 | 9 | 28 | |
| 30 | | | | | | | | | |



Citrate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|-------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | M4HB | M4HC |
| 0 | 7532 | 7244 | 7486 | 7554 | 6648 | 7031 |
| 1 | 7224 | 7439 | 7539 | 7269 | 7684 | 6932 |
| 3 | 7835 | 8213 | 8171 | 7885 | 7488 | 8014 |
| 7 | 7644 | 7472 | 7178 | 6927 | 6976 | 7331 |
| 16 | 8092 | 9020 | 8754 | 8504 | 8702 | 8477 |
| 28 | 9998 | 9828 | 10108 | 9813 | 9407 | 9701 |

Glucose

| day | conc. (mM) | | | | | |
|-----|------------|------|------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | M4HB | M4HC |
| 0 | 16.8 | 17.5 | 16.5 | 17.3 | 17.6 | 15.5 |
| 1 | 18.4 | 18.2 | 19.3 | 18.1 | 17.6 | 17.5 |
| 3 | 18.1 | 18.0 | 18.0 | 16.8 | 16.9 | 16.9 |
| 7 | 11.9 | 17.9 | 18.0 | 16.1 | 16.5 | 17.0 |
| 16 | 0.0 | 14.8 | 15.7 | 15.3 | 14.2 | 14.0 |
| 28 | 0 | 0 | 15.9 | 15.1 | 13.7 | 14.4 |

Volatile Fatty Acids - M4

Lactate

| day | | | conc. (mg/L) | | M4HB | M4HC |
|-----|------|------|--------------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 18 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

Formate

| day | | | conc. (mg/L) | | M4HB | M4HC |
|-----|------|------|--------------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | | |
| 0 | 14 | 13 | 12 | 13 | 12 | 12 |
| 1 | 20 | 20 | 19 | 19 | 20 | 19 |
| 3 | 4j | 6j | 6j | 4j | 5j | 4j |
| 7 | 427 | 17 | 17 | 15 | 17 | 16 |
| 18 | 1477 | 20 | 21 | 18 | 20 | 21 |
| 28 | 2248 | 1883 | 19 | 13 | 16 | 16 |

Acetate

| day | | | conc. (mg/L) | | M4HB | M4HC |
|-----|------|------|--------------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | 277 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 18 | 831 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | 1684 | 1411 | n.d. | n.d. | n.d. | n.d. |

Propionate

| day | | | conc. (mg/L) | | M4HB | M4HC |
|-----|------|------|--------------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | | |
| 0 | 348 | 314 | 265 | 277 | 238j | 242 |
| 1 | 368 | 381 | 359 | 359 | 323 | 349 |
| 3 | 342 | 359 | 239 | 307 | 305 | 278 |
| 7 | n.d. | 384 | 322 | 289 | 302 | 306 |
| 18 | 257 | 449 | 397 | 297 | 350 | 344 |
| 28 | 242 | n.d. | n.d. | n.d. | 397 | 353 |

Formate

| day | | | conc. (mg/L) | | M4HB | M4HC |
|-----|------|------|--------------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | | |
| 0 | 14 | 13 | 12 | 13 | 12 | 12 |
| 1 | 20 | 20 | 19 | 19 | 20 | 19 |
| 3 | 4 | 6 | 6 | 4 | 5 | 4 |
| 7 | 427 | 17 | 17 | 15 | 17 | 16 |
| 18 | 1477 | 20 | 21 | 18 | 20 | 21 |
| 28 | 2248 | 1883 | 19 | 13 | 16 | 16 |

Q52C

| day | formate | acetate | propionate |
|-----|---------|----------|------------|
| 0 | 13 | 0 | 308 |
| 1 | 20 | 0 | 368 |
| 3 | 5 | 0 | 313 |
| 7 | 154 | 9 | 353 |
| 18 | 508 | 277 | 368 |
| 28 | 1383 | 1031.667 | 242 |

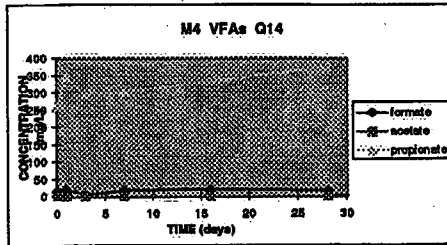
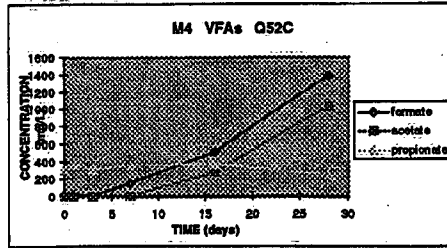
| | |
|------|------|
| 277 | n.d. |
| 831 | n.d. |
| 1684 | 1411 |

Q14

| day | formate | acetate | propionate |
|-----|---------|---------|------------|
| 0 | 12 | 0 | 259 |
| 1 | 20 | 0 | 344 |
| 3 | 4 | 0 | 298 |
| 7 | 16 | 0 | 299 |
| 18 | 19 | 0 | 330 |
| 28 | 15 | 0 | 375 |

Titanium

| day | conc. (mM) | | | | | |
|-----|------------|------|------|------|------|------|
| | M4LD | M4LE | M4LF | M4HA | M4HB | M4HC |
| 0 | 27.7 | 27.8 | 28.2 | 27.7 | 28.8 | 27.3 |



Tetrachloroethane (TeCA)

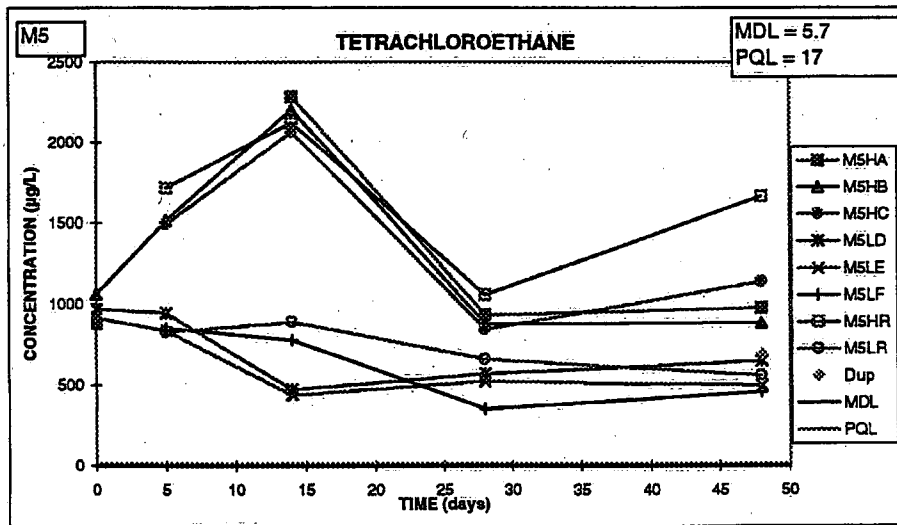
Microcosm 5

Result concentration in µg/L

SITE WATER 2114

695

| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF Dup | |
|------|------|------|------|------|------|------|------|----------|--------------------------|
| 0 | | 885 | 1061 | | | 970 | 913 | | single point calibration |
| 5 | 1716 | | 1514 | 1492 | 830 | 945 | 838 | 852 | |
| 14 | 2124 | 2285 | 2206 | 2065 | 893 | 474 | 437 | 780 | |
| 28 | 1060 | 934 | 878 | 846 | 657 | 569 | 524 | 354 | 0.955 |
| 48 | 1667 | 978 | 884 | 1141 | 560 | 647 | 498 | 458 | 689 0.982 |
| 50 | | | | | | | | | |

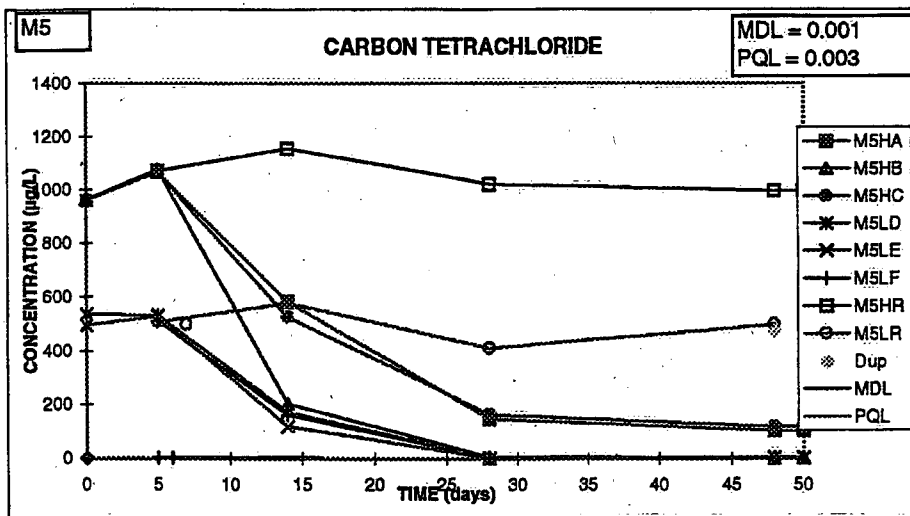


Carbon tetrachloride

Microcosm 5

Result concentration in µg/L

| SITE WATER | 904 | | | | | | | | 364 |
|------------|------|------|------|------|------|------|------|------|-----|
| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF | Dup |
| 0 | | 962 | 963 | | | 535 | 494 | | |
| 5 | 1072 | 1067 | 1075 | 1065 | 507 | 533 | 528 | 512 | |
| 14 | 1155 | 582 | 204 | 528 | 576 | 173 | 119 | 160 | |
| 28 | 1021 | 147 | 4 | 164 | 411 | 3 | n.d. | n.d. | |
| 48 | 997 | 102 | 3 | 116 | 498 | 3 | n.d. | 3 | 474 |
| 50 | | | | | | | | | |



TETRACHLOROETHENE (PERC)

Microcosm 5

Result concentration in µg/L

SITE WATER

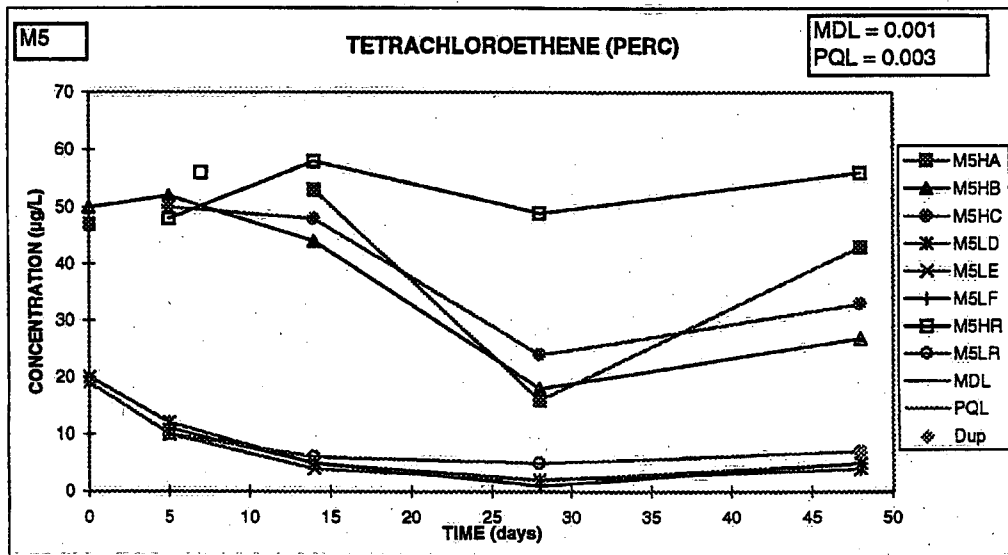
35

6

| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF | Dup | if less than 0.99 |
|------|------|------|------|------|------|------|------|------|-----|----------------------|
| 0 | | 47 | 50 | | | 20 | 19 | | | |
| 5 | 48 | | 52 | 50 | 10 | 12 | 10 | 11 | | 0.923 |
| 14 | 58 | 53 | 44 | 48 | 6 | 5 | 4 | 5 | | C: 0.981; D:0.938 |
| 28 | 49 | 16 | 18 | 24 | 5 | 2 | 2 | 1 | | B: 0.982; D:0.979 |
| 48 | 56 | 43 | 27 | 33 | 7 | 4 | 5 | 5 | | 7: B: 0.924; D:0.935 |
| 50 | | | | | | | | | | |

predicted left after gas removed

| | | | | | | | |
|----|----|----|----|--|----|---|---|
| 14 | | 22 | 33 | | 8 | 4 | 7 |
| 28 | 33 | 15 | 23 | | -2 | 1 | 1 |



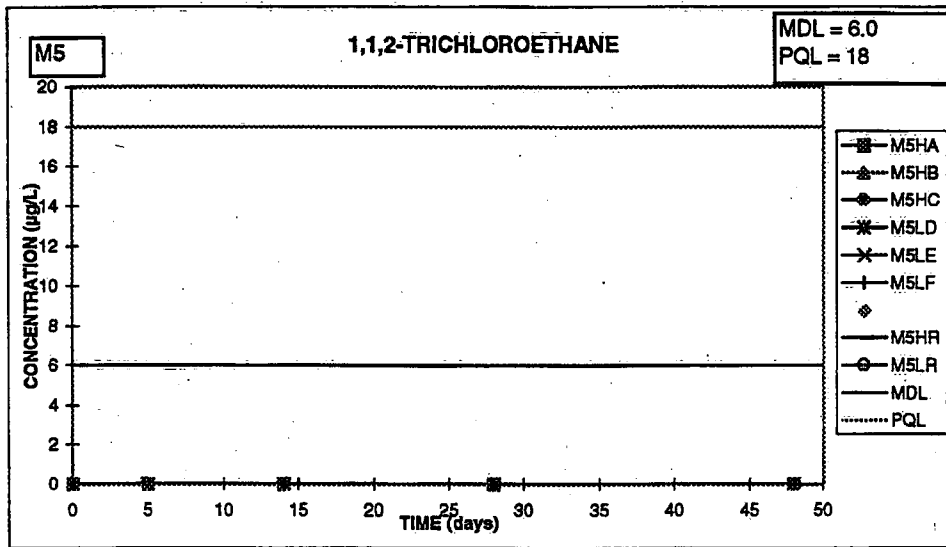
1,1,2-Trichloroethane

Microcosm 5

Result concentration in µg/L

SITE WATER

| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF |
|------|------|------|------|------|------|------|------|------|
| 0 | | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 5 | n.d. | | 12j | 8j | j | 17j | n.d. | 7j |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 48 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 50 | | | | | | | | |



TRICHLOROETHENE (TCE)

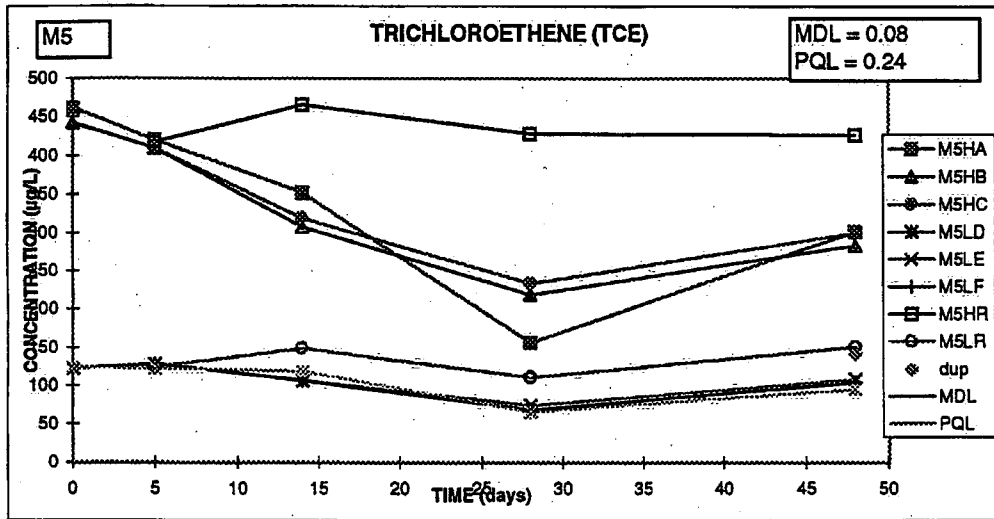
Microcosm 5

Result concentration in µg/L

| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF | dup |
|------|------|------|------|------|------|------|------|------|-----|
| 0 | | 462 | 442 | | | 124 | 122 | | |
| 5 | 418 | 420 | 410 | 410 | 125 | 123 | 129 | 129 | |
| 14 | 466 | 353 | 308 | 319 | 149 | 119 | 107 | 107 | |
| 28 | 428 | 156 | 219 | 234 | 111 | 66 | 74 | 68 | |
| 48 | 427 | 301 | 283 | 300 | 151 | 95 | 108 | 104 | 142 |
| 50 | | | | | | | | | |

