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# Stability of Volatile Organics in Environmental Soil Samples

## Final Report

M. P. Maskarinec
C. K. Bayne
R. A. Jenkins
L. H. Johnson
S. K. Holladay

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# STABILITY OF VOLATILE ORGANICS IN ENVIRONMENTAL SOIL SAMPLES

FINAL REPORT

November 1992

M. P. Maskarinec, C. K. Bayne, R. A. Jenkins,
L. H. Johnson, and S. K. Holladay

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# EXECUTIVE SUMMARY

This report focuses on data generated for the purpose of establishing the stability of 19 volatile organic compounds in environmental soil samples. The study was carried out over a 56 day (for two soils) and a 111 day (for one reference soil) time frame and took into account as many variables as possible within the constraints of budget and time. The objectives of the study were: 1) to provide a data base which could be used to provide guidance on pre-analytical holding times for regulatory purposes; and 2) to provide a basis for the evaluation of data which is generated outside of the currently allowable holding times.

The experimental design consisted of three soil samples. The soil samples were a U.S. Army Toxic and Hazardous Materials Agency reference soil (soil: 7% sand - 67% silt - 26% clay), a Captina silt loam from Roane County, Tennessee (8% sand - 62% silt - 30% clay), and a McLaurin sandy loam from Stone County, Mississippi (75% sand - 20% silt - 5% clay). The analytes consisted of the most commonly encountered volatile organic compounds. All analyses were carried out using methods similar to those in the USEPA Contract Laboratory Program. GC/MS was used for all determinations. All determinations were carried out in quadruplicate along with a storage blank. Each analyte had an initial concentration at a nominal low-level (µg/g or ppm) to represent values that may be encountered in practice. Initial low-level concentrations for most analytes were in the range 50-80 µg/g (i.e., 14 analytes) with 4 analytes having initial concentrations in the 20-50 µg/g range. One analyte (i.e., methylene chloride) had initial concentrations of 58 µg/g, 99 µg/g, and 161 µg/g depending on the soil type. Soil samples were stored at three temperatures, -70° C, -20° C, and 4° C (not used for USATHAMA soil samples). Samples were analyzed at time intervals of 0, 3, 7, 14, 28, 56, and 111 days. A maximum holding time of 56 days was used for the Tennessee and Mississippi soil samples.

Two approaches were taken to estimate the maximum holding time (MHT) for each analyte because a standard definition for MHT has not been adopted by the Environmental Protection Agency (EPA). First, a procedure recommended by the American Society for Testing and Materials (ASTM) was modified and applied to the data base. Secondly, a procedure developed by Environmental Science and Engineering (ESE) for the analysis of a similar data base was applied. Each of these approaches resulted in different estimates of the MHT due to the application of different statistical procedures for the two definitions. Therefore, decisions concerning stability depend on the objective of the individual evaluating the environmental data.

This holding time study has shown that most soil samples are <u>not stable</u> at refrigerator temperatures for a sufficient time to allow distribution and analysis. For silt/clay soils, all VOCs except tetrachlorethane have a maximum holding time of at least 20 days if stored immediately at -20° C. The maximum holding time is 4-8 days for tetrachloroethane under the same conditions. For sandy soils, maximum holding times for only six VOCs can be recommended. The six VOCs are methylene chloride, 1,1,2-trichloroethane, bromoform, 1,1,2,2-tetrachloroethane, styrene, and o-xylene which should be stored at -70° C. The

recommended maximum holding time for these six VOCs is at least 20 days. The remaining 13 VOCs in sandy soil must either be analyzed in situ or an alternate preservation method is required. These recommended maximum holding times are conservative estimates made after reviewing MHTs for all factor combinations and summary statistics.

This report is intended to summarize the findings of the study in such a way as to allow individual decisions to be made regarding the quality of environmental data. The use of the data base may well be different for analyses conducted under RCRA, for example, than for those conducted under NPDES permit requirements. For this reason, the summary statistics for each replicate analysis is presented in Appendix A of this report.

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# 1. INTRODUCTION

During the past two decades, there has been a dramatic expansion of environmental legislation, including the Comprehensive Environmental Response, Compensation, and Liability Act; the Resource Conservation and Recovery Act; the Toxic Substances Control Act; the Clean Water Act; the Safe Drinking Water Act; the Marine Act; and, most recently, the Superfund Amendment and Reauthorization Act. One result of these regulatory measures has been a tremendous increase in the number of samples collected and distributed for analysis. One estimate is that federal, state, and local governments combined with private industry accounted for 500,000-700,000 samples in 1986. Furthermore, this number is growing at a rate of 25-40% per year [*1*]. Obviously, this has put tremendous strain on the capacity of analytical laboratories. In many cases, samples are collected at a particular site, shipped to a central distribution point, and assigned to individual laboratories on the basis of capacity. All of this is done with relatively little knowledge of the stability of the samples, and preanalytical maximum holding times (MHTs) have been established based on the best available information, much of which has been pieced together in a somewhat arbitrary fashion.

In order to provide consistent results from analytical laboratories nationwide, the United States Environmental Protection Agency (USEPA) has issued various analytical methods in the Federal Register to standardize analyses. Among the quality assurance needs in these methods is the requirement for reference samples to enable interlaboratory comparisons to be made. This work focuses on the development of a data base which allows documentation of the stability of 19 volatile organic compounds (VOC) in soil samples, with the goal for documenting MHTs under a variety of soil types and storage conditions. This data base is used to determine for which cases the current EPA specified time of 14 days is appropriate and in which cases the current time should be shortened or lengthened. Soil types investigated were deliberately selected to represent a fairly wide range of sand, clay, and silt fractions.

The generation of a data base establishing preanalytical holding times presents formidable experimental difficulties, including the need for a large number of identical sample aliquots, the need for a variety of sample matrices, and the desire for a large number of potential analytes to be present. The high vapor pressure of these analytes requires that precautions be taken to minimize losses during sample aliquot preparation. In addition, while most environmental samples contain only a few of the potential analytes, a laboratory method for the preparation of samples containing all target compounds must be developed to address a variety of naturally occuring situations. Two criteria must be met by such samples: They should be "real", i.e., they should closely simulate the composition of actual samples; they should also be of defined stability. Fortunately, an analytical method gas chromatography/mass spectrometry (GC/MS), exists which is capable of determining all volatile analytes in a single run. However, there are analytical problems related to the long-term drift of the instrument, the stability of standard compounds, and the use of a method which was originally designed for screening purposes, not for highly accurate quantitative

determinations. In this work, these limitations have been largely overcome, and the data base reported here can be used to make an accurate assessment of the stability of volatile organic compounds in environmental soil samples.

## 2. EXPERIMENTAL

This holding time study of VOCs in soils was designed to take into account as many experimental factors as possible within the limitations of budget and sample capacity. Four experimental factors were examined: volatile organic compounds, soil matrix, storage condition, and storage time. The experimental factors and their levels are presented in Table 1.

Table 1. Experimental factors for the holding time study of VOCs in soils.

| Factors | Factor Levels |
|---|---|
| VOC | 19 Organic Compounds |
| Soil Type | USATHAMA, Tennessee, Mississippi |
| Storage Condition | -70°C, -20°C, 4°C |
| Storage Time (days) | 0, 3, 7, 14, 28, 56, 111 |

### 2.1 Experimental Factors

The 19 VOCs used in this study are: bromomethane; chloroethane; methylene chloride; 1,1-dichloroethene; 1,1-dichloroethane; chloroform; carbon tetrachloride; 1,2-dichloropropane; trichloroethene; benzene; 1,1,2-trichloroethane; bromoform; 1,1,2,2-tetrachloroethane; tetrachloroethene; toluene; chlorobenzene; ethylbenzene; styrene; and o-xylene. All VOC compounds were obtained either from the United States Environmental Protection Agency (USEPA) Quality Assurance Materials Bank (Research Triangle Park, NC) [2] or were of equivalent purity and obtained commercially. Table 2 gives some physical constants for these volatile organic compounds. A nominal low concentration was selected for each VOC to represent values that may be encountered in practice.

The three soil types used for this study were a U.S. Army Toxic and Hazardous Materials Agency soil (USATHAMA)[*3*], a Captina silt loam from Roane County, Tennessee (Tennessee), and a McLaurin sandy loam from Stone County, Mississippi (Mississippi). The USATHAMA soil is THAMA reference soil which contains no semivolatile organics. The Tennessee and Mississippi soils were furnished by the Environmental Science Division of ORNL. Both soils were slightly acidic and low in organic carbons. Figure 1 graphically depicts the composition of the three soils. The Tennessee soil had a higher cation-exchange capacity and microbial respiration rate than those of the Mississippi soil. The biodegradation and microbial activity have been examined [*4,5*] in the Tennessee and Mississippi soils for 19 organic compounds. The results showed that most chemicals depressed carbon dioxide efflux in the two soils when applied at 1,000μg/g soil but this effect disappeared within a few days. These results cannot necessarily be extrapolated to microbial activity for VOCs in this study. Selected physical and chemical properties are given in Table 3 (based on Table 2 of

[5]) for the Tennessee and Mississippi soils. The USATHAMA soil is a high silt, low sand reference material employed by the Army to aid in standardized evaluations of analytical procedures [6].

Spiked Tennessee and Mississippi soils were stored at three temperatures (-70°C, -20°C, and 4°C) while spiked USATHAMA soils were stored at two temperatures (-70°C, and -20°C). Storage conditions were dictated by practicality as well as the possibility that the samples might continuously chilled during collection.

Maximum storage times were 111 days for spiked USATHAMA soils and 56 days for spiked Tennessee and Mississippi soils. The spiked samples were analyzed on equally-spaced logarithmic time intervals to estimate both short-term and long-term concentration decreases.

Table 2. Physical constants for volatile organic compounds included in the study.

| Num | Volatile Organic Compounds | Formula | m.w. (g/mole) | M.P. (°C) | B. P. (°C) | Vapor Pressure[1] (mm,Hg) | Density ($g/cm^3$) |
|---|---|---|---|---|---|---|---|
| 1 | Bromomethane | $CH_3Br$ | 94.94 | -93.6 | 3.6 | 1250 | 1.6755 |
| 2 | Chloroethane | $C_2H_5Cl$ | 64.52 | -136.4 | 12.3 | 1000 | 0.8978 |
| 3 | Methylene Chloride | $CH_3Cl$ | 50.49 | -97.1 | -24.2 | 349 | 0.9159 |
| 4 | 1,1-Dichloroethene | $C_2H_2Cl_2$ | 96.94 | -122.1 | 37 | 500 | 1.2180 |
| 5 | 1,1-Dichloroethane | $CH_3CHCl_2$ | 98.96 | -97 | 57.3 | 180 | 1.1757 |
| 6 | Chloroform | $CHCl_3$ | 119.38 | -63.5 | 61.7 | 160 | 1.4832 |
| 7 | Carbon Tetrachloride | $CCl_4$ | 153.82 | -23 | 76.5 | 90 | 1.5940 |
| 8 | 1,2-Dichloropropane | $CH_3CHClCH_2Cl$ | 112.99 | -100.4 | 96.4 | 42 | 1.1560 |
| 9 | Trichloroethene | $C_2HCl_3$ | 131.29 | -73 | 87 | 60 | 1.4642 |
| 10 | Benzene | $C_6H_6$ | 78.12 | 5.5 | 80.1 | 76 | 0.8765 |
| 11 | 1,1,2-Trichloroethane | $CH_2ClCHCl_2$ | 133.41 | -36.5 | 113.8 | 19 | 1.4397 |
| 12 | Bromoform | $CHBr_3$ | 252.75 | 8.3 | 149.5 | 6[2] | 2.8899 |
| 13 | 1,1,2,2-Tetrachloroethane | $CH_2ClCCl_3$ | 167.85 | -70.2 | 130.5 | 5 | 1.5406 |
| 14 | Tetrachloroethene | $C_2Cl_4$ | 165.83 | -19 | 121 | 18[2] | 1.6227 |
| 15 | Toluene | $C_6H_5CH_3$ | 92.15 | -95 | 110.6 | 22 | 0.8669 |
| 16 | Chlorobenzene | $C_6H_5Cl$ | 112.56 | -45.6 | 132 | 9 | 1.1058 |
| 17 | Ethylbenzene | $C_6H_5C_2H_5$ | 106.17 | -95 | 136.2 | 7 | 0.8670 |
| 18 | Styrene | $C_6H_5CHCH_2$ | 104.16 | -30.6 | 145.2 | 5 | 0.9060 |
| 19 | o-Xylene | $1,2\text{-}C_6H_4(CH_3)_2$ | 106.17 | -25.2 | 144.4 | | 0.8802 |

1. Vapor pressure measured at 20°C except where noted.
2. Vapor pressure measured at 25°C.

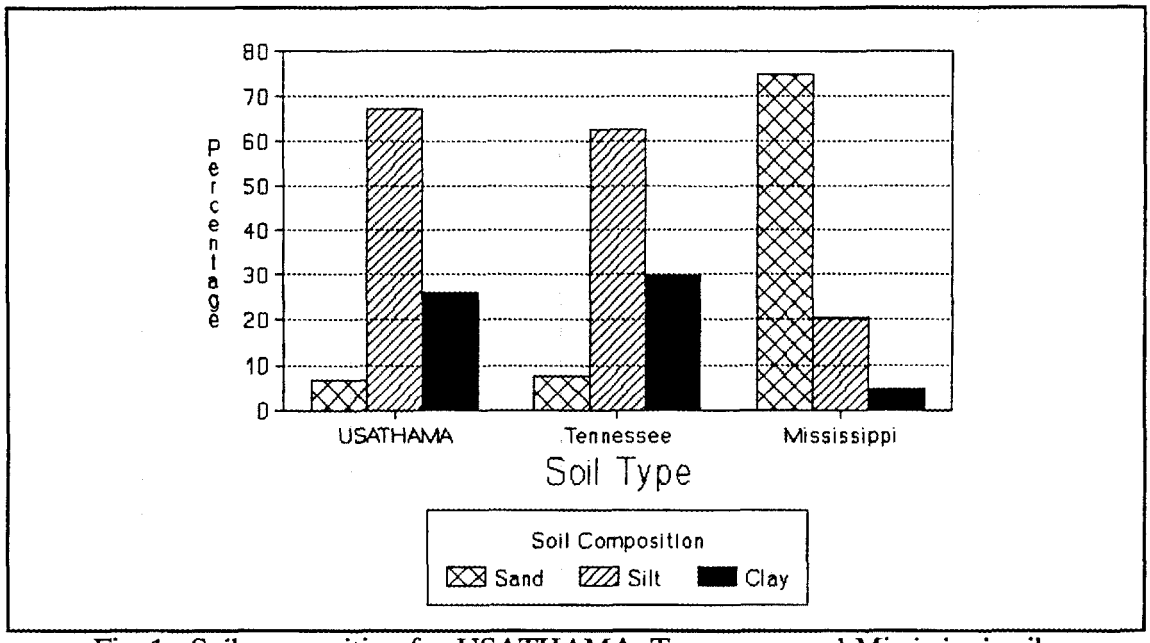


Fig. 1. Soil composition for USATHAMA, Tennessee, and Mississippi soils.

Table 3. Selected physical and chemical properties for Tennessee, Mississippi, and USATHAMA reference soils.

| Characteristics | Captina Silt Loam Roane County, Tennessee | McLaurin Sandy Loam Stone County, Mississippi | USATHAMA Reference |
|---|---|---|---|
| pH (distilled water) | 5.33 | 4.92 | 6.2 |
| pH ($CaCl_2$) | 4.97 | 4.43 | - |
| Total Organic Carbon (%) | 1.49 | 0.66 | 1.84 |
| Sand (%) | 7.7 | 74.9 | 6.7 |
| Silt (%) | 62.5 | 20.4 | 67.2 |
| Clay (%) | 29.8 | 4.7 | 26.1 |
| Nitrogen (mg/g) | 0.18 | 1.3 | 1.3 |
| Phosphorus (mg/g) | 0.04 | 0.49 | .003 |
| Cation-exchange Capacity | | | |
| $NH_4NO_3$ extraction (meq/100 g) | 1.15 | 10.15 | - |
| $NH_4CL$ extraction (meq/100 g) | 0.65 | 10.05 | - |

## 2.2 Experimental Design

The VOC holding time study for soils was designed as a complete factorial experimental design for the factors in Table 1 with four replicate measurements. This complete factorial experiment was modified during the holding time study. The modified holding time study is illustrated in Fig. 2. Prior to its use, the USATHAMA soil was dried, sterilized, and homogenized. The goal was to provide a reference medium in which bacterial action was minimized. Modifications were made to the number of storage temperatures used for USATHAMA soil samples and the maximum storage time used for Tennessee and Mississippi soil samples. Initially USATHAMA soil samples were the first samples to be analyzed. However, we realized that VOCs were escaping because of leaking screw caps. This problem was corrected by using aluminum foil coated teflon faced silicone septas. Before the holding time experiment for USATHAMA soil samples was restarted, data from Tennessee and Mississippi soil samples indicated that 4°C was not a viable storage temperature. Therefore, this storage temperature was not included in the modified holding time study because of lack of time. In addition, the results for Tennessee and Mississippi soil samples showed that maximum storage time for these soils could be truncated at 56 days.

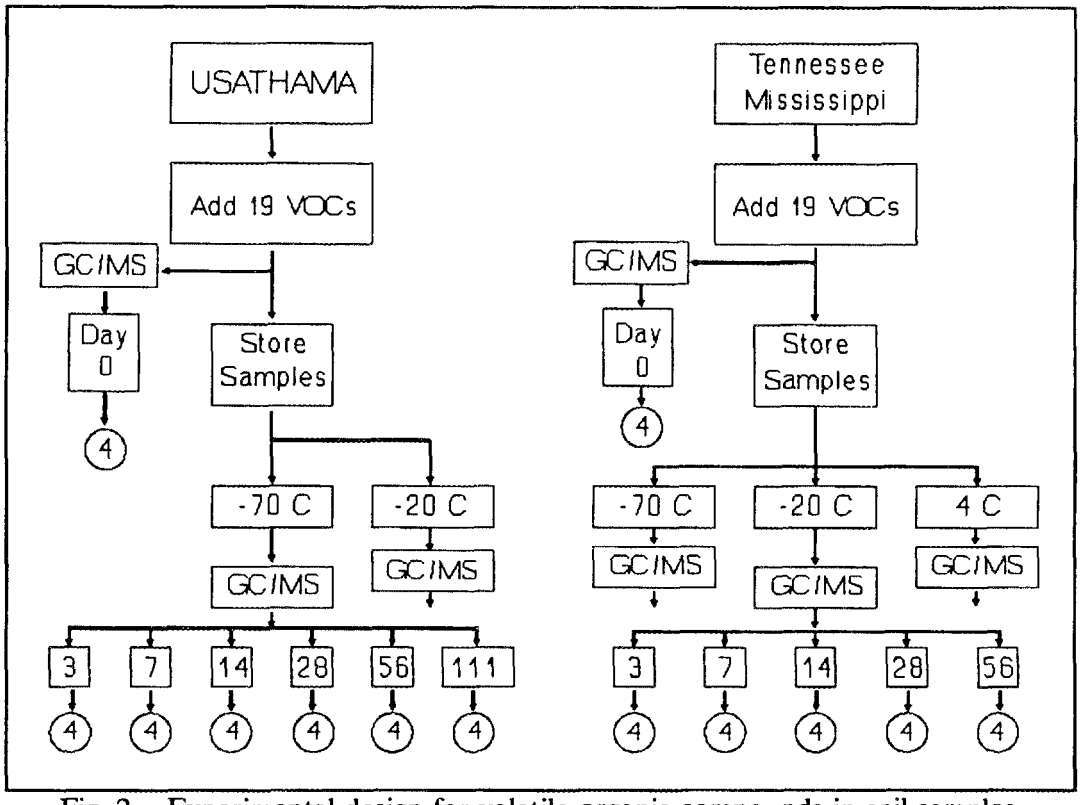


Fig. 2. Experimental design for volatile organic compounds in soil samples.

## 2.3 Analysis Procedure

Soil samples were prepared by weighing 5 g aliquots of soil into 40 mL borosilicate glass vials with aluminum foil coated teflon faced silicone septa and screw caps with holes purchased from Shamrock Glass Company. These vials were received fully assembled and precleaned according to EPA 40 CFR 136 and EPA 40 CFR 141 regulations. Three days prior to spiking with VOC stock solution, the soil samples were wetted with 1.25 mL of reagent grade water (Burdick & Jackson) and agitated with a vortex mixer for 30 s. The soil samples were then stored in the dark at room temperature. This preparation step allowed bacterial growth to come to a steady state.

Water was introduced into a 1-liter Tedlar gas sampling bag and allowed to degas for three days. This gas was then removed. One-liter Tedlar air sampling bags with dual stainless steel fittings (hose/valve fitting and replaceable septum, catalog number 231-01) were obtained from SKC, Inc. On the day that soil samples were spiked, excess air was removed from a water filled Tedlar bag. Thirty soil samples were spiked with 1.25 mL of water and vortex for about 30 s. These soil samples served as quality control blanks. Quality control blanks and soil samples were stored together in order to assess the possibility of cross contamination. The Tedlar bag with the remaining water was weighed to the nearest gram on a double pan balance using an empty Tedlar bag as a tare. Assuming 1.0 g/mL for water density, the remaining volume of water was estimated.

A VOC stock solution for each soil type was prepared by dispensing the 19 VOCs standards into the Tedlar bag with the remaining water. Target compounds were received from the aforementioned sources as methanolic solutions of 1800-2300 μg volatiles per mL methanol. Appropriate volumes of each VOC standard solution were introduced through the septum port using gas tight syringes. The contents of the Tedlar bag were mixed thoroughly by hand agitation for three minutes after which the bags were allowed to sit for thirty minutes. The VOC stock solution was then aliquotted into 40 mL vials with soil samples by gravity flow at the rate of 0.20-0.25 mL aqueous volatiles/g soil. At this point, the soil samples were 80-100% saturated with water. Each soil sample vial was sealed immediately with a Teflon-backed aluminum foil faced septum and screw cap with hole and agitated with a vortex mixer for 30 s. Soil samples were then analyzed or stored at the appropriate temperature ( -70° C, -20° C and 4° C).

Soil samples were purged directly from the 40 mL vials into the trapping system directly. A double threaded Teflon coupling machined in-house with o-ring seals were used. This was done to allow an assessment of the actual content of the vials without the complicating factor of sample weighing and transfer. All volatile organic analyses were performed by gas chromatography with mass spectrometric detection (GC/MS) according to standard EPA Contract Laboratory Program (CLP) methods [7], except for the use of daily external standards (instead of internal standards) to calculate results. Data were used without recovery or blank correction, as is customary with this method.

## 2.4 VOC Concentrations

The response data from a chemical analysis of a soil sample are the peak areas for the soil sample and the external standards. The VOC concentrations ($C_{VOC}$) were determined by using the external standard concentrations ($C_{Std}$ of 50 μg/g):

$$C_{VOC} = C_{Std} \frac{(Soil\ Sample\ Peak\ Area)}{(External\ Standard\ Peak\ Area)} . \qquad (1)$$

Summary statistics for the VOC concentrations are tabulated in Appendix A. Appendix A records the number of replicates (Num), average concentration (Avg), and standard deviation (St. Dev.) for each day at the different level of the experimental factors. Note that the standard deviation is the standard deviation of the Num replicate measurements and not the standard deviation of the average.

In addition, plots of the average VOC concentrations versus Time (Days) are given in the appendices for each level of the experimental factors. The average VOC concentrations are connected with a line to aid in viewing the graph and does not represent a least squares fit. The Time (Days) axis is on a logarithmic scale (base 10) which assists in distinguishing both the short-term VOC concentrations and long-term VOC concentrations. The logarithmic axis may cause distortions when viewing the graphs to judge VOC degradation. For example, Fig. 3 shows the average 1,1,2,2-tetrachloroethane concentrations for Tennessee soil samples stored at 4°C. Figure 3 uses both a linear and logarithmic Time (Days) axis which shows the effect of axis scaling. The logarithmic Time (Days) axis emphasizes the short-term VOC concentrations while the linear Time (Days) axis emphasizes the long-term VOC concentrations.