Predicted left, based on venting

| | | | | | | |
|----|-----|-----|-----|----|----|-----|
| 14 | 377 | 279 | 333 | 99 | 86 | 103 |
| 28 | 281 | 195 | 229 | 28 | 70 | 59 |



VINYL CHLORIDE (VC)

Microcosm 5

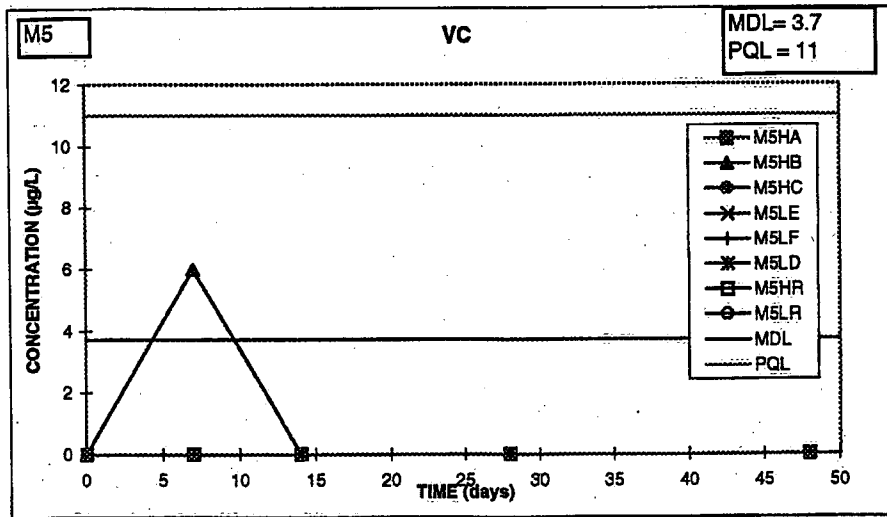
Result concentration in µg/L

SITE WATER

<PQL

<PQL

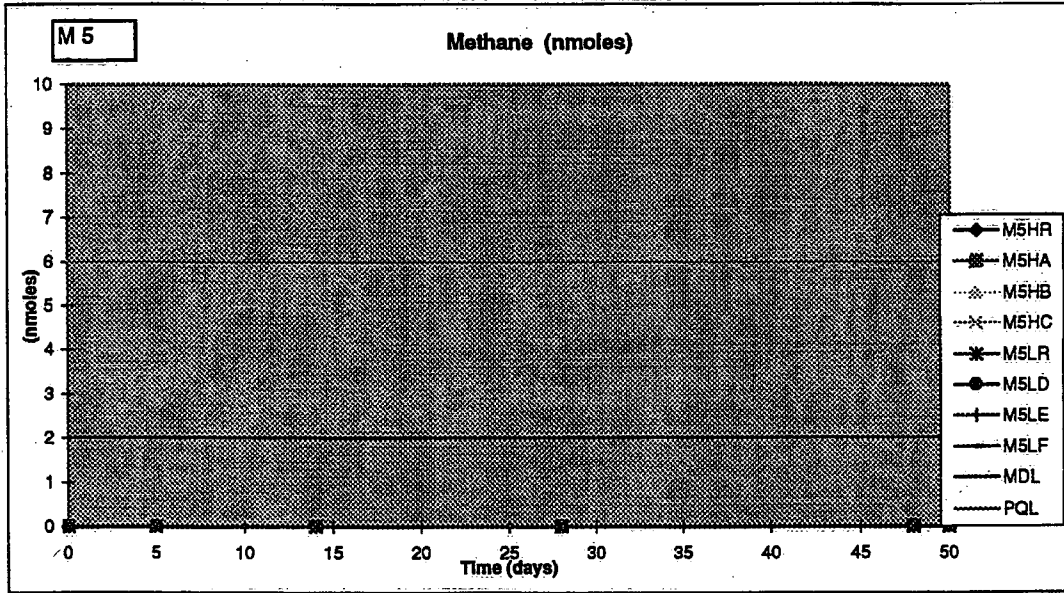
| Time | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF |
|------|------|------|------|------|------|------|------|-------------------------------|
| 0 | | n.d. | n.d. | | | n.d. | n.d. | |
| 7 | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | n.d. | n.d. single point calibration |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 48 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 50 | | | | | | | | |



METHANE

Microcosm 5

| Time | <PQL | | | | <PQL | | | | Dup |
|------|------|------|------|------|------|------|------|------|------|
| | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 5 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 48 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 50 | | | | | | | | | |



ETHENE

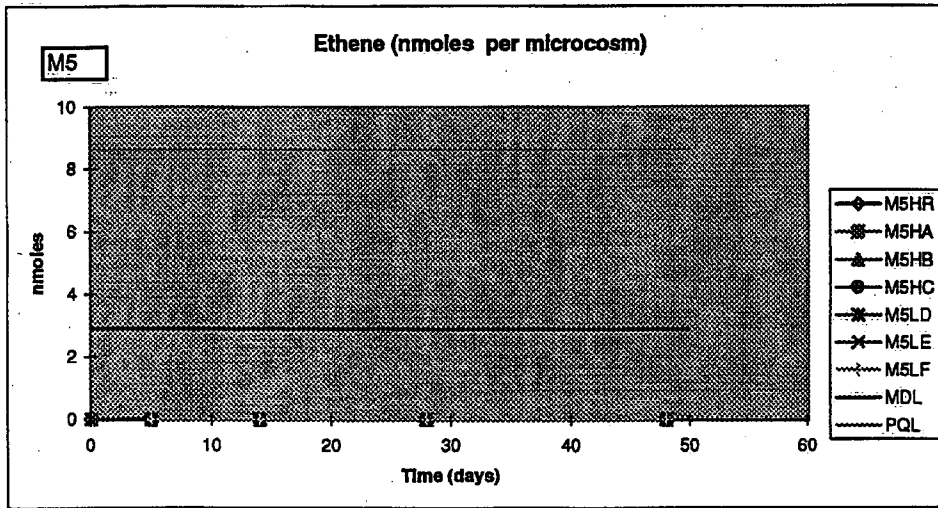
Microcosm 5

SITE WATER

<PQL

<PQL

| Time | ppm/v | | | | | | | |
|------|-------|------|------|------|------|------|------|------|
| | M5HR | M5HA | M5HB | M5HC | M5LR | M5LD | M5LE | M5LF |
| 0 | | n.d. | n.d. | | | n.d. | n.d. | |
| 5 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 48 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 50 | | | | | | | | |



Volatile Fatty Acids-M5

Citrate None Added
Lactate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M5LD | M5LE | M5LF | M5HA | M5HB | M5HC |
| 8 | n.d. | n.d. | n.d. | n.d. | 20j | 15j |
| 14 | 178 | 275 | 163 | 52 | 407 | 69 |
| 28 | 411 | 1498 | 1390 | 1801 | 1175 | 1718 |
| 48 | 607 | 2036 | 2340 | 2842 | 2169 | 2220 |

Formate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M5LD | M5LE | M5LF | M5HA | M5HB | M5HC |
| 8 | 15 | 10j | 13 | 10j | 11j | 13 |
| 14 | 57 | 41 | 38 | 40 | 43 | 44 |
| 28 | 78 | 61 | 62 | 56 | 70 | 99 |
| 48 | 101 | 86 | 76 | 87 | 86 | 79 |

Acetate

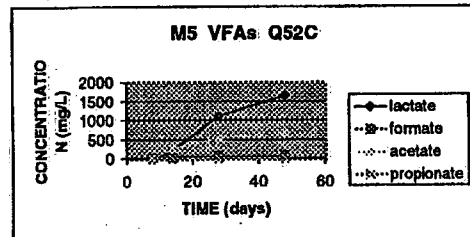
| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M5LD | M5LE | M5LF | M5HA | M5HB | M5HC |
| 8 | n.d. | n.d. | n.d. | 41j | 67 | 53 |
| 14 | 221 | 353 | 235 | 192 | 348 | 174 |
| 28 | 589 | 601 | 443 | 372 | 432 | 323 |
| 48 | 939 | 493 | 558 | 412 | 502 | 366 |

Propionate

| day | conc. (mg/L) | | | | | |
|-----|--------------|------|------|------|------|------|
| | M5LD | M5LE | M5LF | M5HA | M5HB | M5HC |
| 8 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 48 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

This data only for the purposes of graphing
Q52C

| day | lactate | formate | acetate | propionate |
|-----|---------|---------|---------|------------|
| 8 | 0 | 13 | 0 | 0 |
| 14 | 205 | 45 | 270 | 0 |
| 28 | 1100 | 67 | 538 | 0 |
| 48 | 1661 | 88 | 664 | 0 |



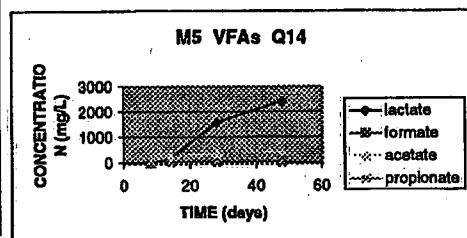
Glucose

| day | conc. (mM) | | | | | |
|-----|------------|------|------|------|------|------|
| | M5LD | M5LE | M5LF | M5HA | M5HB | M5HC |
| 0 | 12.3 | 11.3 | 12.4 | 13.4 | 12.5 | 13.5 |
| 5 | 12.6 | 10.4 | 13.3 | 12.5 | 11 | 13.4 |
| 8 | 12.9 | 12.1 | 12.8 | 12.4 | 10.9 | 12.3 |
| 14 | 10 | 7.6 | 11.6 | 9.9 | 7.8 | 10.9 |
| 15 | 19.3 | 19.6 | 24.6 | 23.8 | 24.7 | 22.7 |
| 20 | 15.4 | 17.0 | 23.2 | 22.4 | 19.0 | 21.9 |
| 28 | 20.8 | 19.4 | 25.2 | 27.0 | 28.0 | 25.2 |
| 35 | 19.2 | 19.5 | 26.5 | 26.7 | 25.3 | 24.1 |
| 42 | 19.3 | 18.8 | 22.4 | 24.4 | 25.6 | 21.9 |
| 48 | 19.1 | 18.4 | 24.0 | 25.7 | 25.7 | 21.4 |

Titanium
None added

Q14

| day | lactate | formate | acetate | propionate |
|-----|---------|---------|---------|------------|
| 8 | 12 | 11 | 54 | 0 |
| 14 | 176 | 42 | 238 | 0 |
| 28 | 1565 | 75 | 378 | 0 |
| 48 | 2411 | 84 | 427 | 0 |

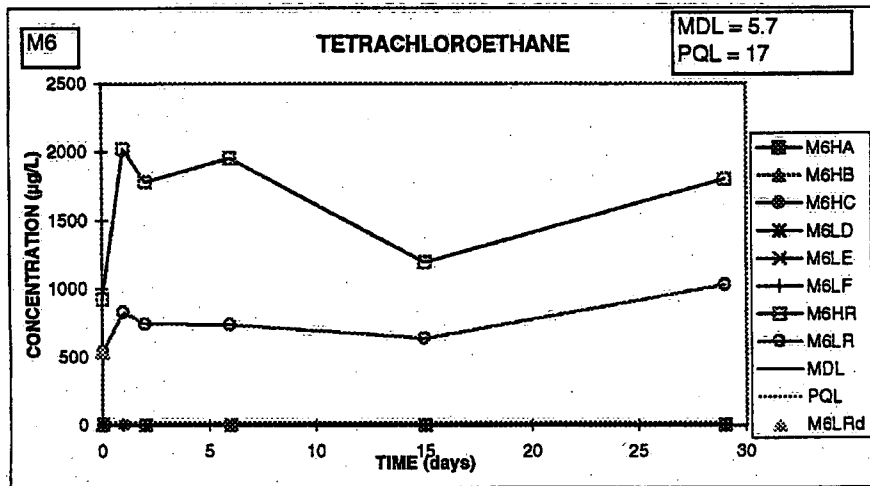


Tetrachloroethane (TeCA)

Microcosm 6

Result concentration in µg/L

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd | r ² (if < 0.99) |
|------|------|------|------|------|------|------|------|------|-------|----------------------------|
| 0 | 929 | 10j | | | 540 | 3j | | | 538 | 0.959 |
| 0.04 | | | n.d. | | | | n.d. | | | |
| 0.08 | | | | n.d. | | | | n.d. | | |
| 1 | 2021 | | | | 831 | | | | | 0.959 |
| 2 | 1778 | n.d. | n.d. | n.d. | 746 | n.d. | n.d. | n.d. | | 0.959 |
| 6 | 1955 | n.d. | n.d. | n.d. | 738 | | | | | |
| 14 | 1194 | n.d. | n.d. | n.d. | 639 | n.d. | n.d. | n.d. | | 0.98 |
| 29 | 1802 | n.d. | n.d. | n.d. | 1030 | n.d. | n.d. | n.d. | | B: 0.981; C: 0.972 |



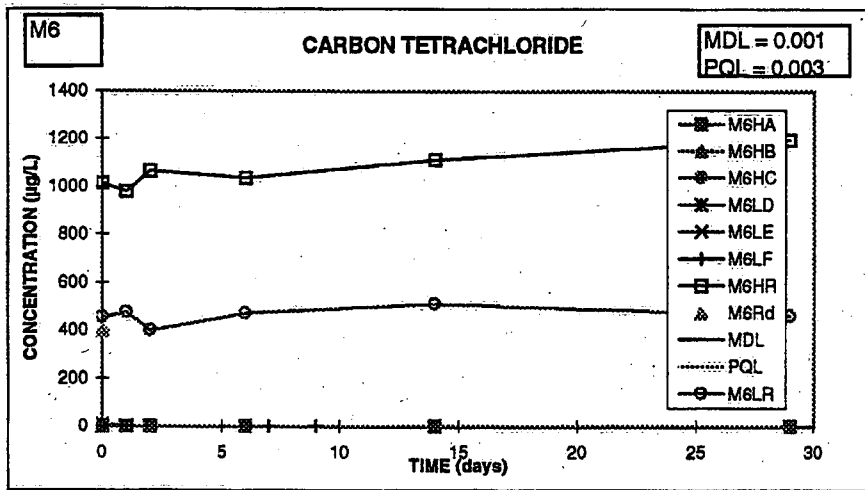
Carbon tetrachloride

Microcosm 6

MAC = 5 µg/L

Result concentration in µg/L

| SITE WATER | | 904 | | | | 364 | | | |
|------------|------|------|------|-------|------|-------|-------|-------|-------|
| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
| 0 | 1014 | 0.3j | | | 454 | 7 | | | 398 |
| 0.04 | | | n.d. | | | | 3j | | |
| 0.08 | | | | 0.05j | | | | 0.3j | |
| 1 | 980 | n.d. | n.d. | n.d. | 474 | n.d. | n.d. | n.d. | n.d. |
| 2 | 1065 | n.d. | n.d. | n.d. | 397 | 0.05j | 0.05j | 0.05j | n.d. |
| 6 | 1035 | n.d. | n.d. | n.d. | 472 | n.d. | n.d. | n.d. | n.d. |
| 14 | 1113 | n.d. | n.d. | n.d. | 510 | n.d. | n.d. | n.d. | n.d. |
| 29 | 1197 | n.d. | n.d. | n.d. | 460 | n.d. | n.d. | n.d. | n.d. |