## 2.5 Outlier Measurements

The total number of chemical analyses used to determine maximum holding times were 3,382 analyses. Although 3,382 chemical analyses were performed, about 30% (i.e., 1,016 analyses) of the data were considered outliers and not used to estimate the maximum holding time values. Potential outliers [8] were first identified by comparing the changes in the standard deviations of neighboring time points for each matrix type and storage condition. Additional potential outliers were also identified by their large (e.g., > 2.5) studentized residuals for the zero-order and first-order regressions of concentrations vs storage times. Studentized residuals are the residuals (observed - predicted) divided by their standard deviations. An identified outlier value was marked in the data set not to be used for estimating maximum holding times after reexamining the raw data from the corresponding GC/MS chemical analysis. Chemical judgement for marking an identified outlier was based on (1) an analysis that resulted in an unusually low or high concentration due to contaminant peak interference of poor separated peaks, or (2) an analysis corresponded to an incorrect analysis of a reference standard, or (3) an analysis that had been compromised by procedural problems (e.g., incorrect spiking concentration, instrument problems, sample bottles not properly filled, data entry errors, etc.). A potential outlier found by the statistical procedure was not necessarily set aside until after considering the chemical analysis.

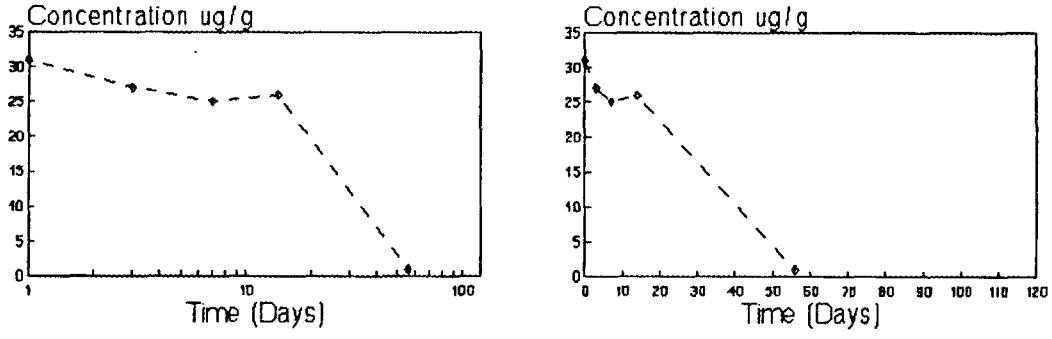


Fig. 3. Average concentrations for 1,1,2,2-tetrachloroethane in Tennessee soil samples stored at 4°C using both logarithm and linear scaling for the Time (Days) axis.

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## 3. RESULTS AND DISCUSSION

Sample handing procedures employed in this study were used primarily because more common procedures were less effective. In particular, earlier experiments had demonstrated that it was exceedingly difficult to spike a large batch of soil, and distribute the soil into vials without substantial losses of VOC's. Instead, we found spiking of individual vials to be much more effectively. Secondly, we found that use of conventional Teflon-coated septa resutled in unacceptably high losses of VOC's. These were replaced with aluminum foil lined Teflon backed septa.

The results of this study are estimated maximum holding times (MHTs), which are the maximum times a sample can be held prior to analysis before sufficient degradation occurs to render the sample unreliable. It is the establishment of criteria which defines "sufficient degradation" that can be a subjective process. Two statistical definitions were used to determine MHT criteria. The first definition was specified by the American Society for Testing and Materials [*9*, ASTM MHT]. The second definition was specified by Environmental Science and Engineering, Inc. [*10*, ESE MHT] for a holding time study conducted in cooperation with U.S. Environmental Protection Agency. The precise statistical details for these two definitions are given in Sect. 4. Both definitions are based on an approximating model for predicting concentration with time. The ASTM defines the MHT as the time the predicted concentration falls below the lower two-sided 99% confidence interval on the initial concentration. The ESE defines the MHT as the time the one-sided 90% confidence interval on the predicted concentration falls below a 10% change in the initial concentration. The main difference between the two definitions is the method of placing a lower bound on the initial concentration. The ASTM MHTs are usually longer than the ESE MHTs because decreasing the initial concentration by 10% is usually a smaller reduction than the lower two-sided 99% confidence limit. The ASTM MHT definition is recommended for analytical methods with precision such that the lower bound on 99% confidence limit for an analyte concentration is less than 10% of the initial analyte concentration. Otherwise, using the ESE MHT definition would be more conservative.

Tables 4 and 5 give the estimated MHTs determined using the two statistical definitions. Maximum holding times depend on the VOC, storage temperature, and soil matrix. Most MHTs results for -20°C storage temperature have longer or equivalent (e.g., ±2 days) MHTs than either -70°C or 4°C storage temperatures. Exceptions to this general rule are for bromoform in USATHAMA soil stored at -70°C; methylene chloride, 1,1,2-trichloroethane (i.e., ASTM MHT only), styrene, and o-xylene in Mississippi soil stored at -70°C. A storage temperature of 4°C is inadequate for all 19 VOCs in Tennessee and Mississippi soils -- except perhaps methylene chloride (ASTM MHT = 14 days) in Tennessee soil. For bromomethane, none of the three storage temperature are adequate for Tennessee and Mississippi soils.

Figure 4 compares MHTs by the three soil matrices using ASTM MHTs. Maximum holding times were truncated at 56 days for USATHAMA soil samples and the best storage temperature (i.e., -20°C for most cases) was used for each VOC. This figure shows that

ASTM MHTs for USATHAMA soil are greater than or equivalent to ASTM MHTs for Tennessee soil for all VOCs except tetrachlorethane, ethylbenzene, and o-xylene. The ASTM MHTs for USATHAMA soil are greater than or equivalent to ASTM MHTs for Mississippi soil for all VOCs. Comparing ASTM MHTs for Tennessee soil and Mississippi soil, Fig. 4 shows greater of equivalent ASTM MHTs for Tennessee soil for all VOCs except bromoform, and 1,1,2,2-tetrachloroethane. This result may occur because Tennessee soil has a similar composition to USATHAMA soil (e.g., 7% sand) while Mississippi soil has a higher percentage of sand (e.g., 75% sand). If the ESE MHT definition is used, the only change in the comparisons is that ESE MHTs for USATHAMA soil is greater than or equivalent to ESE MHTs for Tennessee soil for all VOCs. For Mississippi soil, only seven/six VOCs have ASTM/ESE MHTs greater than five days for all storage temperatures.

Table 4. ASTM MHTs in days for volatile organic compounds in three soils.

| | Volatile Organic | USATHAMA[1] | | Tennessee[2] | | | Mississippi[2] | | |
|---|---|---|---|---|---|---|---|---|---|
| Num | Compounds | -70°C | -20°C | -70°C | -20°C | 4°C | -70°C | -20°C | 4°C |
| 1 | Bromomethane | 2 | 108 | 0 | 1 | 0 | 0 | 3 | 0 |
| 2 | Chloroethane | 4 | 24 | 0 | 23 | 7 | 0 | 3 | 0 |
| 3 | Methylene Chloride | 24 | 111 | 56 | 56 | 14 | 56 | 0 | 2 |
| 4 | 1,1-Dichloroethene | 2 | 111 | 0 | 18 | 4 | 0 | 3 | 0 |
| 5 | 1,1-Dichloroethane | 2 | 111 | 0 | 20 | 6 | 0 | 2 | 1 |
| 6 | Chloroform | 2 | 111 | 0 | 47 | 2 | 0 | 2 | 2 |
| 7 | Carbon Tetrachloride | 2 | 111 | 0 | 18 | 8 | 0 | 3 | 0 |
| 8 | 1,2-Dichloropropane | 3 | 111 | 0 | 56 | 6 | 2 | 2 | 2 |
| 9 | Trichloroethene | 2 | 65 | 0 | 46 | 0 | 0 | 3 | 0 |
| 10 | Benzene | 2 | 81 | 0 | 28 | 0 | 0 | 3 | 2 |
| 11 | 1,1,2-Trichloroethane | 42 | 111 | 56 | 56 | 6 | 16 | 5 | 2 |
| 12 | Bromoform | 111 | 69 | 24 | 40 | 6 | 56 | 56 | 2 |
| 13 | 1,1,2,2-Tetrachloroethane | 75 | 111 | 26 | 27 | 5 | 56 | 56 | 4 |
| 14 | Tetrachloroethane | 2 | 8 | 0 | 22 | 0 | 0 | 3 | 2 |
| 15 | Toluene | 2 | 76 | 1 | 44 | 0 | 0 | 16 | 15 |
| 16 | Chlorobenzene | 4 | 98 | 56 | 56 | 0 | 5 | 3 | 2 |
| 17 | Ethylbenzene | 8 | 28 | 0 | 37 | 2 | 3 | 4 | 2 |
| 18 | Styrene | 63 | 111 | 56 | 56 | 1 | 56 | 26 | 3 |
| 19 | o-Xylene | 2 | 47 | 56 | 56 | 2 | 19 | 11 | 2 |

1. Maximum experimental time was 111 days.
2. Maximum experimental time was 56 days.

Table 5. ESE MHTs in days for volatile organic compounds in three soils.

| Num | Volatile Organic Compounds | USATHAMA[1] -70°C | USATHAMA[1] -20°C | Tennessee[2] -70°C | Tennessee[2] -20°C | Tennessee[2] 4°C | Mississippi[2] -70°C | Mississippi[2] -20°C | Mississippi[2] 4°C |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Bromomethane | 1 | 111 | 1 | 1 | 1 | 1 | 2 | 1 |
| 2 | Chloroethane | 2 | 18 | 1 | 4 | 3 | 1 | 2 | 1 |
| 3 | Methylene Chloride | 21 | 111 | 56 | 56 | 1 | 56 | 2 | 1 |
| 4 | 1,1-Dichloroethene | 1 | 111 | 1 | 3 | 2 | 1 | 2 | 1 |
| 5 | 1,1-Dichloroethane | 1 | 111 | 1 | 4 | 3 | 1 | 1 | 1 |
| 6 | Chloroform | 1 | 111 | 1 | 25 | 1 | 1 | 1 | 1 |
| 7 | Carbon Tetrachloride | 1 | 111 | 1 | 3 | 3 | 1 | 2 | 1 |
| 8 | 1,2-Dichloropropane | 2 | 111 | 1 | 43 | 3 | 1 | 1 | 1 |
| 9 | Trichloroethene | 1 | 52 | 1 | 39 | 1 | 1 | 2 | 1 |
| 10 | Benzene | 1 | 68 | 1 | 5 | 1 | 1 | 1 | 1 |
| 11 | 1,1,2-Trichloroethane | 40 | 111 | 56 | 56 | 3 | 4 | 3 | 1 |
| 12 | Bromoform | 111 | 56 | 10 | 9 | 3 | 56 | 56 | 1 |
| 13 | 1,1,2,2-Tetrachloroethane | 111 | 111 | 12 | 5 | 4 | 56 | 56 | 1 |
| 14 | Tetrachloroethane | 1 | 4 | 1 | 4 | 1 | 1 | 2 | 1 |
| 15 | Toluene | 2 | 46 | 1 | 41 | 1 | 3 | 2 | 2 |
| 16 | Chlorobenzene | 3 | 111 | 56 | 56 | 1 | 1 | 2 | 1 |
| 17 | Ethylbenzene | 3 | 16 | 1 | 8 | 1 | 1 | 1 | 1 |
| 18 | Styrene | 111 | 111 | 56 | 56 | 1 | 56 | 5 | 1 |
| 19 | o-Xylene | 2 | 62 | 56 | 56 | 1 | 13 | 2 | 1 |