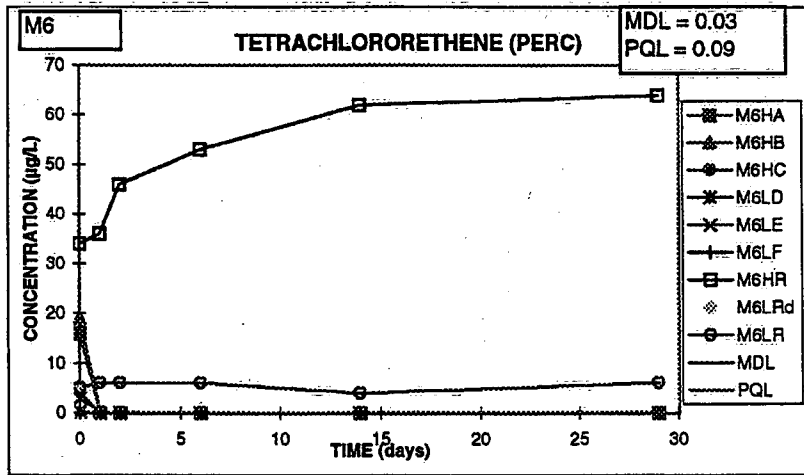


TETRACHLOROETHENE (PERC)

Microcosm 6

Result concentration in µg/L

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd | r ² if less than 0.99 |
|------|------|------|------|------|------|------|------|------|-------|----------------------------------|
| 0 | 34 | 16 | | | 5 | 3 | | | 5 | 0.977 |
| 0.04 | | | 19 | | | | 0.3 | | | 0.884 |
| 0.08 | | | | 4 | | | | | | |
| 1 | 36 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | n.d. | |
| 2 | 45 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | n.d. | 0.923 |
| 6 | 53 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | n.d. | 0.938 |
| 14 | 63 | n.d. | n.d. | n.d. | 4 | n.d. | n.d. | n.d. | n.d. | C:0.901; D:0.939 |
| 29 | 64 | n.d. | n.d. | n.d. | 6 | n.d. | n.d. | n.d. | n.d. | |



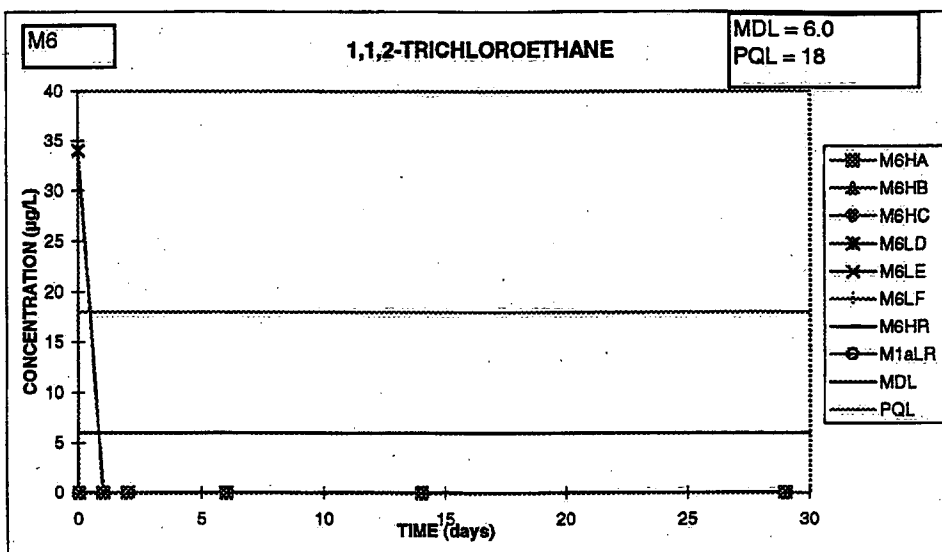
1,1,2-Trichloroethane

Microcosm 6

Result concentration in µg/L

SITE WATER

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | | | n.d. | 34 | | | n.d. |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2 | 7j | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 6 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 29 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |



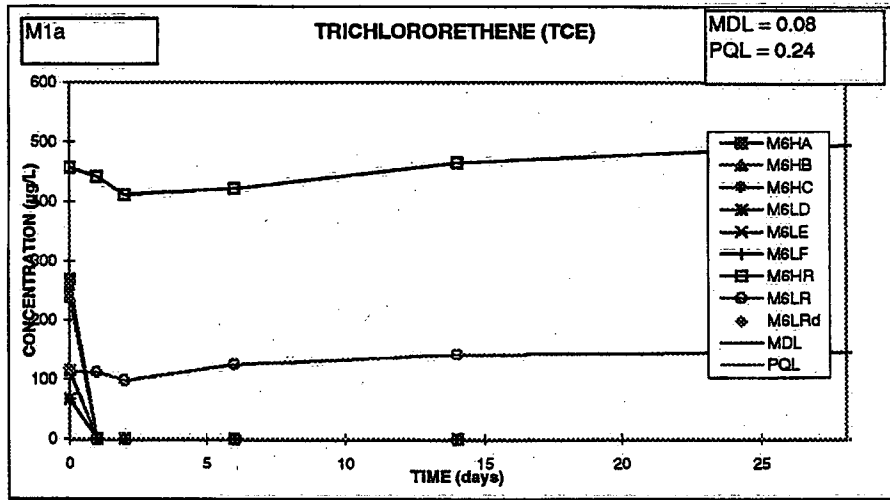
TRICHLOROETHENE (TCE)

Microcosm 6

Result concentration in µg/L

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | 457 | 269 | | | 113 | 115 | | | 119 |
| 0.04 | | | 240 | | | | 68 | | |
| 0.08 | | | | 250 | | | | 68 | |
| 1 | 442 | n.d. | n.d. | n.d. | 112 | n.d. | n.d. | n.d. | n.d. |
| 2 | 411 | n.d. | n.d. | n.d. | 98 | n.d. | n.d. | n.d. | n.d. |
| 6 | 422 | n.d. | n.d. | n.d. | 126 | n.d. | n.d. | n.d. | n.d. |
| 14 | 465 | n.d. | n.d. | n.d. | 142 | n.d. | n.d. | n.d. | n.d. |
| 29 | 497 | n.d. | n.d. | n.d. | 146 | n.d. | n.d. | n.d. | n.d. |

(If not 0.99)

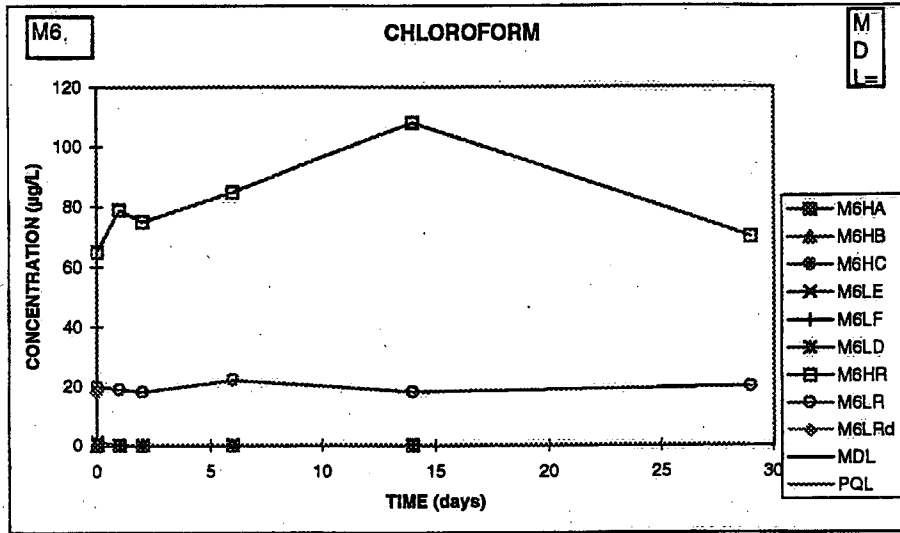


CHLOROFORM

Microcosm 6

Result concentration in µg/L

| SITE WATER | 71 | | | | | | | | | 20 |
|------------|------|------|------|------|------|------|------|------|-------|------------|
| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd | (f = 0.99) |
| 0 | 65 | n.d. | | | 20 | 1 | | | 18 | |
| 0.04 | | | n.d. | | | | n.d. | | | |
| 0.08 | | | | n.d. | | | | n.d. | | |
| 1 | 79 | n.d. | n.d. | n.d. | 19 | n.d. | n.d. | n.d. | | 0.963 |
| 2 | 75 | n.d. | n.d. | n.d. | 18 | n.d. | n.d. | n.d. | | 0.963 |
| 6 | 85 | n.d. | n.d. | n.d. | 22 | n.d. | n.d. | n.d. | | |
| 14 | 108 | n.d. | n.d. | n.d. | 18 | n.d. | n.d. | n.d. | | 0.975 |
| 29 | 70 | n.d. | n.d. | n.d. | 20 | n.d. | n.d. | n.d. | | 0.943 |



cis-DICHLOROETHENE

Microcosm 6

MAC = 70 µg/L

Result concentration in µg/L

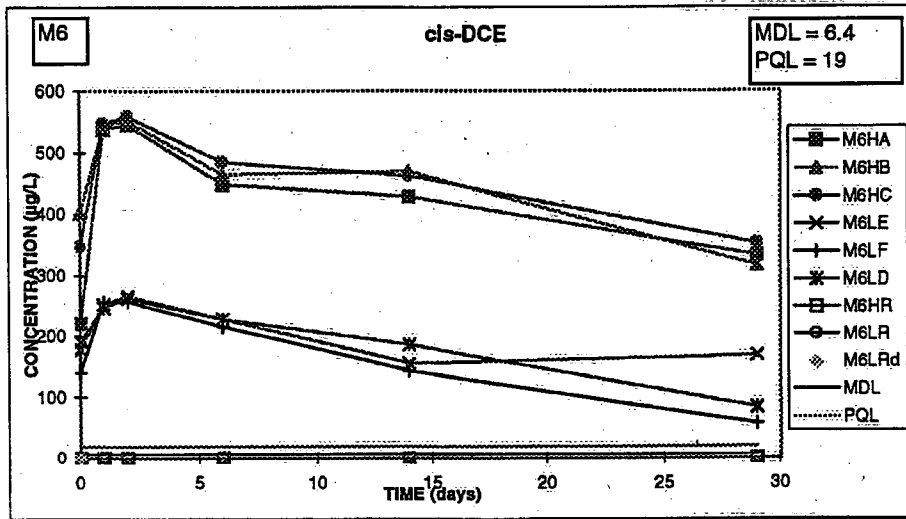
SITE WATER <PQL

<PQL

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd | (if < 0.99) |
|------|------|------|------|------|------|------|------|------|-------|-------------|
| 0 | n.d. | 221 | | | n.d. | 191 | | | | |
| 0.04 | | | 401 | | | | 177 | | | |
| 0.08 | | | | 347 | | | | 139 | | |
| 1 | n.d. | 537 | 544 | 547 | n.d. | 246 | 252 | 255 | | |
| 2 | n.d. | 546 | 552 | 559 | n.d. | 262 | 266 | 256 | | |
| 6 | n.d. | 448 | 465 | 484 | n.d. | 228 | 228 | 215 | | |
| 14 | n.d. | 429 | 470 | 463 | n.d. | 186 | 155 | 143 | | 0.96 |
| 29 | n.d. | 332 | 316 | 352 | n.d. | 83 | 167 | 56 | | |

Predicted concentration

| | | | | | | | | | |
|--------------------|-----|-----|-----|-----|-----|-----|-----|-----|--|
| remaining at day 6 | 403 | 417 | 439 | | 205 | 205 | 193 | | |
| day 14 | | 414 | 449 | 435 | | 178 | 150 | 137 | |
| DAY 29 | | 291 | 280 | 349 | | 72 | 163 | 55 | |



Trans-DICHLOROETHENE

Microcosm 6

MAC = 100 µg/L

Result concentration in µg/L

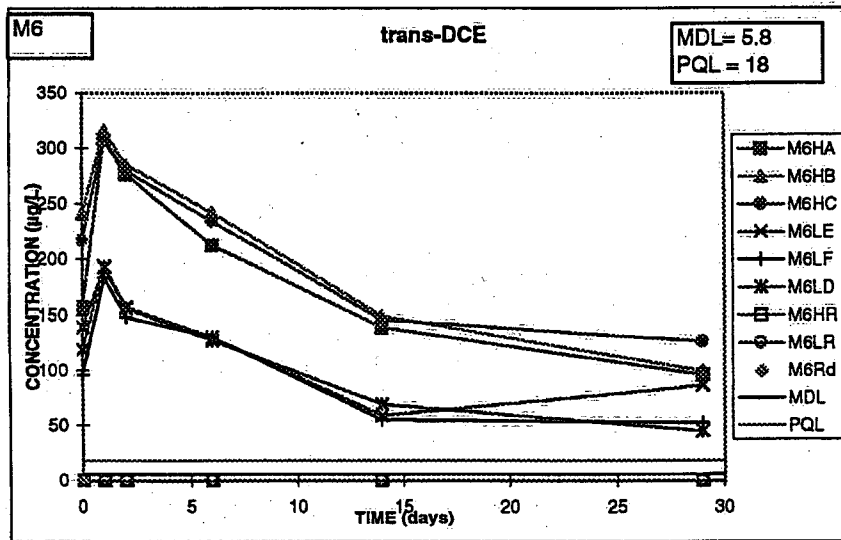
SITE WATER <PQL

<PQL

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd | (#-0.99) |
|------|------|------|------|------|------|------|------|------|-------|----------|
| 0 | n.d. | 157 | | | n.d. | 139 | | | n.d. | |
| 0.04 | | | 241 | | | | 119 | | | |
| 0.08 | | | | 217 | | | | 96 | | |
| 1 | n.d. | 308 | 316 | 308 | n.d. | 193 | 194 | 184 | | |
| 2 | n.d. | 277 | 286 | 280 | n.d. | 156 | 157 | 148 | | 0.98 |
| 6 | n.d. | 213 | 242 | 234 | n.d. | 127 | 130 | 130 | | |
| 14 | n.d. | 139 | 149 | 146 | n.d. | 69 | 59 | 55 | | |
| 29 | n.d. | 96 | 99 | 126 | n.d. | 45 | 86 | 52 | | 0.906 |

Predicted value based on amount of gas removed

| remaining at day | 6 | 14 | 29 |
|------------------|-----|-----|-----|
| 170 | 192 | 190 | 101 |
| 129 | 136 | 128 | 63 |
| 72 | 76 | 124 | 33 |
| | | | 103 |
| | | | 55 |
| | | | 49 |



VINYL CHLORIDE (VC)