1. Maximum experimental time was 111 days.
2. Maximum experimental time was 56 days.

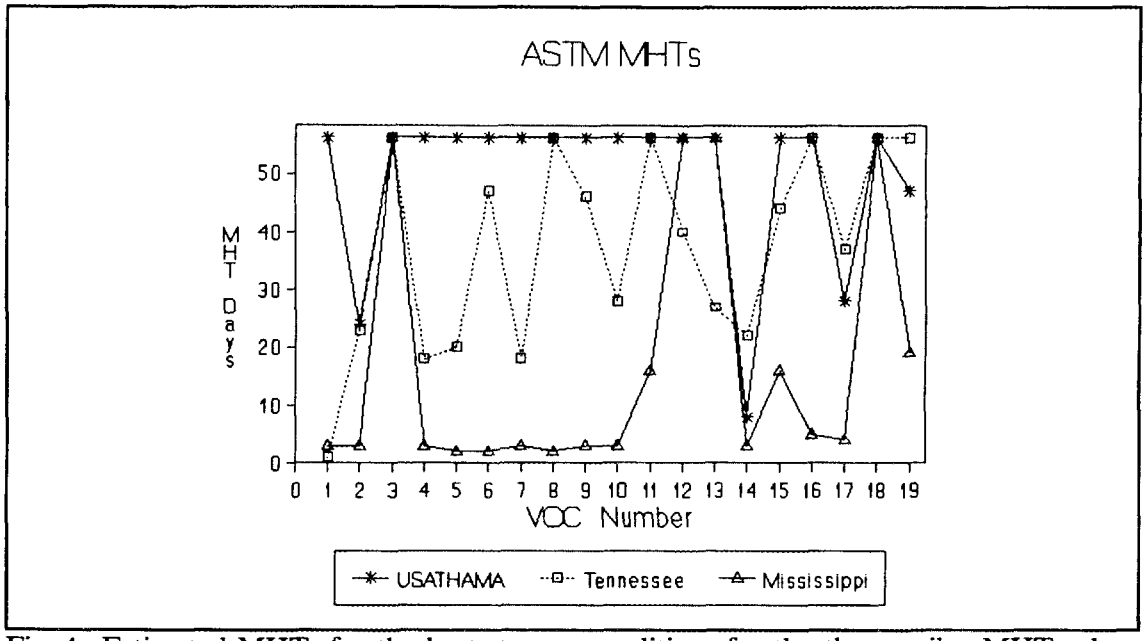


Fig. 4. Estimated MHTs for the best storage conditions for the three soils. MHT values for USATHAMA soil samples are truncated at 56 days.

Integrating the results of this study into actual field operations is not straightforward. The fact that the pre-sterilized USATHAMA soil exhibited reasonably long MHT's at one storage condition suggests that microbial action is extremely important to contaminant stability in environmental samples. Of course, it is impractical to sterilize real samples without loss of volatiles. The highly sandy soil (Mississippi) had the shortest MHT's. For the two soils with higher organic, silt, and clay content, the best overall storage conditions appear to be -20° C. The typical storage condition of 4° C, commonly used in the United States, appears to be of minimal utility for maintaining contaminant levels. A storage condition of -70° C appears to be too low, although their were insufficient data generated in this study to provide a definitive reason. Producing a -20° C condition under field conditions would probably most easily be achieved using a salt brine and ice mixture. Maintenance of such a mixture would seem more problematic during shipment. However, the data shows that -20° C is not the best storage condition for all contaminants and all soils. It would appear that selection of storage condition will be dependent on soil and contaminant type.

These results add to the increasing body of evidence which indicates that current sampling handling and storage practices result in unacceptably high losses of VOC's prior to analysis. There is a growing sense that the sample must be acquired in a manner which disturbs the soil to a minimum extent, that the sample must be transferred to a container which is immediately sealed, and that the container must not be opened or penetrated until after the analysis is underway. Recently, a team of scientists assembled by the EPA has reported on apparati and methods for doing just that [*11*]. There is now commercially available sub-coring devices which permit the removal of a small soil plug from a larger core, and

deposition of the plug in a 40 mL VOA vial, as well as a specialized cap for such vials, which permits the vial to remain tightly sealed until a small Teflon ball seal is pushed out of the way as the vail is loaded onto a purge and trap device. Alternatively, the sample can be immersed in methanol. A recent study [*12*] has shown that approach to be very promising, in terms of minimizing losses of VOC's. However, analysis of an aliquot of the methanol extract via purge and trap GC/MS has the advantage of inherently higher limits of detection.

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# 4. DETERMINATION OF MAXIMUM PRE-ANALYTICAL HOLDING TIMES BY STATISTICAL METHODS

The purpose of the work described herein was to determine the maximum length of time which a sample can be held without processing prior to analysis for a specific contaminant. One obvious criterion for "how long is too long" is the point in time where the concentration of the target constituent begins to fall outside the range of acceptability limits for the recovery of a matrix spike. However, the EPA CLP matrix spike recovery limit range can be so large that unacceptably large changes in target analyte concentration can occur without exceeding the range limits. Therefore, another approach was developed which established more stringent criteria for the concept of a pre-analytical holding time. These criteria were defined in terms of the time at which the measured sample concentration falls outside confidence interval boundaries. These boundaries were calculated from a mathematical model that approximated the change in sample concentration with time. The two primary definitions used for the MHT criteria were those by the American Society for Testing and Materials (ASTM) and by Environmental Science and Engineering, Inc. (ESE), the latter developed in cooperation with EPA's Environmental Monitoring and Support Laboratory.

## 4.1 Approximating Models

Maximum holding time (MHT) was defined as the maximum period of time during which a properly collected and stored sample can be stored before some degradation of the analyte occurs in the sample matrix. Calculating the MHT depends on the approximating model used to predict the expected concentration for any time during the experimental period (i.e., 111 days for USATHAMA soil, and 56 days for Tennessee and Mississippi soils). A major problem of estimating an approximating model for VOC concentrations in soils is the large variability of the data. This variability is reflected in the number of chemical analyses that were considered outliers (e.g., 30% in soils as compared to 5-11% in water [*13*]). These outlier data values were not used in data analysis only after careful statistical and chemical considerations (see Sect. 2.5). The remaining data set still has considerable variability as indicated by the standard deviations in Tables A1-A19. The variability problem supports the need for additional research regarding preservation and analysis techniques for VOCs in soil samples.

Analyte concentrations as a function of time were initially approximated by a zero-order kinetic model. A zero-order kinetic model represents a constant change in the expected concentration with time. Zero-order approximating models were estimated for the 152 cases examined by fitting least-square lines to the concentration measurements as a function of time. For many cases, the zero-order kinetic model was not an adequate approximating model. A first-order kinetic model (e.g. concentration is an exponential function of time) did not show any significant improvement for fitting the data because of data variability.

The failure of zero-order and first-order kinetic models to adequately fit 96 cases is because concentrations decreased too rapidly or concentrations were stable for a number of days and then decreased rapidly. Empirical models were investigated to approximate these cases.

Figure 5 shows the five empirical models used to approximate VOCs concentration as a function of time. Table 6 defines the mathematical form of four approximating models; zero-order (56 cases), first-order (0 cases), log-term (43 cases), and inverse-term (34 cases). These empirical models were selected based on their shape and on their derivative (i.e., rate of concentration change). The log-term and inverse-term models have derivatives that can decrease rapidly with time. Coefficients of the models in Table 6 are estimated by the method of least-squares from the VOCs concentration data. The estimated coefficients were calculated by PROC REG in the SAS computer programming language [*14*].

The fifth approximating model is the cubic spline which accounts for an initial stable concentration followed by a rapid decrease in concentration. The cubic spline has the mathematical form as a function of time, f(D) with D = day, is defined as:

$$f(D) = \begin{cases} C_0 & \text{if } D \le D_0 \\ a + bD + cD^2 + dD^3 & \text{if } D_0 \le D \le D_1 \\ C_1 & \text{if } D \ge D_1 \end{cases}$$

The continuity condition and the initial and final concentration conditions place two restrictions on f(D):

1. $f(D_0) = C_0$ and $f(D_1) = C_1$.

2. $f'(D_0) = 0$ and $f'(D_1) = 0$, where f′ is the derivative with respect to $D_0$ and $D_1$, respectively.

Using these two restrictions for the cubic spline, the coefficients a, b, c, and d can be determined in terms of $D_0$ and $D_1$.

$$a = (C_0H_1 - C_1H_0)/(H_1 - H_0) \qquad c = -1.5(C_1 - C_0)(D_0 + D_1)/(H_1 - H_0)$$

$$b = 3(C_1 - C_0)D_0D_1/(H_1 - H_0) \qquad d = (C_1 - C_0)/(H_1 - H_0)$$

where

$$H_0 = 0.5D_0^2(3D_1 - D_0) \text{ and } H_1 = 0.5D_1^2(3D_0 - D_1).$$

The estimates of the parameters $D_0$ and $D_1$ for the cubic splines are calculated by the method of non-linear least squares. The cubic splines were estimated for 19 cases. The estimated parameters were calculated with the non-linear procedure PROC NLIN with METHOD=MARQUARDT in the SAS computer programming language [*14*].

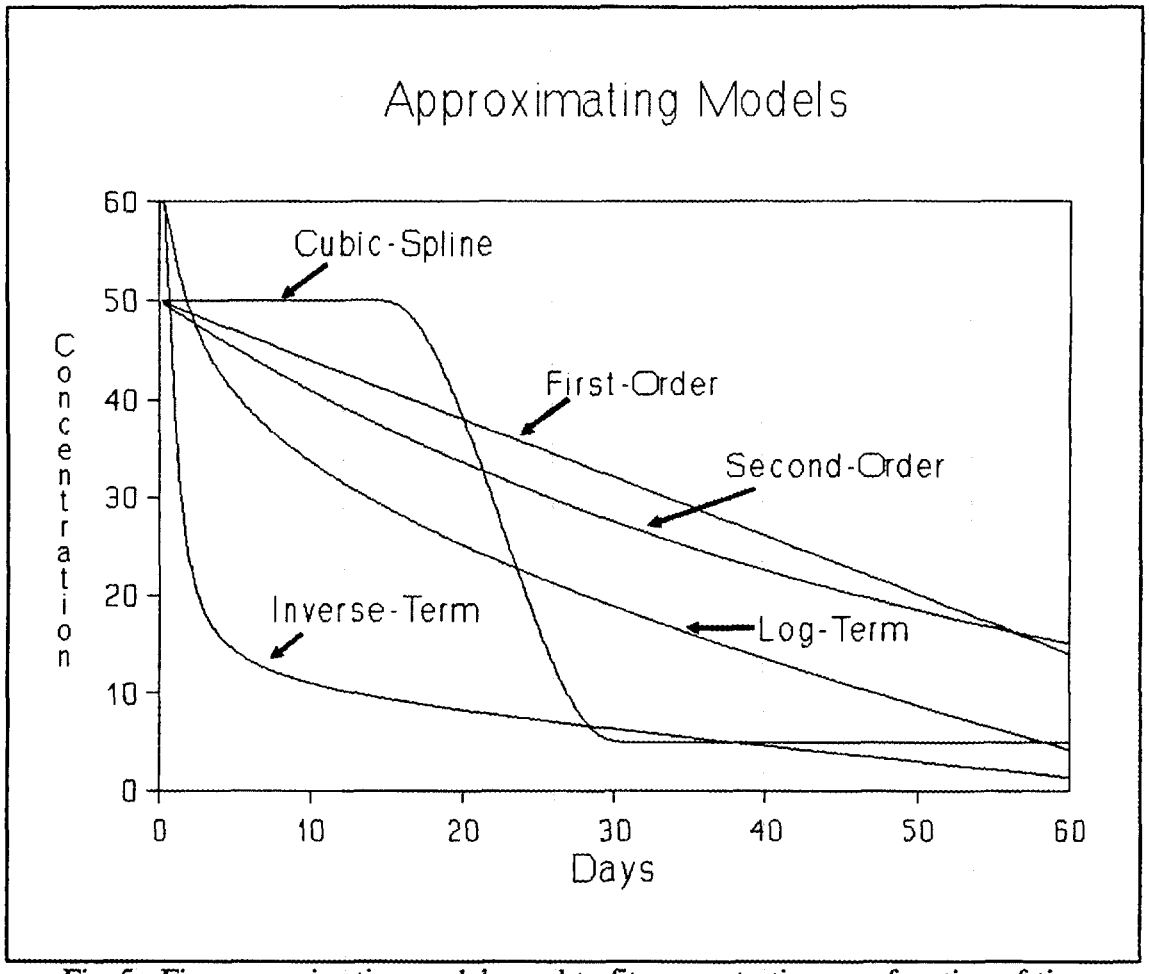


Fig. 5. Five approximating models used to fit concentration as a function of time.