Microcosm 6

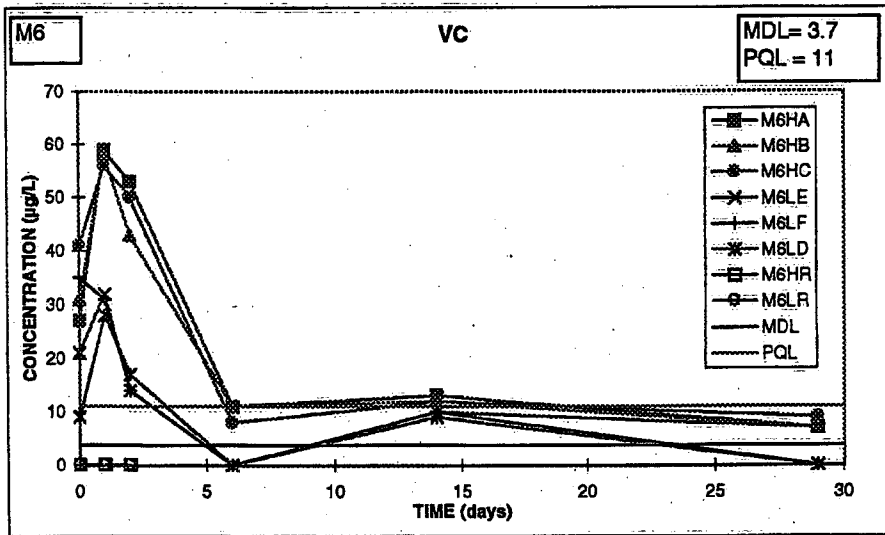
PQL = 11 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | 27 | | | n.d. | 9j | | | n.d. |
| 0.04 | | | 31 | | | | 21 | | |
| 0.08 | | | | 41 | | | | 35 | |
| 1 | n.d. | 59 | 58 | 56 | n.d. | 28 | 32 | 31 | |
| 2 | n.d. | 53 | 43 | 50 | n.d. | 17 | 14 | 14 | |
| 6 | n.d. | 11 | 11 | 8 | n.d. | n.d. | n.d. | n.d. | |
| 14 | n.d. | 13 | 12 | 12 | n.d. | 9j | 10j | 10j | |
| 29 | n.d. | 7j | 7j | 9j | n.d. | n.d. | 7j | n.d. | |



ETHANE

Microcosm 6

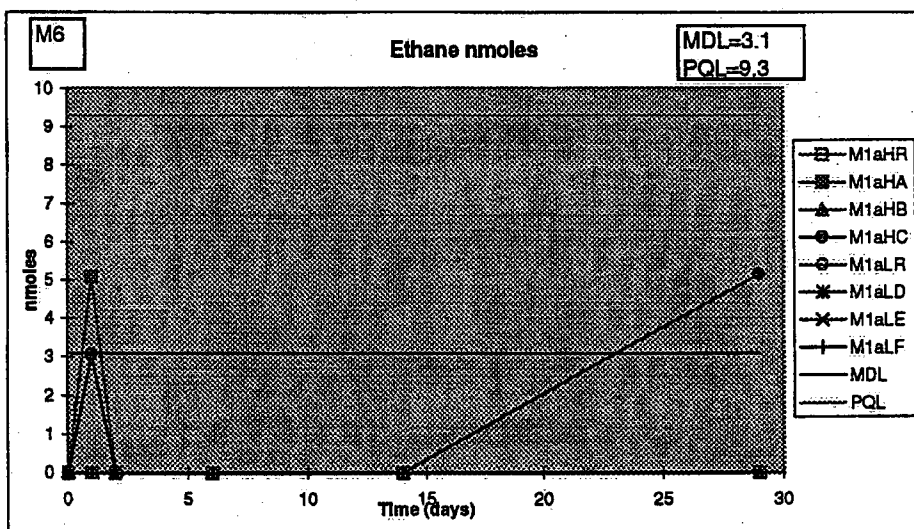
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | 5j | 5j | 3j | n.d. | n.d. | n.d. | n.d. | |
| 2 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 6 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 29 | n.d. | n.d. | n.d. | 4j | n.d. | n.d. | n.d. | n.d. | |



ETHENE

Microcosm 6

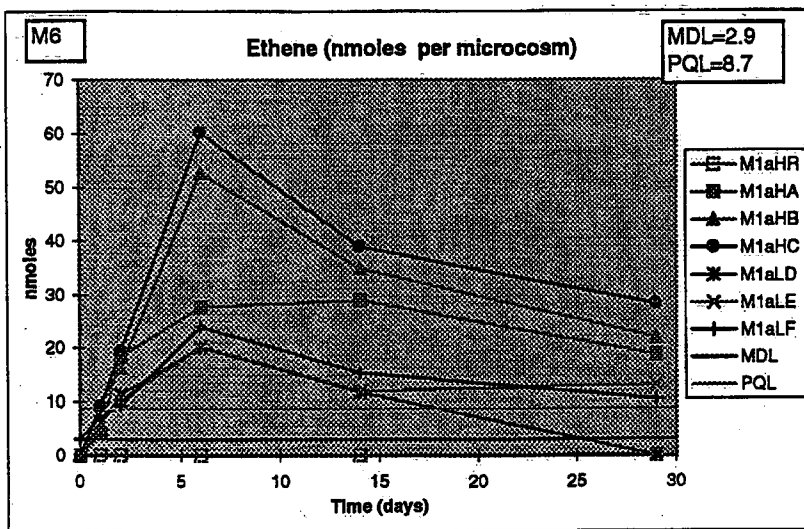
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | n.d. |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | 1 | | | | 3 | |
| 1 | n.d. | 4j | 9 | 9 | n.d. | 6j | 6j | 7j | |
| 2 | n.d. | 18 | 16 | 19 | n.d. | 11 | 10 | 9 | |
| 6 | n.d. | 7j | 13 | 16 | n.d. | 5j | 5j | 6j | |
| 14 | n.d. | 14 | 15 | 14 | n.d. | 5j | 6j | 7j | |
| 29 | n.d. | 4j | 5j | 22 | n.d. | n.d. | 8j | 6j | |



ACETYLENE

Microcosm 6

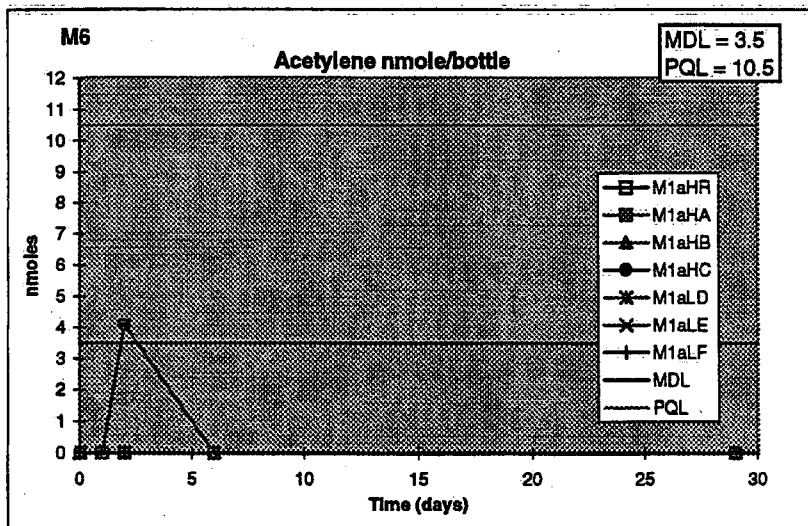
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M6HR | M6HA | M6HB | M6HC | M6LR | M6LD | M6LE | M6LF | M6LRd |
|------|------|------|------|------|------|------|------|------|-------|
| 0 | n.d. | n.d. | | | n.d. | n.d. | | | n.d. |
| 0.04 | | | n.d. | | | | n.d. | | |
| 0.08 | | | | n.d. | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2 | n.d. | n.d. | n.d. | 4j | n.d. | n.d. | n.d. | n.d. | n.d. |
| 6 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 29 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |



Volatile Fatty Acids - M6

Citrate

Glucose

| day | M6LD | | | M6LE | | | M6LF | | | M6HA | | | M6HB | | | M6HC | | |
|-----|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|--|
| | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | |
| 1 | 7179 | 6954 | 6702 | 6739 | 6751 | 6711 | 6719 | 6754 | 6848 | 6639 | 6719 | 6754 | 6848 | 6639 | 6719 | 6754 | 6848 | |
| 2 | 7402 | 6820 | 6773 | 6773 | 7450 | 6419 | 7402 | 6820 | 6773 | 7450 | 6419 | 7402 | 6820 | 6773 | 7450 | 6419 | 7402 | |
| 6 | 8715 | 8302 | 6372 | 6372 | 6378 | 6941 | 8715 | 8302 | 6372 | 6378 | 6941 | 8715 | 8302 | 6372 | 6378 | 6941 | 8715 | |
| 14 | 7334 | 6982 | 6842 | 6842 | 6958 | 6941 | 7334 | 6982 | 6842 | 6958 | 6941 | 7334 | 6982 | 6842 | 6958 | 6941 | 7334 | |
| 29 | 7298 | 5482 | 843 | 7019 | 843 | 6339 | 7298 | 5482 | 843 | 7019 | 843 | 7298 | 5482 | 843 | 7019 | 843 | 7298 | |

| day | M6LD | | | M6LE | | | M6LF | | | M6HA | | | M6HB | | | M6HC | | |
|-----|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|--|
| | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | |
| 1 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | |
| 29 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | |

Titanium

| day | M6LD | M6LE | M6LF | M6HA | M6HB | M6HC |
|-----|------|------|------|------|------|------|
| 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 1 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 |
| 29 | 318 | 32.1 | 31.4 | 31.8 | 32.1 | 31.6 |

Glucose

| day | M6LD | | | M6LE | | | M6LF | | | M6HA | | | M6HB | | | M6HC | | |
|-----|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|--|
| | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | |
| 1 | 18.2 | 21.2 | 18.9 | 18.4 | 20.3 | 21.0 | 18.2 | 21.2 | 18.9 | 18.4 | 20.3 | 21.0 | 18.2 | 21.2 | 18.9 | 18.4 | 20.3 | |
| 2 | 17.8 | 18.4 | 17.7 | 17.7 | 18.3 | 18.2 | 17.8 | 18.4 | 17.7 | 17.7 | 18.3 | 18.2 | 17.8 | 18.4 | 17.7 | 17.7 | 18.3 | |
| 6 | 1.4 | 1.8 | 1.8 | 1.9 | 2.1 | 2.0 | 1.4 | 1.8 | 1.8 | 1.9 | 2.1 | 2.0 | 1.4 | 1.8 | 1.8 | 1.9 | 2.1 | |
| 14 | 1.3 | 1.4 | 1.6 | 1.7 | 2.0 | 1.8 | 1.3 | 1.4 | 1.6 | 1.7 | 2.0 | 1.8 | 1.3 | 1.4 | 1.6 | 1.7 | 2.0 | |
| 29 | 0.0 | 1.2 | 1.2 | 0.8 | 0.7 | 1.3 | 0.0 | 1.2 | 1.2 | 0.8 | 0.7 | 1.3 | 0.0 | 1.2 | 1.2 | 0.8 | 0.7 | |

Acetate

| day | M6LD | | | M6LE | | | M6LF | | | M6HA | | | M6HB | | | M6HC | | |
|-----|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|--|
| | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | 28 | 28 | 28 | 28 | 25 | n.d. | 28 | 28 | 25 | 25 | 25 | n.d. | 28 | 28 | 25 | 25 | |
| 2 | n.d. | 60 | 77 | 80 | 73 | 62 | n.d. | 60 | 77 | 80 | 73 | 62 | n.d. | 60 | 77 | 80 | 73 | |
| 6 | 1818 | 1738 | 1718 | 1745 | 1719 | 1719 | 1818 | 1738 | 1718 | 1745 | 1719 | 1719 | 1818 | 1738 | 1718 | 1745 | 1719 | |
| 14 | 2132 | 1914 | 1931 | 2008 | 1858 | 1858 | 2132 | 1914 | 1931 | 2008 | 1858 | 1858 | 2132 | 1914 | 1931 | 2008 | 1858 | |
| 29 | 2238 | 927 | 1932 | 1898 | 1781 | 1781 | 2238 | 927 | 1932 | 1898 | 1781 | 1781 | 2238 | 927 | 1932 | 1898 | 1781 | |

Propionate

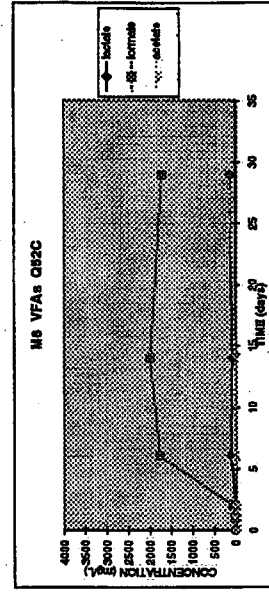
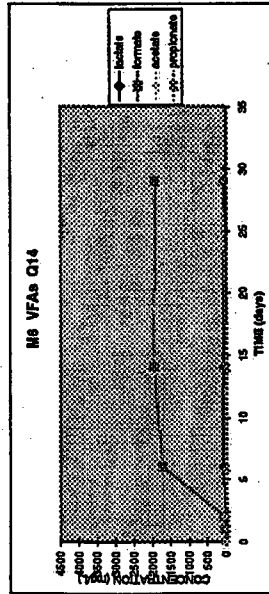
| day | M6LD | | | M6LE | | | M6LF | | | M6HA | | | M6HB | | | M6HC | | |
|-----|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|--|
| | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | concentration | |
| 0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 2 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 6 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 14 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | |
| 29 | 259 | n.d. | 168 | n.d. | 112 | 112 | 259 | n.d. | 168 | n.d. | 112 | 112 | 259 | n.d. | 168 | n.d. | 112 | |

OS2C

| day | lactate | formate | acetate | propionate |
|-----|---------|---------|---------|------------|
| 0 | 0 | 0 | 0 | 0 |
| 1 | 0 | 29 | 0 | 0 |
| 2 | 0 | 79 | 0 | 0 |
| 6 | 98 | 1784 | 921 | 0 |
| 14 | 72 | 1933 | 1098 | 0 |
| 29 | 138 | 1689 | 3882 | 142.3333 |

Q14

| day | lactate | formate | acetate | propionate |
|-----|---------|---------|---------|------------|
| 0 | 0 | 0 | 0 | 0 |
| 1 | 0 | 25 | 0 | 0 |
| 2 | 0 | 69 | 0 | 0 |
| 6 | 89 | 1728 | 815 | 0 |
| 14 | 73 | 1988 | 1042 | 0 |
| 29 | 67 | 1929 | 3947 | 78.33333 |



Tetrachloroethane (TeCA)

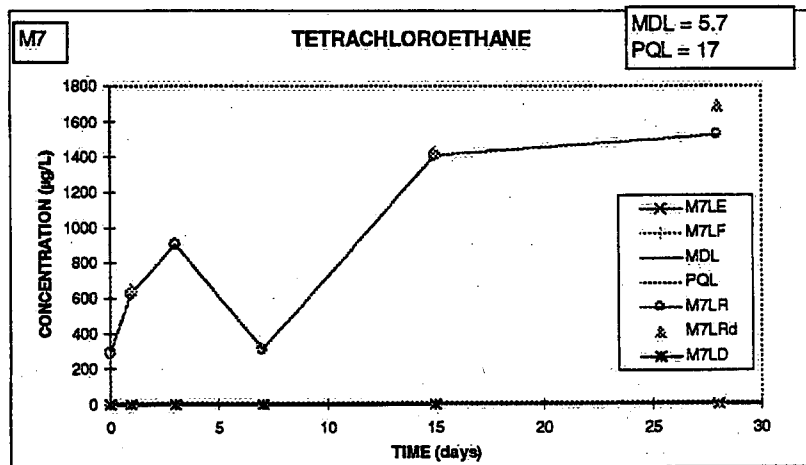
Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER

695

| Time | M7LR | M7LD | M7LE | M7LF | Dup r^2 (if < 0.99) |
|------|------|------|------|------|-----------------------|
| 0 | 291 | n.d. | | | 0.981 |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | 627 | n.d. | n.d. | n.d. | 0.981 |
| 3 | 905 | n.d. | n.d. | n.d. | 0.974 |
| 7 | 317 | | | | 0.974 |
| 15 | 1409 | n.d. | n.d. | n.d. | 0.982 |
| 28 | 1518 | n.d. | n.d. | n.d. | 0.966 |



Carbon tetrachloride

Microcosm 7 Alternate chelator

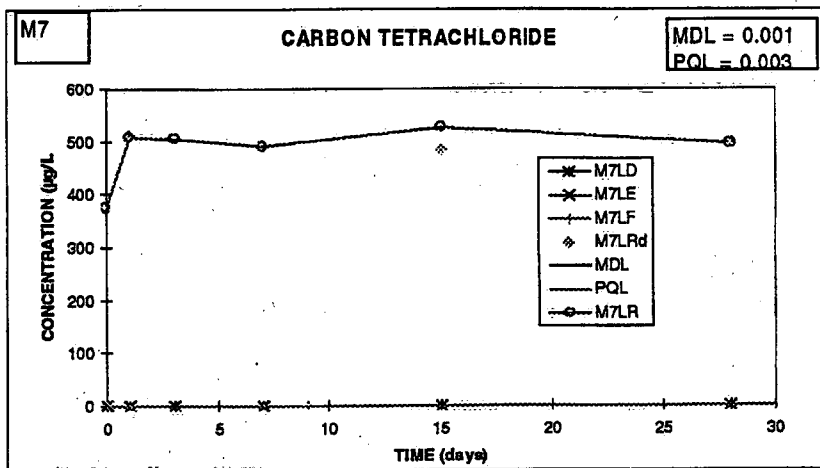
MAC = 5 µg/L

Result concentration in µg/L

SITE WATER

364

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|-----|
| 0 | 374 | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | 506 | n.d. | n.d. | n.d. | 511 |
| 3 | 503 | n.d. | n.d. | n.d. | |
| 7 | 490 | n.d. | n.d. | n.d. | |
| 15 | 526 | n.d. | n.d. | n.d. | 486 |
| 28 | 495 | n.d. | n.d. | n.d. | 497 |



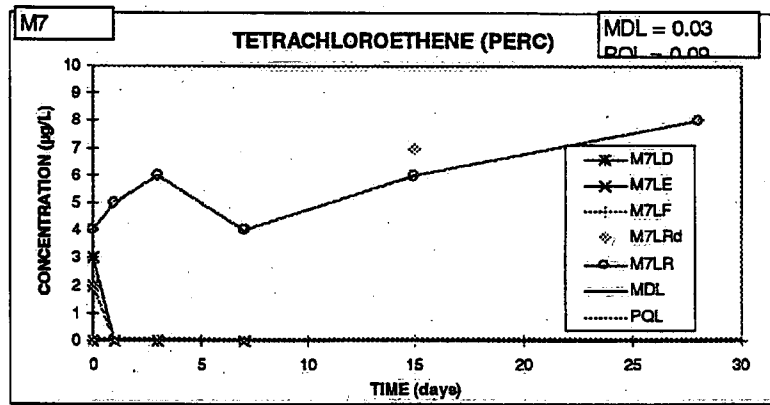
TETRACHLOROETHENE (PERC)

Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|-----|
| 0 | 4 | 3 | | | 6 |
| 0.04 | | | 2 | | |
| 0.08 | | | | 2 | |
| 1 | 5 | n.d. | n.d. | n.d. | 5 |
| 3 | 6 | n.d. | n.d. | n.d. | |
| 7 | 4 | n.d. | n.d. | n.d. | |
| 15 | 6 | n.d. | n.d. | n.d. | 7 |
| 28 | 8 | n.d. | n.d. | n.d. | 8 |



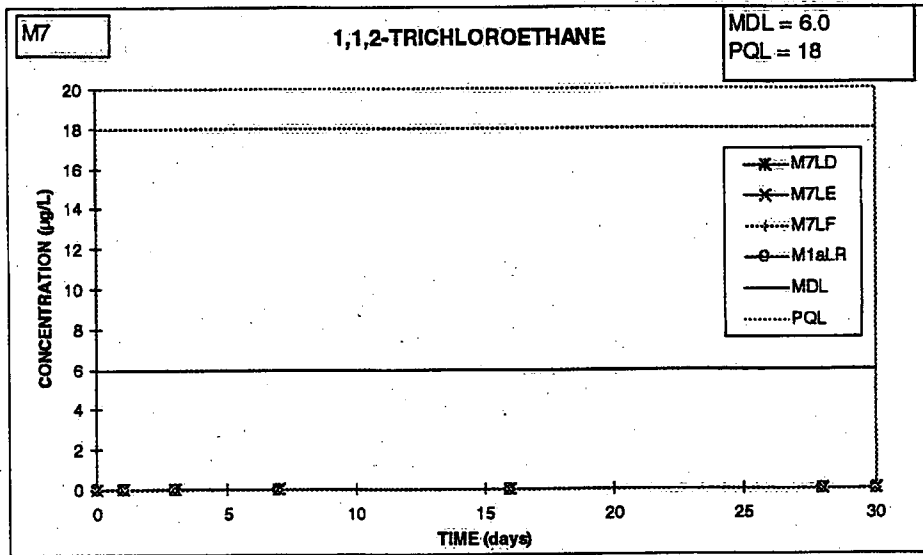
1,1,2-Trichloroethane

Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. |



TRICHLOROETHENE (TCE)

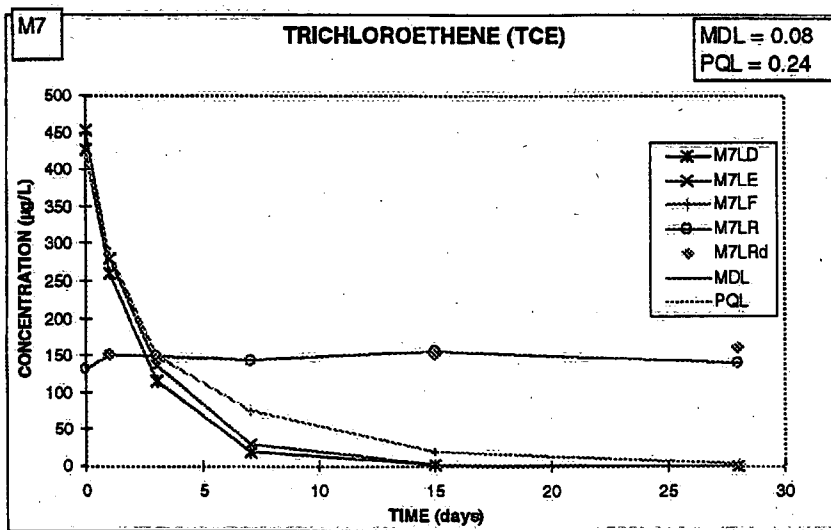
Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER

86

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|-----|
| 0 | 131 | 426 | | | |
| 0.04 | | | 453 | | |
| 0.08 | | | | 419 | |
| 1 | 151 | 261 | 280 | 287 | 151 |
| 3 | 149 | 115 | 136 | 149 | |
| 7 | 143 | 20 | 29 | 75 | |
| 15 | 155 | 2 | 2 | 20 | 152 |
| 28 | 140 | n.d. | n.d. | 3 | 160 |



CHLOROFORM

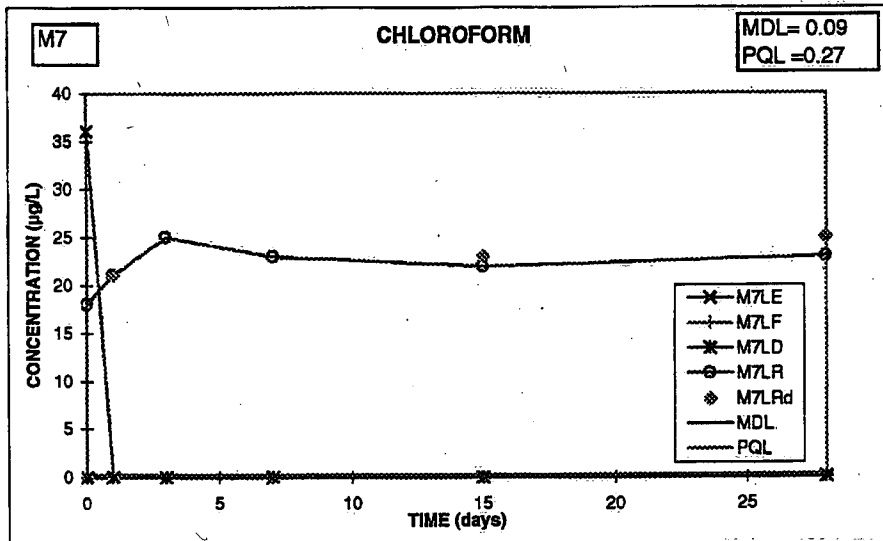
Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER

20

| Time | M7LR | M7LD | M7LE | M7LF | Dup | r^2 (t) < 0.99 |
|------|------|------|------|------|-----|------------------|
| 0 | 18 | 36 | | | | |
| 0.04 | | | n.d. | | | |
| 0.08 | | | | n.d. | | |
| 1 | 21 | n.d. | n.d. | n.d. | 21 | 0.976 |
| 3 | 25 | n.d. | n.d. | n.d. | | |
| 7 | 23 | n.d. | n.d. | n.d. | | |
| 15 | 22 | n.d. | n.d. | n.d. | 23 | |
| 28 | 23 | n.d. | n.d. | n.d. | 25 | |



DICHLOROMETHANE

Microcosm 7 Alternate chelator

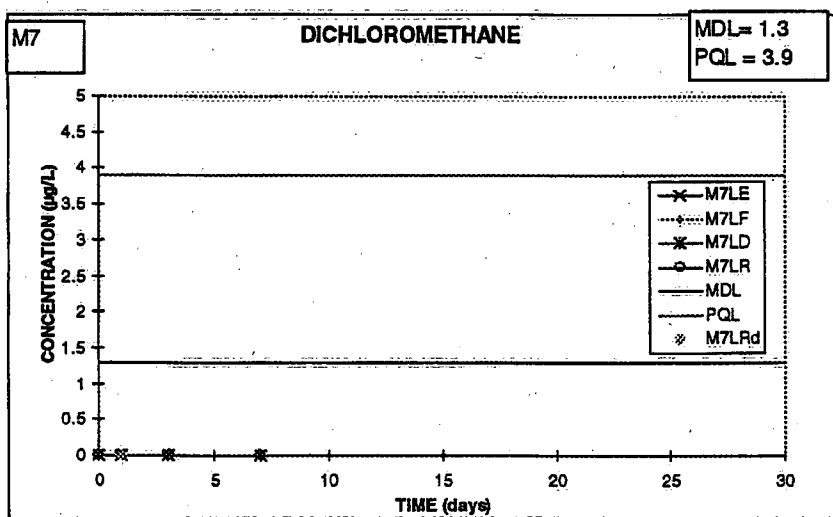
MAC = 5 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | | | |
| 0.08 | | | n.d. | | |
| 1 | | | | n.d. | |
| 3 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. |



cis-DICHLOROETHENE

Microcosm 7 Alternate chelator

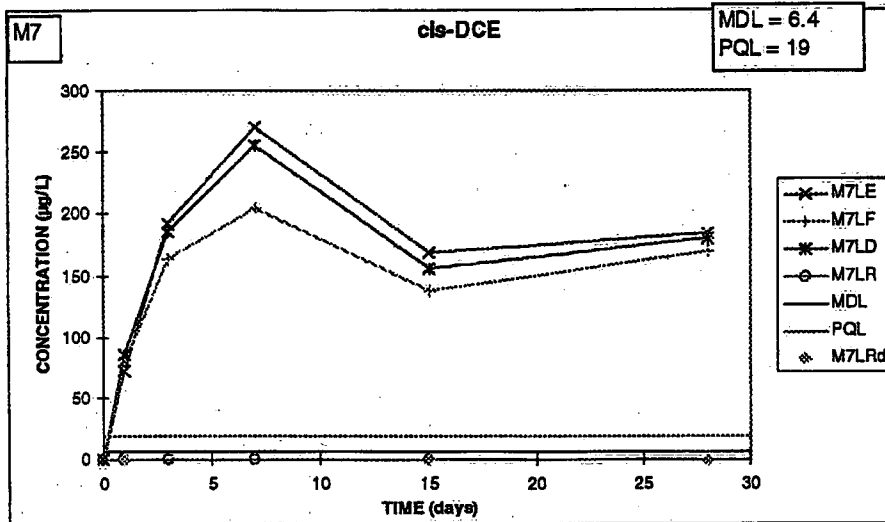
MAC = 70 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M7LR | M7LD | M7LE | M7LF | Dup t^2 ($t < 0.99$) |
|------|------|------|------|------|--------------------------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | 86 | 72 | 78 | n.d. 0.982 |
| 3 | n.d. | 185 | 192 | 163 | 0.956 |
| 7 | n.d. | 255 | 270 | 209 | 0.956 |
| 15 | n.d. | 156 | 169 | 138 | n.d. |
| 28 | n.d. | 180 | 184 | 170 | n.d. |



Trans-DICHLOROETHENE

Microcosm 7 Alternate chelator

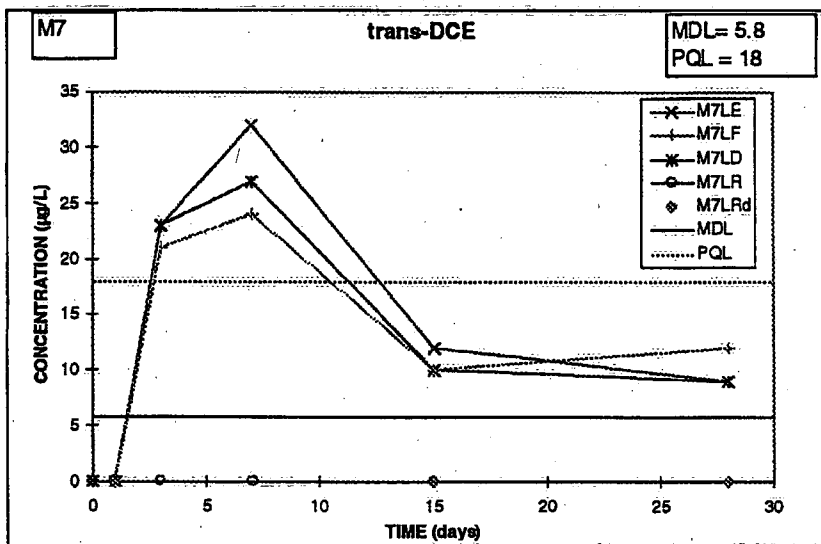
MAC = 100 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------------|
| 0 | n.d. | n.d. | | | (f < 0.99) |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | 23 | 23 | 21 | 0.973 |
| 7 | n.d. | 27 | 32 | 24 | |
| 15 | n.d. | 10 | 12 | 10 | n.d. |
| 28 | n.d. | 9j | 9j | 12j | n.d. |