Table 6. Models and their derivatives used to approximate special cases of VOCs in soil samples.

| Model | Equation | Derivative |
|---|---|---|
| Zero-Order | $C = C_0 + B(day)$ | $dC/d(day) = B$ |
| First-Order | $C = C_0 exp[B(day)]$ | $dC/d(day) = BC_0 exp[B(day)]$ |
| Log-Term | $C = C_0 + B(day) + A\ ln(day)$ | $dC/d(day) = B + A/(day)$ |
| Inverse-Term | $C = C_0 + B(day) + A/(day)$ | $dC/d(day) = B - A/(day)^2$ |

Coefficients for the approximating models were estimated by the method of least squares [*15*]. This method estimates the model coefficients that minimizes the squared difference between the "N" measured concentrations and the predicted concentrations (i.e., sum-of-squares for error = SSE)

$$SSE = \sum_{j=1}^{N} (Measured - Predicted)^2 \quad .$$

An approximating model was chosen by evaluating the mean square error (i.e., MSE = SSE/DF) for all five empirical models for a particular VOC, soil, and storage temperature combination. The MSE is the sum-of-squares for error divided by the number of degrees of freedom (i.e., DF = number of data points - number of estimated coefficients). Usually the model with the smallest MSE was chosen as the approximating model. However, all MSEs were compared to the MSE for the zero-order model. The zero-order model was chosen if no significant difference between the MSEs was detected at the 5% significant level by an F-test and if the graphical representation was visually judged to be best approximated by a line. Table 7 gives the approximating models used to represent concentration as a function of time.

Concentration values that did not decrease significantly with time can be approximated with the average of all measured concentrations during the experiment. These cases have approximating lines with slopes equal to zero (e.g., nonsignificant slopes). The MHTs are estimated to be the maximum time of the experiment. Concentrations that deviate from the average indicate a decomposition with time. These deviations are accounted for by one of the five approximating models. A measure of "goodness-of-fit" is the percentage of the sum-of-squares of the deviations of the measured concentrations from the average concentration (i.e., total sum-of-squares, SST) attributed to the approximating model. An approximating model could account for a maximum of 100% of SST if only one concentration was measured on each day of the experiment. For example, this percentage is often reported as $\%R^2$ or the multiple correlation coefficient for regression analyses. For replicate concentration measurements on each day, an approximating model can at best fit the average concentration at each replicate. The maximum percentage of SST is usually less than 100% that can be attributed to the approximating model. Table 8 shows the percentage of SST attributed to the approximating model relative to the maximum possible percentage that can be obtained. For example, if an approximating model accounts for 85% of SST and the maximum possible percentage is 90% of SST, Table 8 reports 94% [i.e., 94% = 100% (85/90)]. These percentages are not reported for line models with nonsignificant slopes.

Table 7. Approximating models used to estimate MHTs.

| Num | Volatile Organic Compounds | USATHAMA | | Tennessee | | | Mississippi | | |
|---|---|---|---|---|---|---|---|---|---|
| | | -70°C | -20°C | -70°C | -20°C | 4°C | -70°C | -20°C | 4°C |
| 1 | Bromomethane | Log | Zero | Inverse | Inverse | Inverse | Inverse | Spline | Inverse |
| 2 | Chloroethane | Spline | Spline | Inverse | Zero | Zero | Inverse | Spline | Inverse |
| 3 | Methylene Chloride | Spline | Zero | Zero | Zero | Zero | Zero | Log | Inverse |
| 4 | 1,1-Dichloroethene | Log | Zero | Inverse | Zero | Log | Inverse | Spline | Inverse |
| 5 | 1,1-Dichloroethane | Log | Zero | Inverse | Zero | Zero | Inverse | Log | Log |
| 6 | Chloroform | Log | Zero | Inverse | Spline | Inverse | Inverse | Log | Log |
| 7 | Carbon Tetrachloride | Log | Zero | Inverse | Zero | Zero | Inverse | Spline | Inverse |
| 8 | 1,2-Dichloropropane | Log | Zero | Inverse | Spline | Zero | Log | Log | Log |
| 9 | Trichloroethene | Log | Zero | Inverse | Spline | Inverse | Inverse | Spline | Inverse |
| 10 | Benzene | Log | Zero | Inverse | Zero | Inverse | Inverse | Log | Log |
| 11 | 1,1,2-Trichloroethane | Zero | Zero | Zero | Zero | Zero | Spline | Log | Log |
| 12 | Bromoform | Zero | Zero | Zero | Zero | Zero | Zero | Zero | Log |
| 13 | 1,1,2,2-Tetrachloroethane | Zero | Zero | Zero | Zero | Zero | Zero | Zero | Log |
| 14 | Tetrachloroethene | Log | Log | Inverse | Zero | Inverse | Inverse | Spline | Log |
| 15 | Toluene | Log | Zero | Inverse | Spline | Inverse | Spline | Log | Log |
| 16 | Chlorobenzene | Spline | Zero | Zero | Zero | Inverse | Log | Log | Log |
| 17 | Ethylbenzene | Spline | Spline | Inverse | Zero | Log | Log | Log | Log |
| 18 | Styrene | Zero | Zero | Zero | Zero | Log | Zero | Zero | Log |
| 19 | o-Xylene | Log | Zero | Zero | Zero | Log | Spline | Log | Log |

Table 8. Percentage of the total sum-of-squares attributed to the approximating model relative to the maximum possible percentage that can be obtained.

| Num | Volatile Organic Compounds | USATHAMA | | Tennessee | | | Mississippi | | |
|---|---|---|---|---|---|---|---|---|---|
| | | -70°C | -20°C | -70°C | -20°C | 4°C | -70°C | -20°C | 4°C |
| 1 | Bromomethane | 88.3 | (a) | 97.7 | 88.7 | 99.8 | 96.1 | 97.4 | 99.7 |
| 2 | Chloroethane | 95.2 | 63.5 | 99.1 | 56.5 | 97.5 | 98.1 | 99.5 | 99.7 |
| 3 | Methylene Chloride | 85.1 | (a) | (a) | (a) | 87.3 | (a) | 86.6 | 95.9 |
| 4 | 1,1-Dichloroethene | 98.4 | (a) | 99.3 | 70.2 | 99.5 | 99.3 | 99.9 | 99.9 |
| 5 | 1,1-Dichloroethane | 99.7 | (a) | 99.4 | 65.5 | 98.3 | 95.9 | 97.4 | 99.6 |
| 6 | Chloroform | 95.6 | (a) | 99.3 | 70.9 | 99.4 | 92.6 | 95.3 | 99.9 |
| 7 | Carbon Tetrachloride | 98.5 | (a) | 99.0 | 70.8 | 97.9 | 99.4 | 99.7 | 99.0 |
| 8 | 1,2-Dichloropropane | 96.0 | (a) | 98.5 | 48.7 | 98.4 | 90.7 | 91.9 | 99.9 |
| 9 | Trichloroethene | 95.2 | 30.2 | 98.8 | 81.5 | 99.3 | 97.3 | 97.9 | 99.4 |
| 10 | Benzene | 96.5 | (a) | 98.9 | 51.4 | 99.8 | 95.7 | 95.4 | 99.4 |
| 11 | 1,1,2-Trichloroethane | 32.8 | (a) | (a) | (a) | 95.4 | 68.7 | 61.8 | 98.9 |
| 12 | Bromoform | (a) | 17.3 | 52.3 | 35.4 | 94.9 | (a) | (a) | 99.1 |
| 13 | 1,1,2,2-Tetrachloroethane | (a) | (a) | 54.3 | 61.6 | 97.1 | (a) | (a) | 99.5 |
| 14 | Tetrachloroethene | 97.3 | 80.5 | 98.8 | 66.3 | 99.3 | 98.5 | 98.4 | 99.3 |
| 15 | Toluene | 95.5 | 33.3 | 85.2 | 90.9 | 98.4 | 82.6 | 91.1 | 93.0 |
| 16 | Chlorobenzene | 96.6 | (a) | (a) | (a) | 99.1 | 73.1 | 82.8 | 98.6 |
| 17 | Ethylbenzene | 97.9 | 77.0 | 94.2 | 36.4 | 99.9 | 87.5 | 95.9 | 99.1 |
| 18 | Styrene | (a) | (a) | (a) | (a) | 99.8 | (a) | 81.7 | 97.8 |
| 19 | o-Xylene | 89.2 | 43.1 | (a) | (a) | 95.4 | 85.5 | 97.4 | 97.9 |

(a) slope of zero-order model not significantly different than zero (5% significance level).

## 4.2 MHT Definitions

The ASTM and the ESE definitions were used to calculate the MHT criteria after choosing the approximating model for the expected concentrations. The ASTM definition [9] is described in volume 11.02 of the 1991 Annual Book of ASTM Standards. For the purposes of this study, the ASTM definition was applied to the zero-order model as follows:

**ASTM**

1. Fit the appropriate approximating model to the holding time data by the method of least squares.

2. Estimate the intercept, $C_0$, and its standard deviation, $S_0$.

3. Calculate the two-sided 99% confidence interval on the intercept (i.e. $C_0 \pm t(df,0.005)S_0$, where $t(df,0.005)$ is the 99.5 percentile point of the t-distribution with df = degrees of freedom and $S_0$ is the standard deviation of the intercept).

4. The ASTM MHT is the time at which the approximating model is equal to the value of the lower confidence limit on the intercept if the estimated slope is negative. For positive estimated slopes, the MHT is the time at which the approximating model is equal to the value of the upper confidence limit on the intercept. MHT can be calculated for an approximating line model by:

$$MHT = t(df,0.005)S_0/|B|,$$

where

$|B|$ = absolute value of the slope.

5. Estimated MHT values greater than the time of the experimental study are set equal to maximum storage time (e.g., 56 days or 111 days).

This working definition differs slightly from the exact ASTM definition because this holding time study did not employ the same experimental design as recommended by ASTM. The differences between the two definitions are that confidence intervals on the intercepts are used rather than the confidence intervals on the mean of ten replicate concentrations measured on day 0 (it was impractical to make ten replicate analyses within one day). Also, the intercept and slope of the approximating models were estimated by the method of least squares rather than the "best graphical fit" of the average concentration for each day. Figure 6 illustrates the ASTM method for estimating the MHT for ethylbenzene in Tennessee soil at -20° C storage temperature.

For approximating models other than a line, the same procedure was adapted for estimating ASTM MHTs. The appropriate intercept confidence interval was estimated for time = 0 depending on the approximating model. Estimated ASTM MHTs were found by solving for the time when the intercept confidence interval and approximating model are equal. For the cubic spline, ASTM MHTs are estimated by iteratively calculating the time the intercept confidence interval and cubic spline intersect.

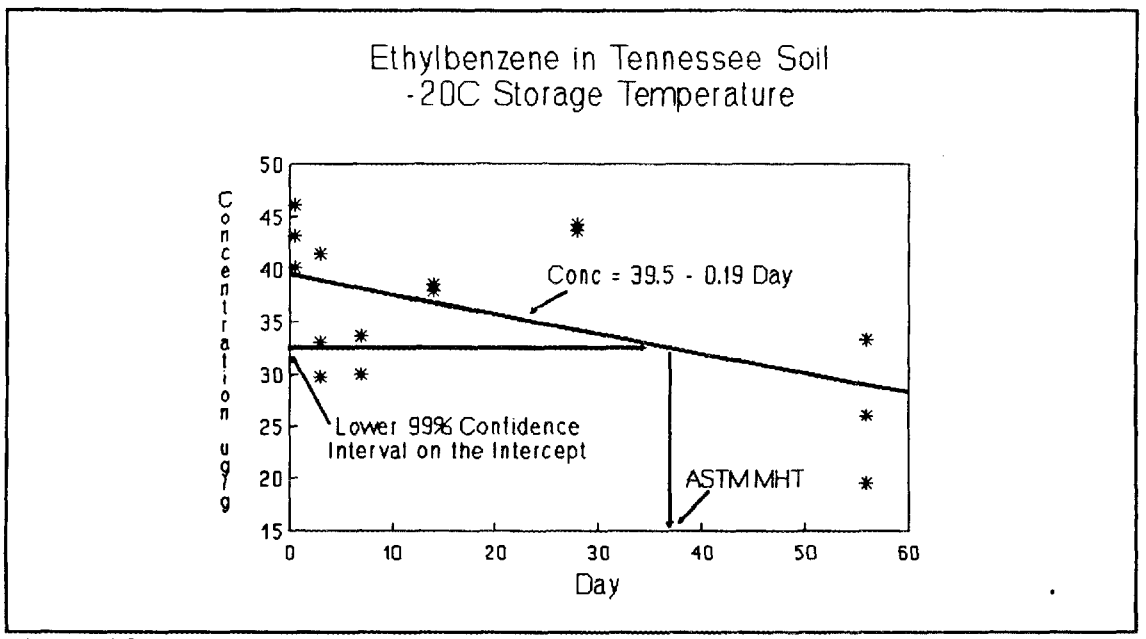


Fig. 6. ASTM method for estimating maximum holding time from data (stars) for ethylbenzene in Tennessee soil at -20° C storage temperature.