1,1_Dichloroethene

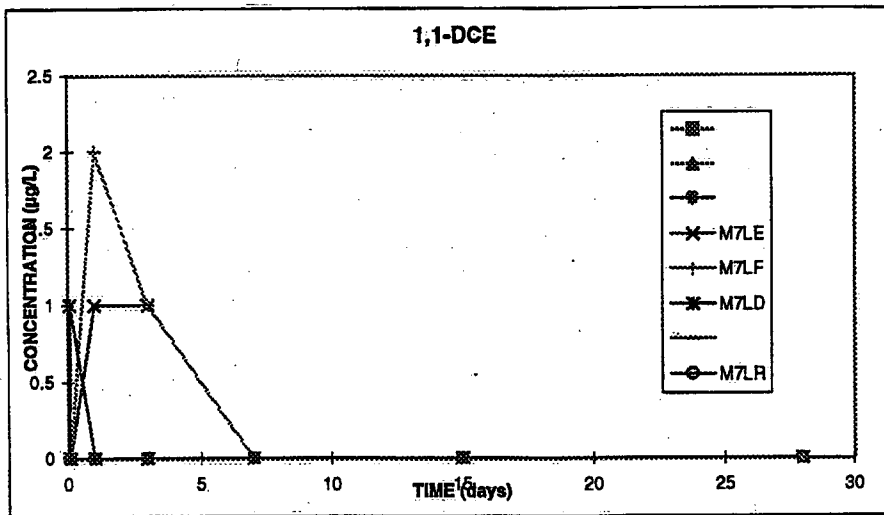
Microcosm 7 Alternate chelator

PQL = 11 µg/L

Result concentration in µg/L

| Time | SITE WATER | <PQL | | | | Dup |
|------|------------|------|------|------|------|------|
| | | M7LR | M7LD | M7LE | M7LF | |
| 0 | | | | | | |
| 0.04 | | | | | | |
| 0.08 | | | | | | |
| 1 | | n.d. | n.d. | n.d. | n.d. | |
| 3 | | n.d. | 1 | 1 | 2 | n.d. |
| 7 | | n.d. | n.d. | 1 | 1 | |
| 15 | | n.d. | n.d. | n.d. | n.d. | |
| 28 | | n.d. | n.d. | n.d. | n.d. | n.d. |

Note: needs to be confirmed by MS



VINYL CHLORIDE (VC)

Microcosm 7 Alternate chelator

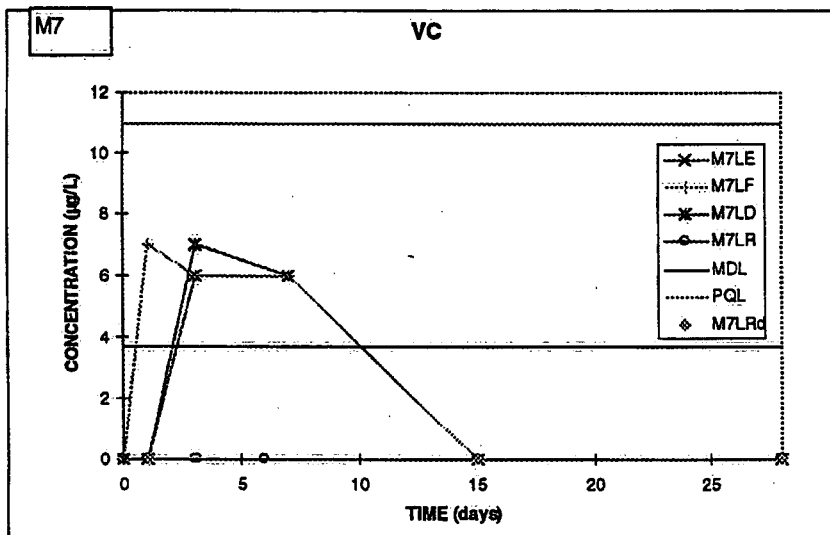
PQL = 11 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | 7j | 6j | 6j | |
| 7 | n.d. | 6j | 6j | 6j | |
| 15 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. | n.d. |



CHLOROMETHANE

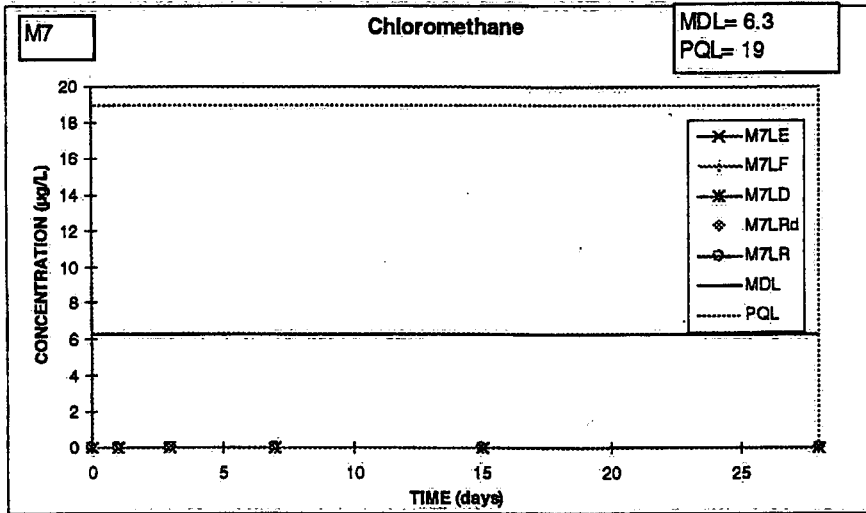
Microcosm 7 Alternate chelator

Result concentration in µg/L

SITE WATER <PQL

<PQL

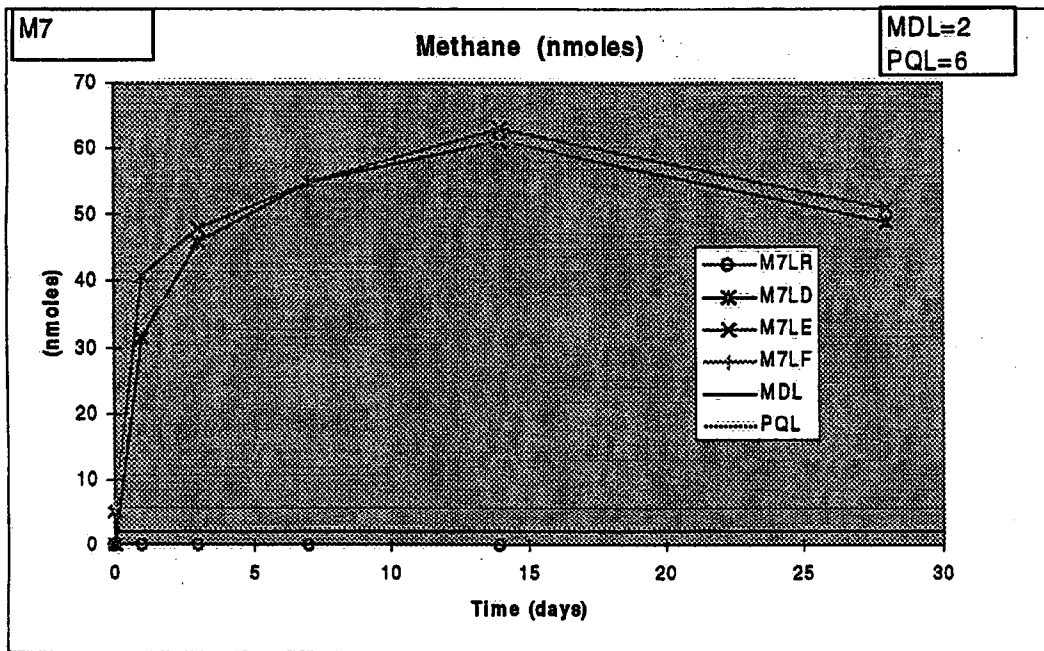
| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | n.d. | |



METHANE

Microcosm 7 Alternate chelator

| Time | SITE WATER | RESULTS in ppmv | | | | Dup |
|------|------------|-----------------|------|------|------|------|
| | | M7LR | M7LD | M7LE | M7LF | |
| 0 | <PQL | n.d. | n.d. | | | |
| 0.04 | | | | 5j | | |
| 0.08 | | | | | 6 | |
| 1 | | n.d. | 31 | 40 | 34 | n.d. |
| 3 | | n.d. | 45 | 47 | 40 | |
| 7 | | n.d. | 54 | 54 | 52 | |
| 15 | | n.d. | 60 | 62 | 60 | n.d. |
| 28 | | n.d. | 48 | 50 | 46 | n.d. |



ETHANE

Microcosm 7 Alternate chelator

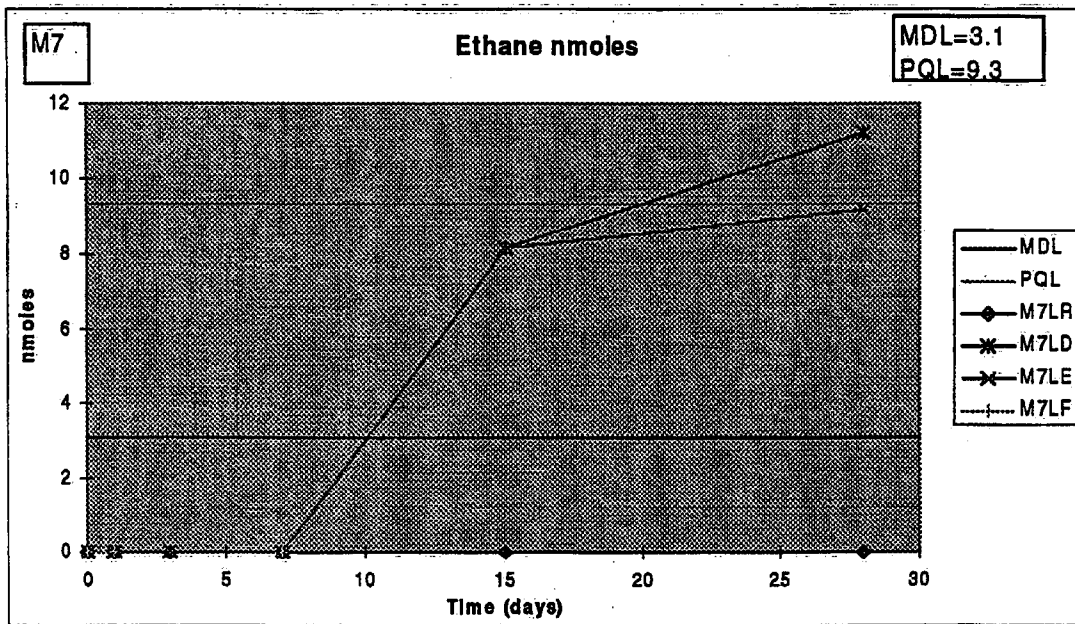
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | 3j | 3j | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | 8j | 8j | 6j | n.d. |
| 28 | n.d. | 11 | 9 | 5j | n.d. |



ETHENE

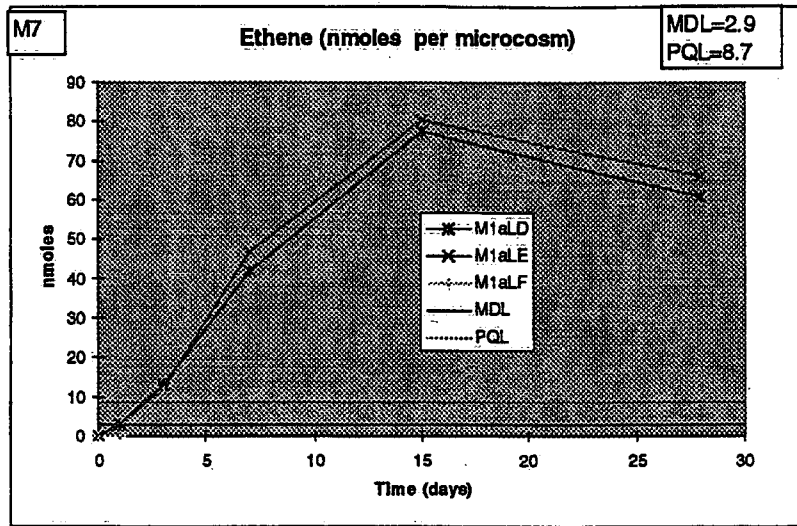
Microcosm 7 Alternate chelator

SITE WATER <PQL

<PQL

Results in ppmv

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | 3j | 3j | n.d. | |
| 3 | n.d. | 13 | 12 | 11 | |
| 7 | n.d. | 41 | 46 | 38 | |
| 15 | n.d. | 76 | 79 | 72 | n.d. |
| 28 | n.d. | 60 | 65 | 54 | n.d. |



ACETYLENE

Microcosm 7 Alternate chelator

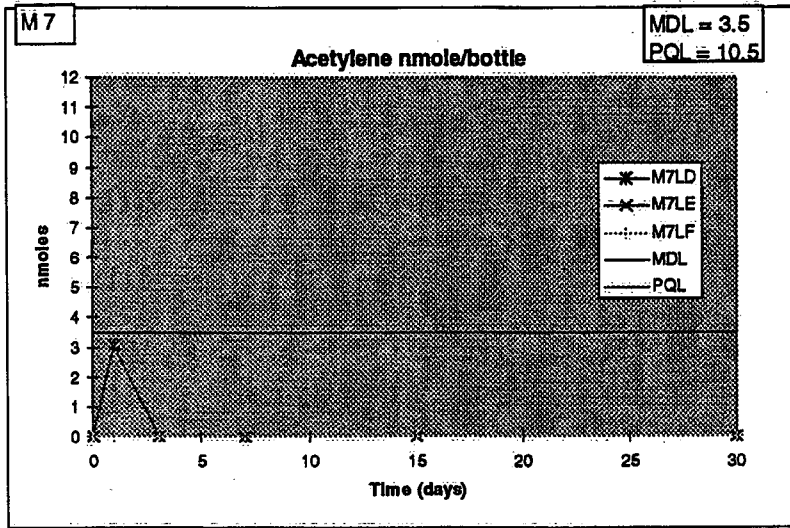
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M7LR | M7LD | M7LE | M7LF | Dup |
|------|------|------|------|------|------|
| 0 | n.d. | n.d. | | | |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | n.d. | 3j | 3j | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | n.d. | |



Volatile Fatty Acids - M7

Citrate

| day | M7LD | M7LE | conc. (mg/L) | |
|-----|------|------|--------------|--|
| | | | M7LF | |
| 0 | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | |

Titanium

| day | M7LD | M7LE | conc. (mM) | |
|-----|------|------|------------|--|
| | | | M7LF | |
| 0 | 25.1 | 24.8 | 24.8 | |

Lactate

| day | M7LD | M7LE | conc. (mg/L) | |
|-----|------|------|--------------|--|
| | | | M7LF | |
| 0 | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | |

Formate

| day | M7LD | M7LE | conc. (mg/L) | |
|-----|------|------|--------------|--|
| | | | M7LF | |
| 0 | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | |
| 28 | 31 | 23 | 26 | |

Acetate

| day | M7LD | M7LE | conc. (mg/L) | |
|-----|------|------|--------------|--|
| | | | M7LF | |
| 0 | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | |

Propionate

| day | M7LD | M7LE | conc. (mg/L) | |
|-----|------|------|--------------|--|
| | | | M7LF | |
| 0 | n.d. | n.d. | n.d. | |
| 1 | n.d. | n.d. | n.d. | |
| 3 | n.d. | n.d. | n.d. | |
| 7 | n.d. | n.d. | n.d. | |
| 15 | n.d. | n.d. | n.d. | |
| 28 | n.d. | n.d. | n.d. | |

Tetrachloroethane (TeCA)

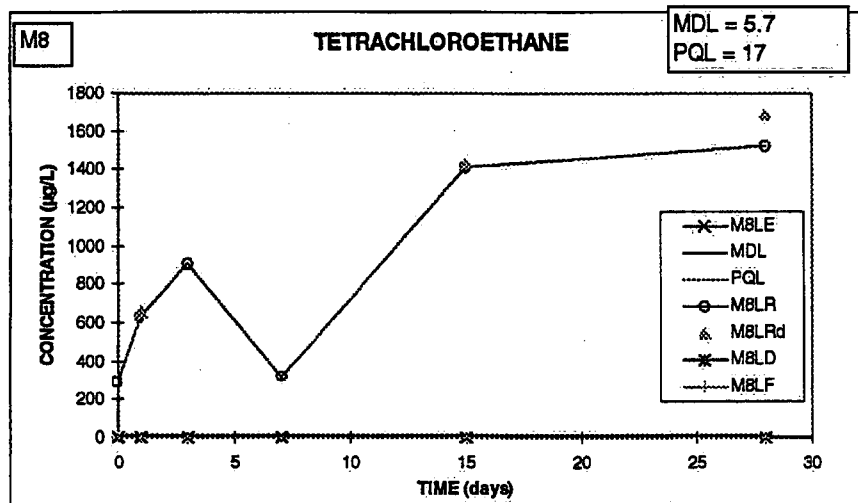
Microcosm 8 B12, TI citrate and yeast extract