A second definition for MHT was used in holding time studies on inorganic analytes conducted by Environmental Science and Engineering, Inc. (ESE) in cooperation with EPA's Environmental Monitoring and Support Laboratory [*10*]. The ESE definition is based on intersecting a 10% change in the intercept with a one-sided 90% confidence interval on the predicted concentration. Figure 7 portrays the ESE method for estimating maximum holding times for the same case examined in Fig 6. For this holding time study, the ESE definition of MHT was applied to a zero-order model as follows:

## ESE

1. Fit the zero-order model to the holding time data by the method of least squares.

2. Test that the slope is significantly different than zero with a two-sided t-test at 10% significance level (e.g., $|B| \geq t(df,0.05)S_1$, where t(df,0.05) is the 95 percentile point of the t-distribution with df = degrees of freedom and $S_1$ is the standard deviation of the slope). If the slope is not significantly different than zero then set MHT equal to the maximum storage time (e.g., 56 days or 111 days).

3. Construct a ±10% interval about the intercept [e.g., (0.9C0, 1.1C0)]. Test that the 10% change is outside the 90% confidence interval on $C_0$ with a two-sided t-test at the 10% significance level [e.g., $0.1C_0 \geq t(df,0.05)S_0$ for zero-order, where t(df,0.05) is the 95 percentile point of the t-distribution with df = degrees of freedom and $S_0$ is the standard deviation of the intercept].

4. If a 10% change is not outside the 90% confidence interval, calculate the concentration change (i.e., $C_0 \pm KC_0$) that does occur outside the limits

$$K = t(df,0.05)S_0/C_0 \ .$$

If K > 0.15, the zero-order model is usually not appropriate for estimating the expected concentrations. The MHT can't be estimated with this model and other approximating models must be investigated. However, large variability in the data may also cause K > 0.15.

5. Calculate the critical time ($C_T$) when the predicted concentration line intersects the significant concentration change ($0.10 \leq K \leq 0.15$) by

$$C_T = KC_0/|B| \ .$$

6. The MHT is defined as the one-sided lower 90% confidence interval on CT and can be calculated by

$$MHT = C_T - t(df,0.10)[Var(C_T)]^{1/2},$$

where,

t(df,0.10) = the 90 percentile point of the t-distribution, and

$Var(C_T)$ = the variance of $C_T$ approximated by

$$Var(C_T) = C_T^2[Var(C_0)/C_0^2 + Var(B)/B^2 - 2Cov(C_0,B)/BC_0].$$

with Var, and Cov indicating estimated variance and covariance, respectively.

The one-sided lower 90% confidence interval on $C_T$ is equivalent to the time the one-sided lower(upper) 90% confidence interval on the predicted concentration has the value $C_0 \pm KC_0$. For this equivalent definition, the MHT is the smallest solution to a quadratic equation:

$$a(MHT)^2 + b(MHT) + c = 0, \text{ so}$$

$$MHT = -(b/2a) - [b^2 - 4ac]^{1/2}/2a.$$

The quadratic coefficients a, b, and c are functions of the slope (B) and intercept ($C_0$) for the zero-order approximating model:

$$a = B^2 - t^2(df,0.10)Var(B)$$

$$b = -2[|B|C_0 + t^2(df,0.10)Cov(C_0,B)], \text{ and}$$

$$c = (KC_0)^2 - t^2(df,0.10)Var(C_0).$$

7. Estimated MHT values greater than the time of the experimental study are set equal to the maximum storage time (e.g., 56 days or 111 days).

For approximating models other than a line, the same procedure was adapted for estimating ESE MHTs. The appropriate 90% lower confidence interval was estimated for each approximating model. Estimated ESE MHTs were found by solving for the time when the 10% (or K%) intercept change and 90% lower confidence interval are equal. ESE MHTs are estimated by iteratively calculating the time that the 10% (or K%) intercept change and 90% confidence interval intersect.

From the results of these statistical analyses, it can be shown that each analyte has a MHT which can be established. Obviously, these are not related to the administrative/political aspects of the environmental analysis. Therefore, it is necessary to consider the end use of the data when determining the maximum holding time.

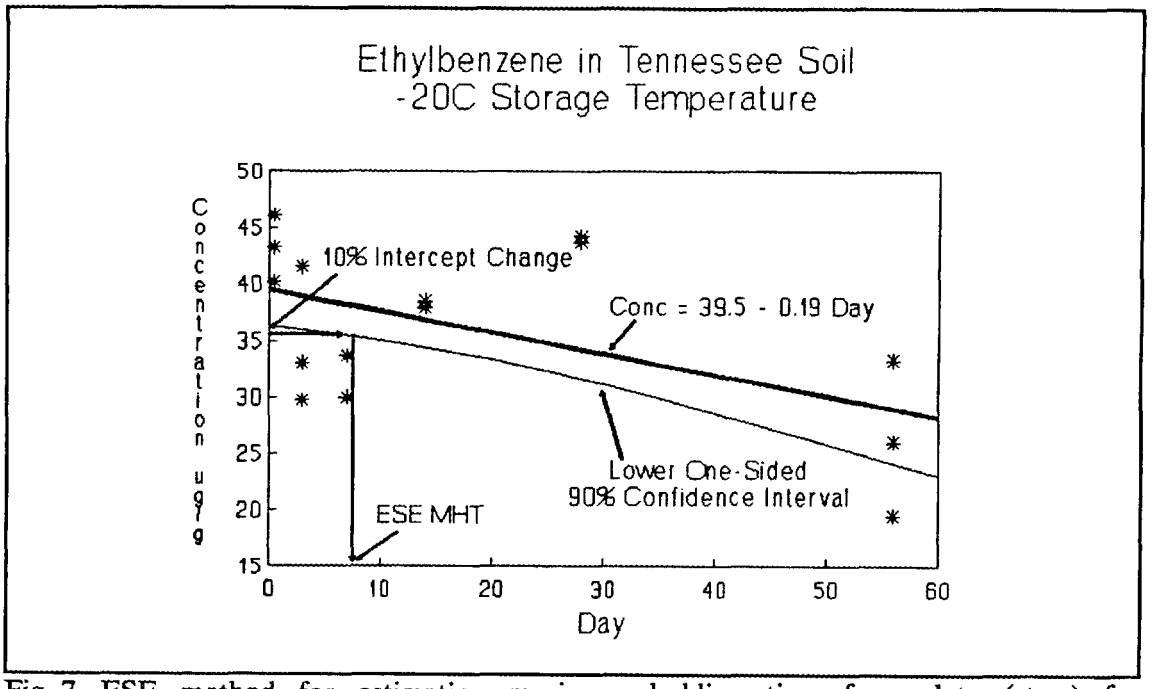


Fig. 7. ESE method for estimating maximum holding time from data (stars) for ethylbenzene in Tennessee soil at -20°C storage temperature.

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## 5. CONCLUSIONS

From a regulatory point of view, extension of sample holding times without compromising data quality would reduce the cost associated with waste site characterization and remedial action by reducing the possibility that additional sampling will be required due to the failure to meet the holding times. This has an important economic effect on investigations carried out under SARA. From the point of view of RCRA, where quarterly soil monitoring is carried out, preservation of the samples would allow direct comparison with the samples collected during the subsequent quarter. Because regulatory decisions are made based on changes in soil concentrations of contaminants, this would be important in reducing analytical variability. According to the regulated community, the ability to preserve and archive important samples for later verification would greatly reduce the possibility of error in regulatory decision-making, and would certainly eliminate the need for resampling.

From the analytical standpoint, improvements in the quality assurance process are expected. This holding time study has shown that most soil samples are <u>not stable</u> at refrigerator temperatures for a sufficient time to allow distribution and analysis. Although silt/clay and sandy soils were used to estimate maximum holding times, these factors are not necessarily known prior to sampling and chemical analysis. Therefore, the choice may not be clear in practice which maximum holding time to select from Tables 4-5 because of unknown factor combinations. Unfortunately, the soil type is such an important factor that a general recommendation can't be made for both soil types. For silt/clay soils, all VOCs except tetrachlorethane have a maximum holding time of at least 20 days if stored immediately at -20°C. The maximum holding time is 4-8 days for tetrachloroethane under the same conditions. For sandy soils, maximum holding times for only six VOCs can be recommended. The six VOCs are methylene chloride, 1,1,2-trichloroethane, bromoform, 1,1,2,2-tetrachloroethane, styrene, and o-xylene which should be stored at -70°C. The recommended maximum holding time for these six VOCs is at least 20 days. The remaining 13 VOCs in sandy soil must either be analyzed in situ or an alternate preservation method is required. These recommended maximum holding times are conservative estimates made after reviewing MHTs for all factor combinations and summary statistics in Appendix A.

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# APPENDIX A

Summary Statistics for Volatile Organic Compounds in Soil Samples.

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Table A.1 Summary concentrations (ug/g) for Bromomethane.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | N | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 53.9 | . | . | . | . | . | . | 53.9 |
| | | St Dev | 8.2 | . | . | . | . | . | . | 8.2 |
| | -70°C | N | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 47.2 | 23.4 | 14.9 | 13.5 | 16.7 | 10.9 | 22.5 |
| | | St Dev | . | 15.2 | 11.9 | 2.8 | 0.9 | 2.8 | . | 15.0 |
| | -20°C | N | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 68.1 | 48.1 | 57.3 | 76.5 | 87.9 | 60.2 | 64.7 |
| | | St Dev | . | 3.0 | 5.3 | 4.9 | 6.6 | 2.5 | 7.6 | 14.0 |
| Tennessee | None | N | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 59.7 | . | . | . | . | . | . | 59.7 |
| | | St Dev | 5.8 | . | . | . | . | . | . | 5.8 |
| | -70°C | N | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 2.7 | 3.9 | 6.5 | 7.6 | 5.2 | . | 5.3 |
| | | St Dev | . | 3.9 | 0.1 | 0.6 | 0.2 | 0.6 | . | 2.2 |
| | -20°C | N | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 36.9 | 19.7 | 33.4 | 29.9 | 2.2 | . | 23.6 |
| | | St Dev | . | 9.9 | 8.6 | 5.6 | 3.6 | 0.8 | . | 15.2 |
| | 4°C | N | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 4.5 | 0.3 | 0.0 | . | 0.0 | . | 1.0 |
| | | St Dev | . | 1.0 | 0.1 | 0.0 | . | 0.0 | . | 1.9 |
| Mississippi | None | N | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 46.6 | . | . | . | . | . | . | 46.6 |
| | | St Dev | 5.1 | . | . | . | . | . | . | 5.1 |
| | -70°C | N | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 13.6 | 16.5 | 19.8 | 10.5 | 9.2 | . | 13.5 |
| | | St Dev | . | 0.1 | 0.2 | 1.1 | 2.0 | 8.0 | . | 5.2 |
| | -20°C | N | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 31.3 | 19.1 | 20.7 | 17.4 | 16.8 | . | 21.7 |
| | | St Dev | . | 11.7 | . | 1.7 | 3.8 | . | . | 7.2 |
| | 4°C | N | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 12.5 | 4.3 | 1.2 | 0.0 | 0.0 | . | 1.8 |
| | | St Dev | . | . | 4.9 | 1.7 | 0.0 | 0.0 | . | 3.8 |

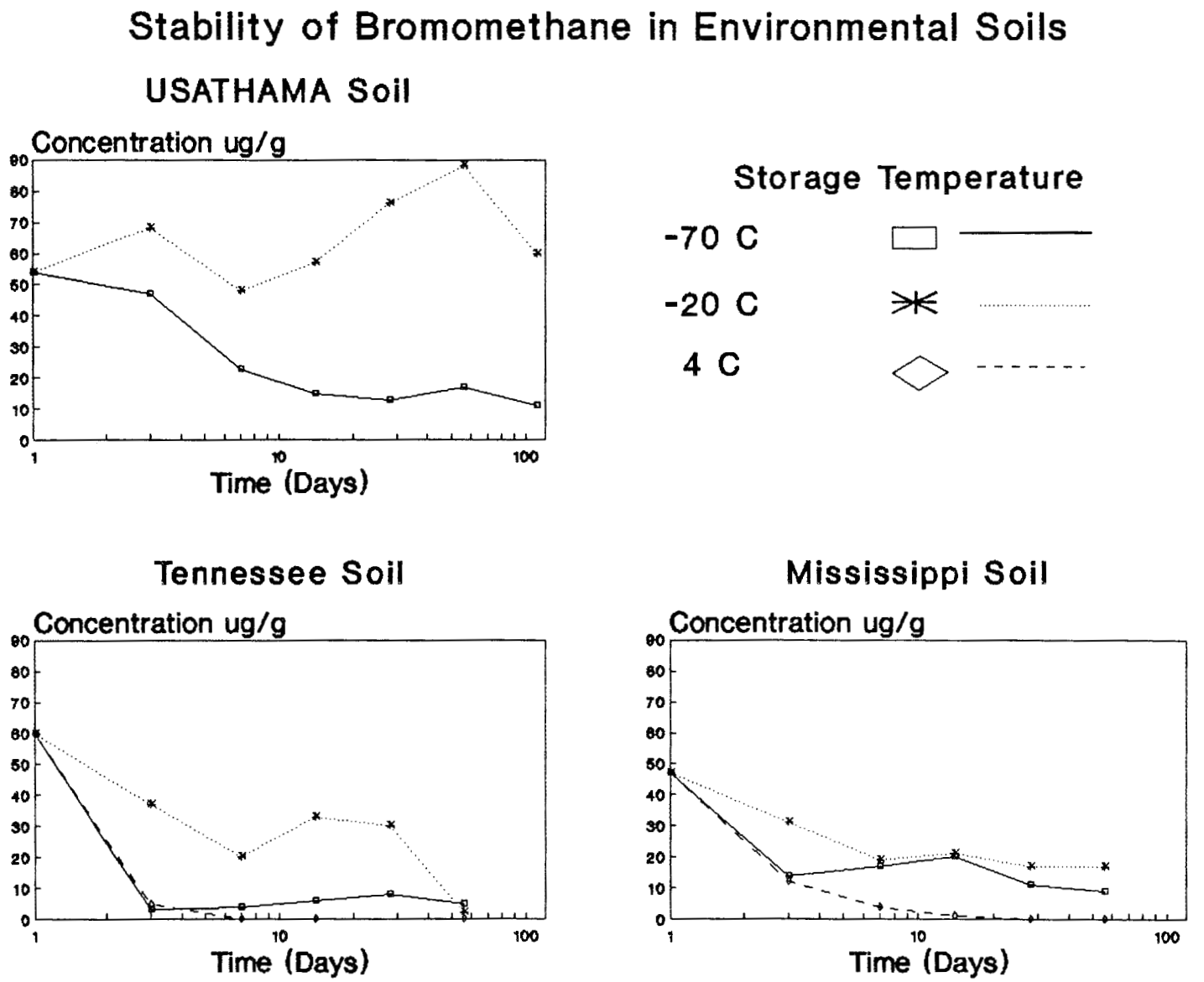


Fig. A.1 Bromomethane in soil samples.

Table A.2 Summary concentrations (ug/g) for Chloroethane.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 68.3 | . | . | . | . | . | . | 68.3 |
| | | St Dev | 13.5 | . | . | . | . | . | . | 13.5 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 46.9 | 27.6 | 16.6 | 13.1 | 18.7 | 9.7 | 23.7 |
| | | St Dev | . | 15.5 | 14.8 | 2.6 | 1.7 | 3.6 | . | 15.2 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 67.9 | 63.9 | 68.4 | 91.2 | 104 | 75.1 | 77.1 |
| | | St Dev | . | 9.9 | 7.6 | 7.0 | 6.2 | 6.0 | 10.5 | 15.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 80.5 | . | . | . | . | . | . | 80.5 |
| | | St Dev | 6.6 | . | . | . | . | . | . | 6.6 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 10.8 | 10.3 | 10.3 | 12.9 | 13.0 | . | 11.4 |
| | | St Dev | . | 2.4 | 0.9 | 0.2 | 1.5 | 2.5 | . | 1.8 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 70.8 | 49.2 | 68.0 | 88.7 | 16.3 | . | 56.1 |
| | | St Dev | . | 14.2 | 20.0 | 2.4 | 8.7 | 4.8 | . | 28.4 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 74.9 | 58.4 | 58.7 | . | 4.5 | . | 46.6 |
| | | St Dev | . | 6.5 | 6.7 | 17.9 | . | 1.2 | . | 29.9 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 57.6 | . | . | . | . | . | . | 57.6 |
| | | St Dev | 9.2 | . | . | . | . | . | . | 9.2 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 11.3 | 13.2 | 15.6 | 7.9 | 6.2 | . | 10.4 |
| | | St Dev | . | 0.2 | 0.4 | 0.4 | 2.4 | 6.2 | . | 4.5 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 37.5 | 16.0 | 17.4 | 13.9 | 17.2 | . | 20.9 |
| | | St Dev | . | 17.0 | . | 1.9 | 3.3 | . | . | 11.4 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 15.0 | 4.6 | 1.7 | 0.4 | 0.0 | . | 2.2 |
| | | St Dev | . | . | 6.5 | 1.8 | 0.4 | 0.0 | . | 4.5 |

# Stability of Chloroethane in Environmental Soils

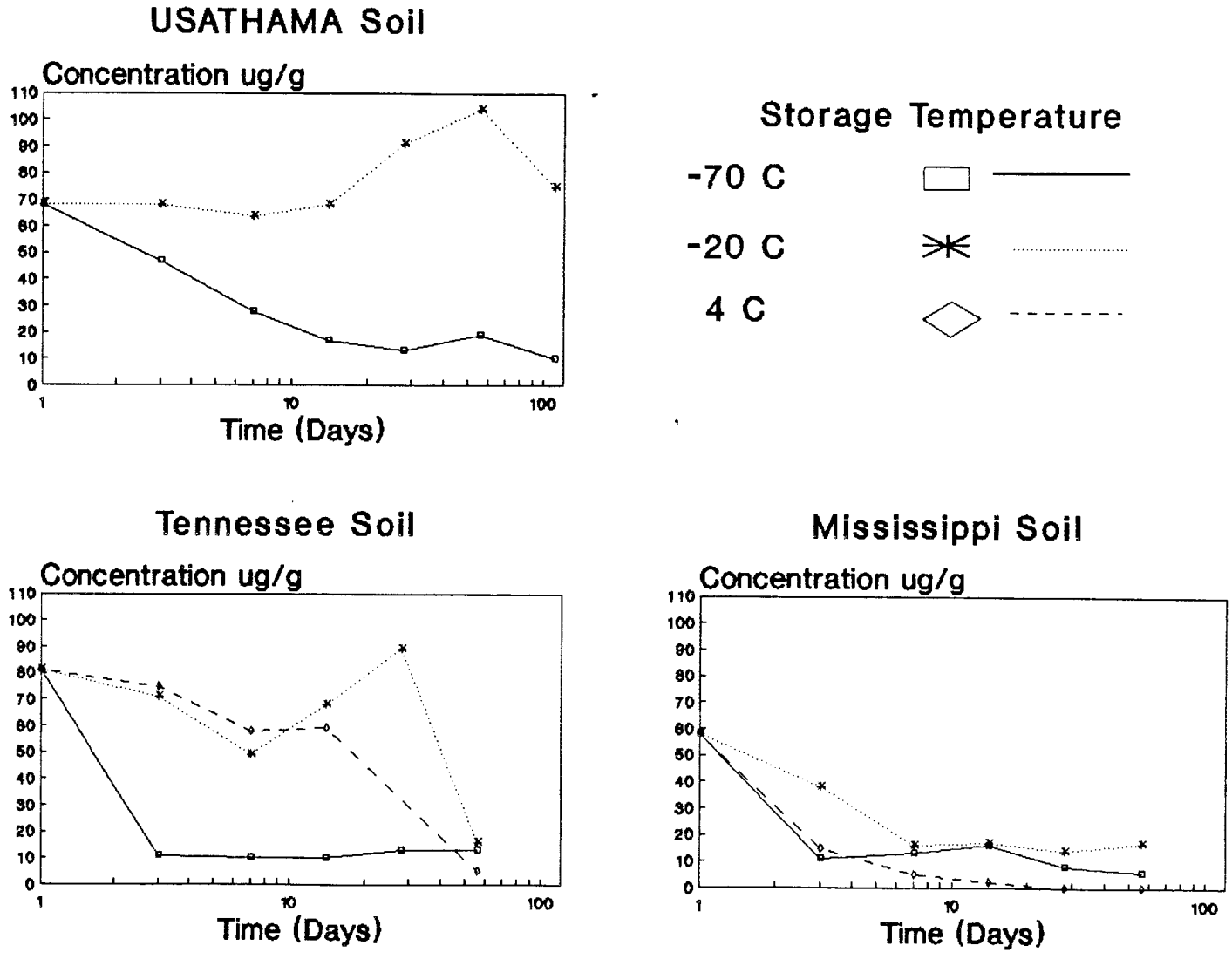


Fig. A.2 Chloroethane in soil samples.

Table A.3 Summary concentrations (ug/g) for Methylene Chloride.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 58.4 | . | . | . | . | . | . | 58.4 |
| | | St Dev | 5.7 | . | . | . | . | . | . | 5.7 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 53.4 | 44.0 | 45.5 | 79.5 | 125 | 91.9 | 71.9 |
| | | St Dev | . | 7.8 | 9.2 | 7.0 | 19.7 | 4.0 | . | 32.7 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 67.0 | 57.3 | 65.2 | 74.0 | 69.5 | 57.8 | 64.4 |
| | | St Dev | . | 1.3 | 3.9 | 2.4 | 2.5 | 1.2 | 3.2 | 6.6 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 161 | . | . | . | . | . | . | 161 |
| | | St Dev | 18.3 | . | . | . | . | . | . | 18.3 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 121 | 130 | 130 | 191 | 161 | . | 145 |
| | | St Dev | . | 25.6 | 29.5 | 36.6 | 2.0 | 24.7 | . | 34.4 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 124 | 169 | 152 | 181 | 116 | . | 144 |
| | | St Dev | . | 47.4 | 42.5 | 2.1 | 74.1 | 43.3 | . | 46.1 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 119 | 105 | 138 | . | 8.4 | . | 89.7 |
| | | St Dev | . | 62.8 | 25.0 | 33.4 | . | 2.8 | . | 62.9 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 99.0 | . | . | . | . | . | . | 99.0 |
| | | St Dev | 23.9 | . | . | . | . | . | . | 23.9 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 91.0 | 115 | 131 | 103 | 127 | . | 113 |
| | | St Dev | . | 24.0 | 37.5 | 20.8 | 48.7 | 21.1 | . | 31.5 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 85.0 | 77.6 | 68.6 | 58.6 | 88.0 | . | 73.2 |
| | | St Dev | . | 38.0 | . | 10.2 | 20.1 | . | . | 19.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 3.0 | 4.0 | . | 13.0 |
| | | Avg | . | 47.1 | 65.6 | 59.7 | 30.4 | 6.4 | . | 36.5 |
| | | St Dev | . | . | 31.8 | 13.2 | 23.5 | 6.8 | . | 28.5 |