Result concentration in µg/L

SITE WATER

695

| Time | M8LR | M8LD | M8LE | M8LF | r^2 ($t < 0.99$) |
|------|------|------|------|------|----------------------|
| 0 | 291 | n.d. | | | 0.981 |
| 0.04 | | | n.d. | | |
| 0.08 | | | | n.d. | |
| 1 | 627 | n.d. | n.d. | n.d. | 0.981 |
| 3 | 905 | n.d. | n.d. | n.d. | 0.974 |
| 7 | 317 | n.d. | n.d. | n.d. | 0.974 |
| 15 | 1409 | n.d. | n.d. | n.d. | 0.982 |
| 28 | 1518 | n.d. | n.d. | n.d. | 0.966 |



Carbon tetrachloride

Microcosm 8 B12, TI citrate and yeast extract

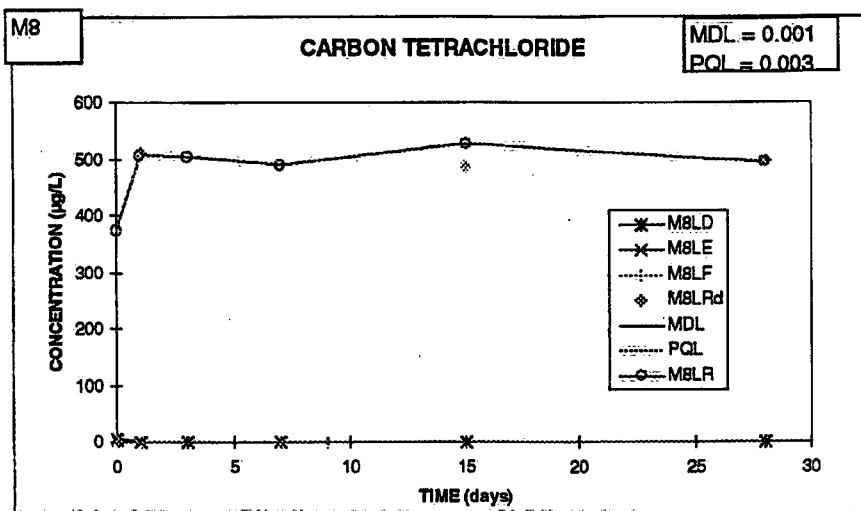
MAC = 5 µg/L

Result concentration in µg/L

SITE WATER

364

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | 374 | 5 | | |
| 0.04 | | | 3j | |
| 0.08 | | | | 3j |
| 1 | 506 | n.d. | n.d. | n.d. |
| 3 | 503 | n.d. | n.d. | n.d. |
| 7 | 490 | n.d. | n.d. | n.d. |
| 15 | 526 | n.d. | n.d. | n.d. |
| 28 | 495 | n.d. | n.d. | n.d. |



TETRACHLOROETHENE (PERC)

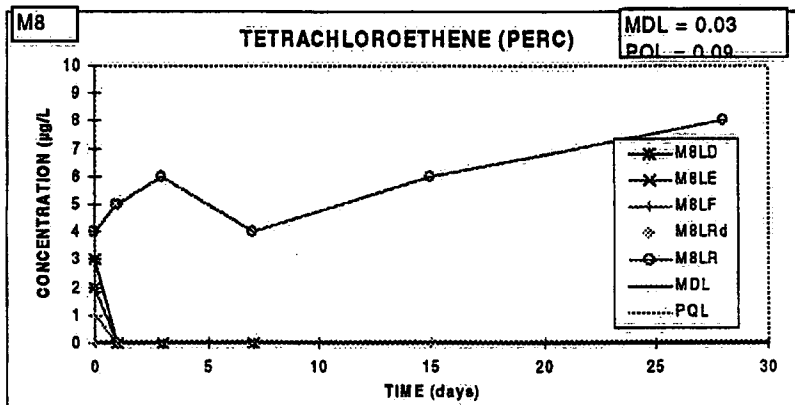
Microcosm 8 B12, TI citrate and yeast extract

Result concentration in µg/L

SITE WATER

6

| Time | M8LR | M8LD | M8LE | M8LF | P (t < 0.99) |
|------|------|------|------|------|--------------|
| 0 | 4 | 3 | | | |
| 0.04 | | | 2 | | |
| 0.08 | | | | 1 | |
| 1 | 5 | n.d | n.d | n.d | 5 |
| 3 | 6 | n.d | n.d | n.d | |
| 7 | 4 | n.d | n.d | n.d | |
| 15 | 6 | n.d | n.d | n.d | |
| 28 | 8 | n.d | n.d | n.d | 8 |



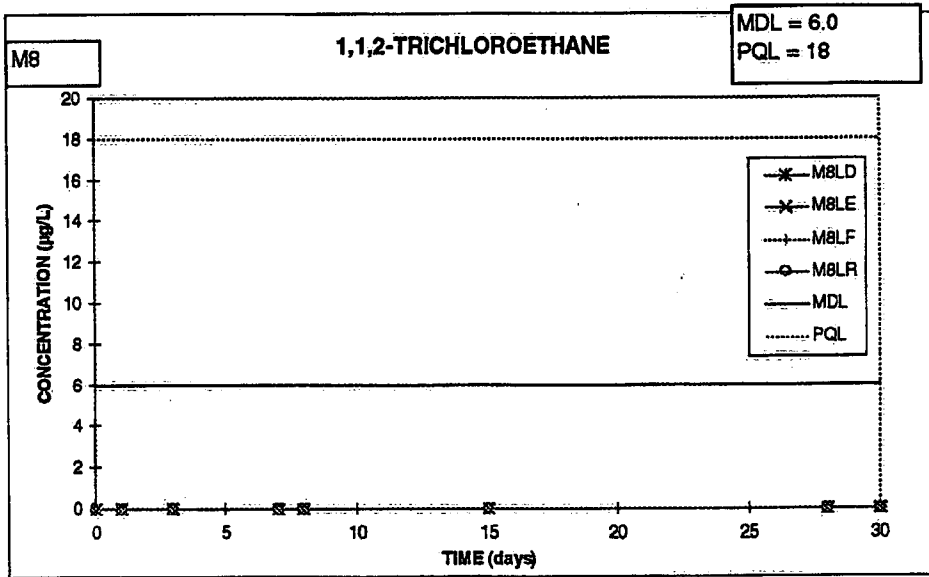
1,1,2-Trichloroethane

Microcosm 8 B12, TI citrate and yeast extract

Result concentration in µg/L

SITE WATER

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | 27 | | |
| 0.04 | | | n.d. | |
| 0.08 | | | | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. |



TRICHLOROETHENE (TCE)

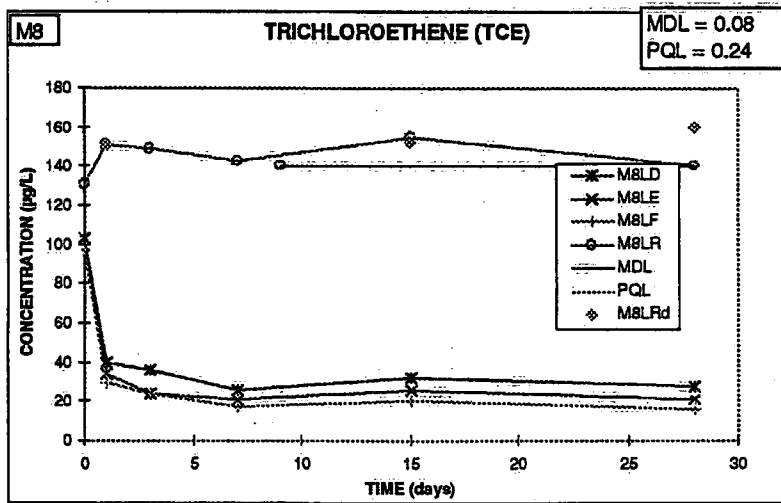
Microcosm 8 B12, T1 citrate and yeast extract

Result concentration in µg/L

SITE WATER 197

86

| Time | M8LR | M8LD | M8LE | M8LF | |
|------|------|------|------|------|-------|
| 0 | 131 | 103 | | | 0.99 |
| 0.04 | | | 103 | | |
| 0.08 | | | | 97 | |
| 1 | 151 | 40 | 34 | 30 | 0.98 |
| 3 | 149 | 36 | 24 | 24 | 0.915 |
| 7 | 143 | 26 | 21 | 17 | |
| 15 | 155 | 32 | 26 | 20 | 0.953 |
| 28 | 140 | 28 | 21 | 16 | 0.954 |



CHLOROFORM

Microcosm 8 B12, TI citrate and yeast extract

Result concentration in µg/L

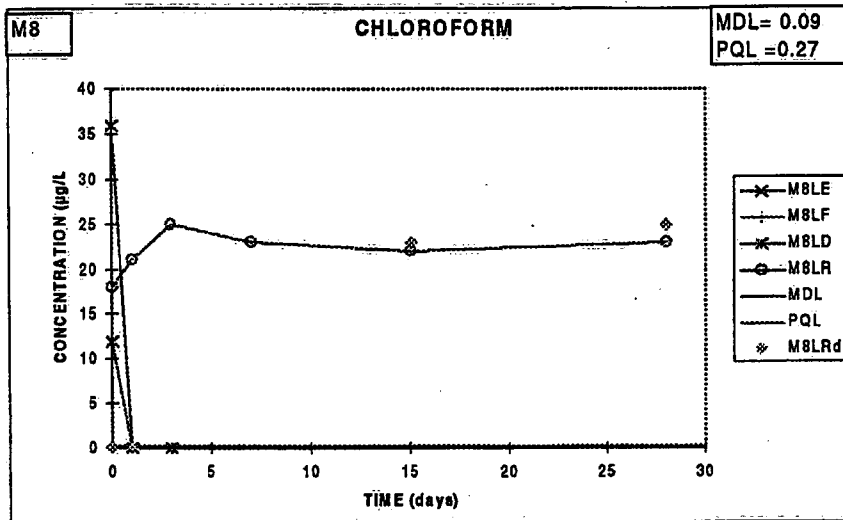
SITE WATER

20

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | 18 | 12 | | |
| 0.04 | | | n.d. | |
| 0.08 | | | | n.d. |
| 1 | 21 | n.d. | n.d. | n.d. |
| 3 | 25 | n.d. | n.d. | n.d. |
| 7 | 23 | n.d. | n.d. | n.d. |
| 15 | 22 | n.d. | n.d. | n.d. |
| 28 | 23 | n.d. | n.d. | n.d. |

0.99

0.975



DICHLOROMETHANE

Microcosm 8 B12, T1 citrate and yeast extract

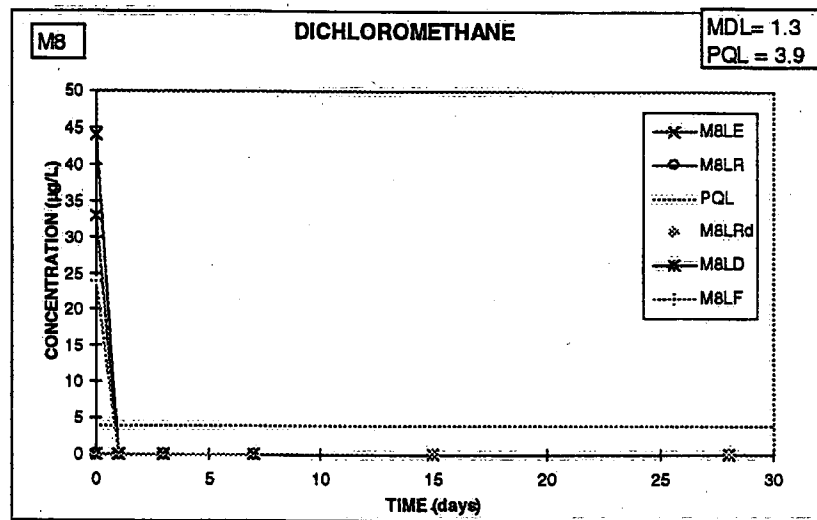
Result concentration in µg/L

MAC = 5 µg/L

SITE WATER <PQL

<PQL

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | 44 | | |
| 0.04 | | | 33 | |
| 0.08 | | | | 24 |
| 1 | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. |



cis-DICHLOROETHENE

Microcosm 8 B12, TI citrate and yeast extract

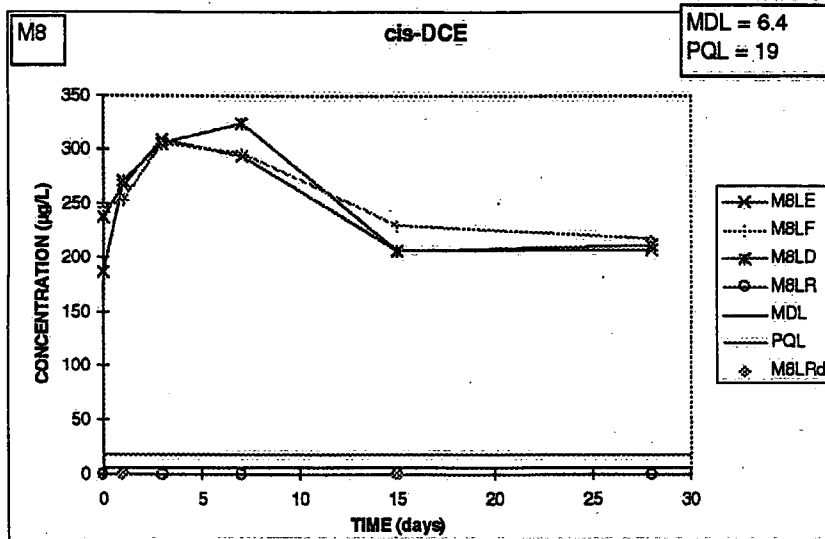
MAC = 70 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M8LR | M8LD | M8LE | M8LF | r ² (n=3, 99) |
|------|------|------|------|------|--------------------------|
| 0 | n.d. | 187 | | | |
| 0.04 | | | 237 | | |
| 0.08 | | | | 246 | |
| 1 | n.d. | 271 | 267 | 254 | |
| 3 | n.d. | 305 | 309 | 306 | 0.956 |
| 7 | n.d. | 324 | 294 | 297 | 0.956 |
| 15 | n.d. | 207 | 207 | 230 | |
| 28 | n.d. | 211 | 208 | 218 | |



Trans-DICHLOROETHENE

Microcosm 8 B12, TI citrate and yeast extract

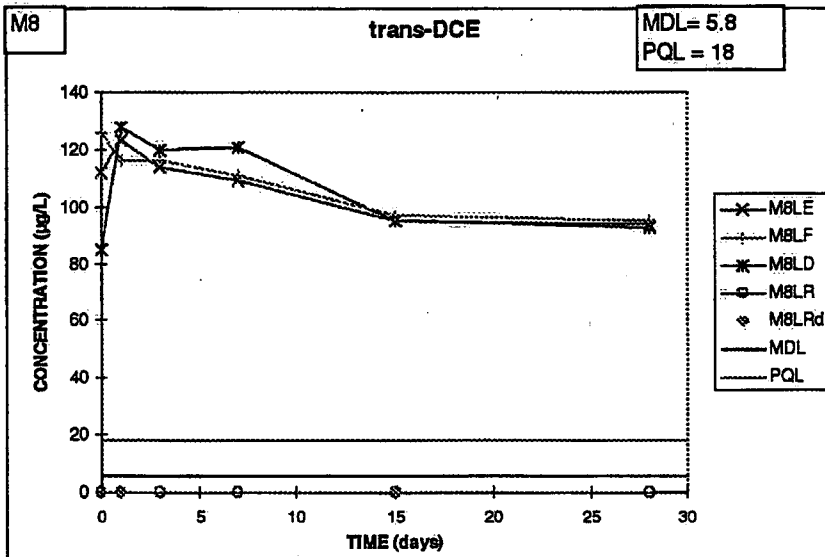
MAC = 100 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M8LR | M8LD | M8LE | M8LF | r ² (n=0.99) |
|------|------|------|------|------|-------------------------|
| 0 | n.d. | 85 | | | 0.975 |
| 0.04 | | | 112 | | |
| 0.08 | | | | 126 | |
| 1 | n.d. | 128 | 123 | 116 | 0.975 |
| 3 | n.d. | 120 | 114 | 116 | 0.973 |
| 7 | n.d. | 121 | 109 | 111 | |
| 15 | n.d. | 95 | 95 | 97 | |
| 28 | n.d. | 93 | 94 | 95 | |



VINYL CHLORIDE (VC)

Microcosm 8 B12, TI citrate and yeast extract

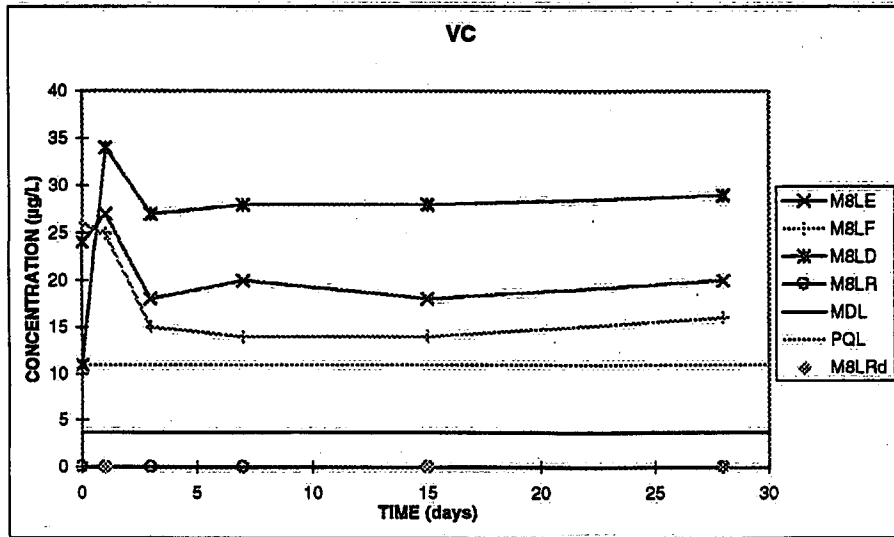
PQL = 11 µg/L

Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | 11 | | |
| 0.04 | | | 24 | |
| 0.08 | | | | 26 |
| 1 | n.d. | 34 | 27 | 25 |
| 3 | n.d. | 27 | 18 | 15 |
| 7 | n.d. | 28 | 20 | 14 |
| 15 | n.d. | 28 | 18 | 14 |
| 28 | n.d. | 29 | 20 | 16 |



CHLOROMETHANE

Microcosm 8 B12, TI citrate and yeast extract

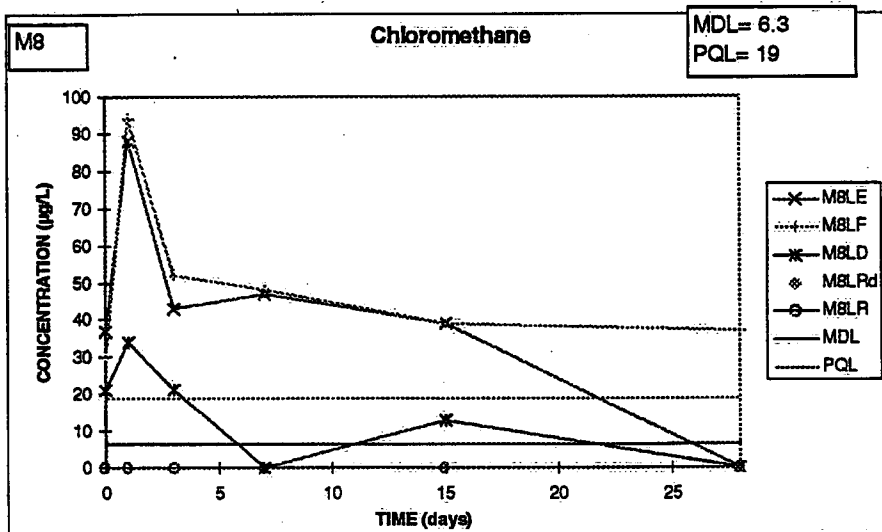
Result concentration in µg/L

SITE WATER <PQL

<PQL

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | 21 | | |
| 0.04 | | | 37 | |
| 0.08 | | | | 30 |
| 1 | n.d. | 34 | 88 | 94 |
| 3 | n.d. | 21 | 43 | 52 |
| 7 | n.d. | n.d. | 47 | 48 |
| 15 | n.d. | 13 | 39 | 39 |
| 28 | n.d. | n.d. | n.d. | 37 |

May be not chloromethane - need MS confirmation



METHANE

Microcosm 8 B12, Ti citrate and yeast extract

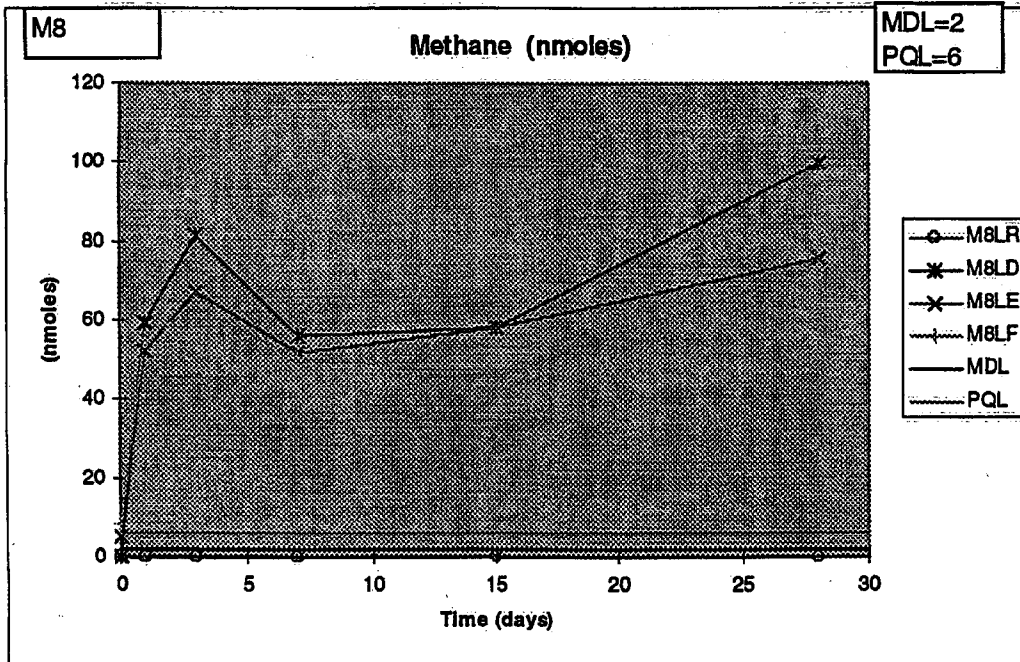
SITE WATER

<PQL

<PQL

RESULTS in ppmv

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | n.d. | | |
| 0.04 | | | 5 | |
| 0.08 | | | | 8 |
| 1 | n.d. | 48 | 42 | 43 |
| 3 | n.d. | 46 | 42 | 41 |
| 7 | n.d. | 48 | 45 | 42 |
| 15 | n.d. | 57 | 57 | 45 |
| 28 | n.d. | 98 | 74 | 72 |



ETHANE

Microcosm 8 B12, TI citrate and yeast extract

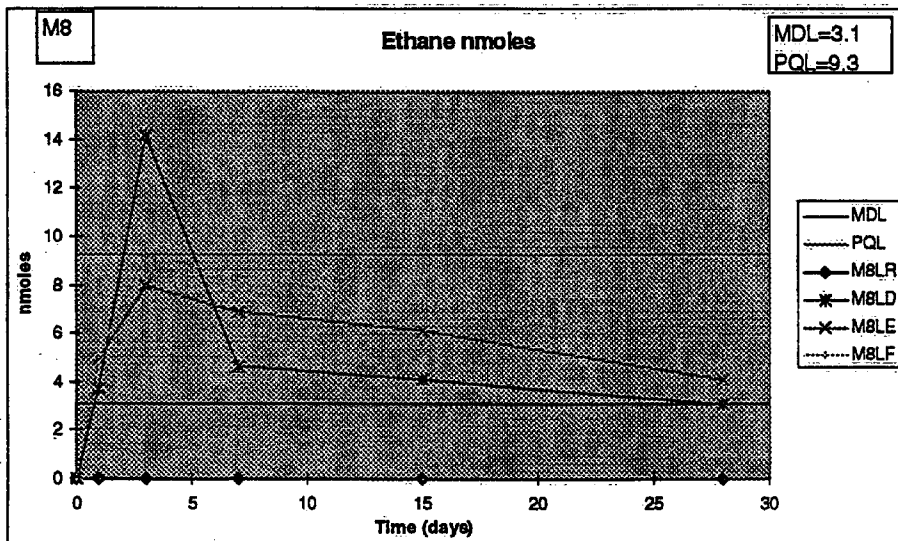
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | n.d. | | |
| 0.04 | | | n.d. | |
| 0.08 | | | | n.d. |
| 1 | n.d. | 3j | 4j | 4j |
| 3 | n.d. | 8j | 5j | 6j |
| 7 | n.d. | 4j | 6j | 7j |
| 15 | n.d. | 4j | 6j | 7j |
| 28 | n.d. | 3j | 4j | 5j |



ETHENE

Microcosm 8

B12, TI citrate and yeast extract

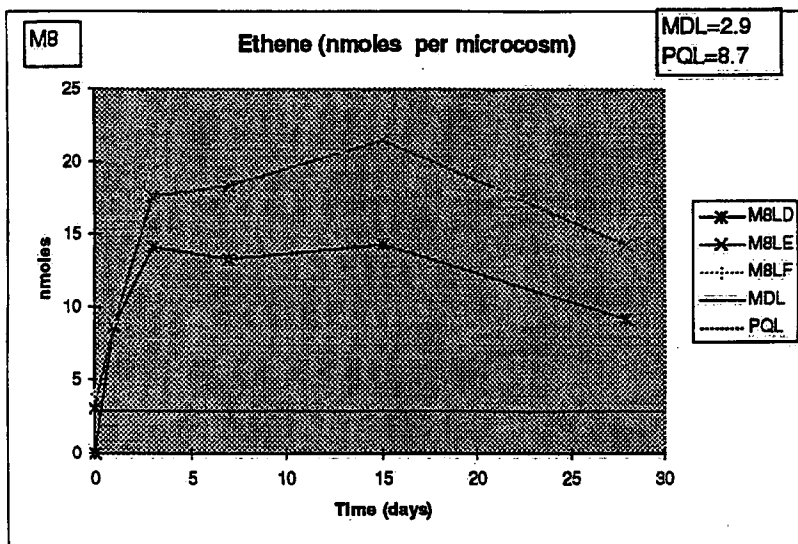
Result concentration in µg/L

SITE WATER <PQL

Results in ppmv

<PQL

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | n.d. | | |
| 0.04 | | | 3j | |
| 0.08 | | | | 4j |
| 1 | n.d. | 7j | 7j | 7j |
| 3 | n.d. | 8j | 11 | 13 |
| 7 | n.d. | 13 | 18 | 21 |
| 15 | n.d. | 14 | 21 | 20 |
| 28 | n.d. | 9 | 14 | 15 |



ACETYLENE

Microcosm 8 B12, TI citrate and yeast extract

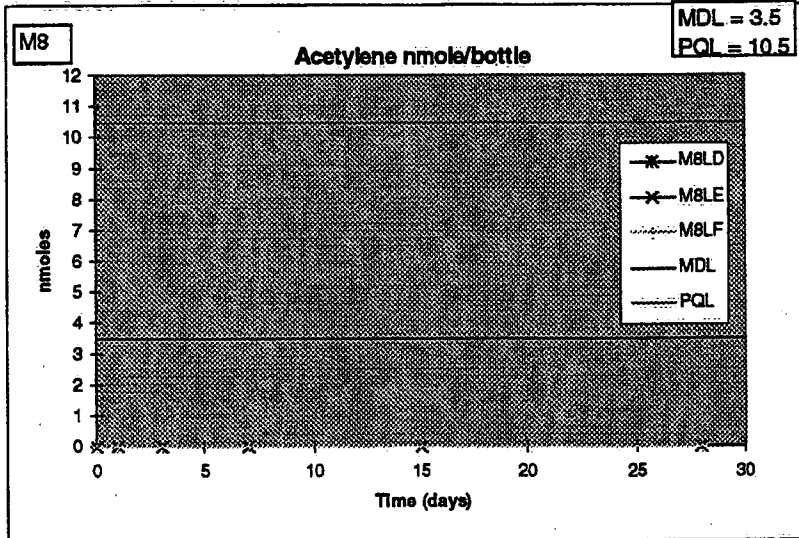
SITE WATER

<PQL

<PQL

Results in ppmv

| Time | M8LR | M8LD | M8LE | M8LF |
|------|------|------|------|------|
| 0 | n.d. | n.d. | | |
| 0.04 | | | n.d. | |
| 0.08 | | | | n.d. |
| 1 | n.d. | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. | n.d. |



Volatile Fatty Acids - M8

Citrate

| day | conc. (mg/L) | | |
|-----|--------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | 6679 | 6474 | 6678 |
| 1 | 2507 | 1387 | 1627 |
| 3 | n.d. | n.d. | 890j |
| 7 | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. |

Titanium

| day | conc. (mM) | | |
|-----|------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | 28.9 | 29.0 | 29.2 |

Lactate

| day | conc. (mg/L) | | |
|-----|--------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | n.d. | n.d. | n.d. |
| 1 | n.d. | n.d. | n.d. |
| 3 | n.d. | n.d. | n.d. |
| 7 | n.d. | n.d. | n.d. |
| 15 | n.d. | n.d. | n.d. |
| 28 | n.d. | n.d. | n.d. |

Formate

| day | conc. (mg/L) | | |
|-----|--------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | 38 | 50 | 46 |
| 1 | 603 | 957 | 982 |
| 3 | 936 | 943 | 1051 |
| 7 | 722 | 832 | 861 |
| 15 | 726 | 743 | 913 |
| 28 | 309 | 378 | 554 |

Acetate

| day | conc. (mg/L) | | |
|-----|--------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | 344 | 373 | 285 |
| 1 | 2554 | 3642 | 3679 |
| 3 | 4958 | 4265 | 4335 |
| 7 | 4565 | 5089 | 4919 |
| 15 | 4444 | 4992 | 4768 |
| 28 | 4941 | 4374 | 4501 |

Propionate

| day | conc. (mg/L) | | |
|-----|--------------|------|------|
| | M8LD | M8LE | M8LF |
| 0 | 265 | 269 | 241 |
| 1 | 403 | 375 | 359 |
| 3 | 414 | 301 | 267 |
| 7 | 313 | 309 | 292 |
| 15 | 364 | 370 | 310 |
| 28 | 494 | 391 | 408 |

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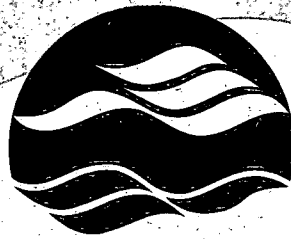
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