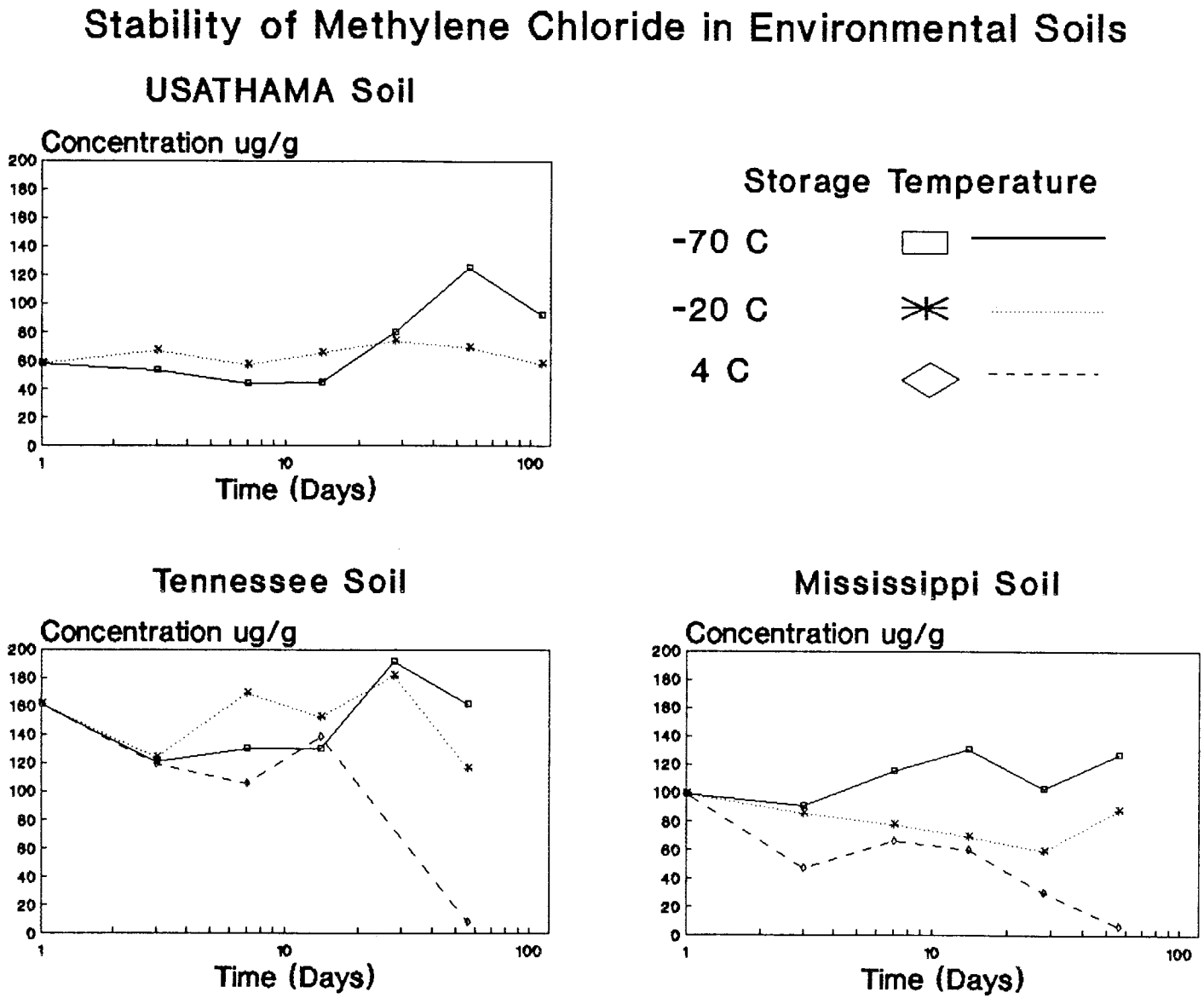


Fig. A.3 Methylene Chloride in soil samples.

Table A.4 Summary concentrations (ug/g) for 1,1-Dichloroethene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 73.9 | . | . | . | . | . | . | 73.9 |
| | | St Dev | 7.5 | . | . | . | . | . | . | 7.5 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 50.7 | 28.4 | 18.5 | 9.9 | 9.2 | 7.4 | 22.3 |
| | | St Dev | . | 18.7 | 13.4 | 5.8 | 1.5 | 2.2 | . | 18.4 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 86.1 | 73.7 | 80.6 | 95.0 | 82.5 | 78.4 | 82.0 |
| | | St Dev | . | 3.2 | 6.0 | 7.2 | 9.4 | 4.6 | 10.4 | 9.3 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 80.0 | . | . | . | . | . | . | 80.0 |
| | | St Dev | 3.0 | . | . | . | . | . | . | 3.0 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 9.0 | 8.8 | 7.5 | 8.7 | 9.2 | . | 8.6 |
| | | St Dev | . | 2.4 | 0.1 | 0.5 | 2.2 | 2.0 | . | 1.4 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 80.0 | 57.2 | 61.9 | 84.3 | 14.6 | . | 57.5 |
| | | St Dev | . | 17.9 | 23.2 | 1.2 | 5.7 | 5.8 | . | 29.9 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 73.7 | 59.2 | 50.7 | . | 4.5 | . | 44.2 |
| | | St Dev | . | 7.5 | 5.5 | 16.9 | . | 1.8 | . | 28.8 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 59.9 | . | . | . | . | . | . | 59.9 |
| | | St Dev | 11.2 | . | . | . | . | . | . | 11.2 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 6.9 | 6.5 | 7.5 | 3.4 | 5.3 | . | 5.7 |
| | | St Dev | . | 0.1 | 0.5 | 0.0 | 1.5 | 0.8 | . | 1.7 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 36.1 | 9.2 | 8.8 | 6.4 | 8.0 | . | 14.3 |
| | | St Dev | . | 19.3 | . | 0.9 | 1.7 | . | . | 14.2 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 13.2 | 5.3 | 1.6 | 0.3 | 0.0 | . | 2.1 |
| | | St Dev | . | . | 5.4 | 1.8 | 0.2 | 0.0 | . | 4.0 |

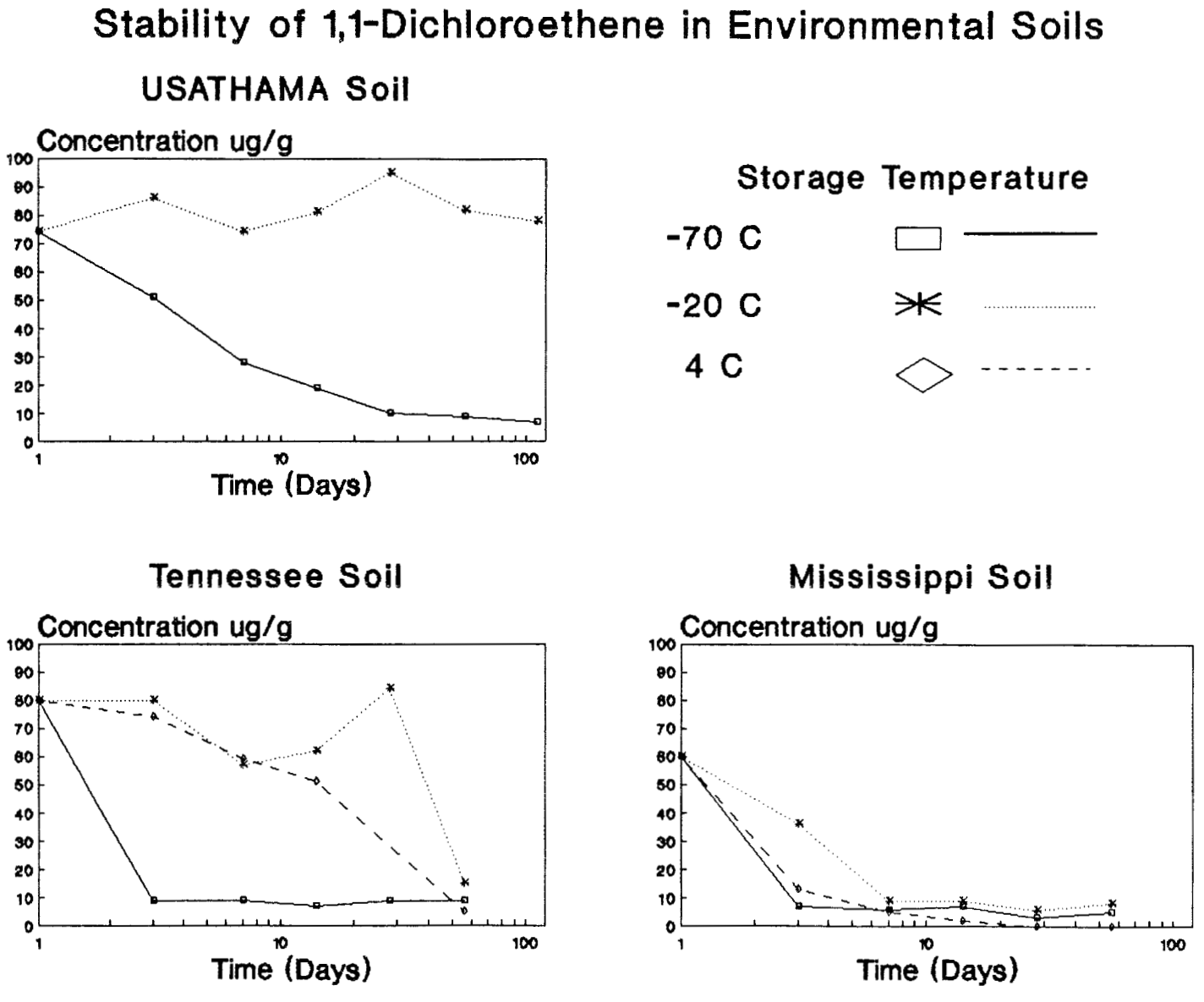


Fig. A.4 1,1-Dichloroethene in soil samples.

Table A.5 Summary concentrations (ug/g) for 1,1-Dichloroethane.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 74.8 | . | . | . | . | . | . | 74.8 |
| | | St Dev | 7.8 | . | . | . | . | . | . | 7.8 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 49.4 | 40.4 | 29.8 | 21.8 | 19.6 | 15.9 | 31.0 |
| | | St Dev | . | 19.6 | 15.1 | 6.3 | 3.5 | 2.7 | . | 15.4 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 79.4 | 73.4 | 86.9 | 84.0 | 81.5 | 76.1 | 80.0 |
| | | St Dev | . | 6.9 | 8.5 | 6.7 | 5.6 | 4.0 | 8.5 | 7.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 77.3 | . | . | . | . | . | . | 77.3 |
| | | St Dev | 1.5 | . | . | . | . | . | . | 1.5 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 20.3 | 20.7 | 18.8 | 21.4 | 23.6 | . | 20.8 |
| | | St Dev | . | 3.2 | 0.6 | 0.6 | 3.4 | 3.0 | . | 2.5 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 65.7 | 62.6 | 62.2 | 79.5 | 28.6 | . | 57.6 |
| | | St Dev | . | 12.3 | 17.1 | 1.7 | 3.9 | 8.2 | . | 20.3 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 66.0 | 58.6 | 52.9 | . | 3.3 | . | 42.8 |
| | | St Dev | . | 5.5 | 6.9 | 14.4 | . | 0.9 | . | 27.4 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 62.9 | . | . | . | . | . | . | 62.9 |
| | | St Dev | 9.0 | . | . | . | . | . | . | 9.0 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 20.4 | 22.5 | 23.3 | 10.5 | 18.9 | . | 18.7 |
| | | St Dev | . | 1.2 | 1.5 | 0.0 | 2.6 | 3.5 | . | 5.3 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 44.5 | 27.0 | 26.3 | 18.3 | 30.2 | . | 29.1 |
| | | St Dev | . | 11.2 | . | 1.8 | 4.3 | . | . | 10.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 25.0 | 12.1 | 5.0 | 0.7 | 0.0 | . | 4.8 |
| | | St Dev | . | . | 11.3 | 6.2 | 0.4 | 0.0 | . | 8.2 |

# Stability of 1,1-Dichloroethane in Environmental Soils

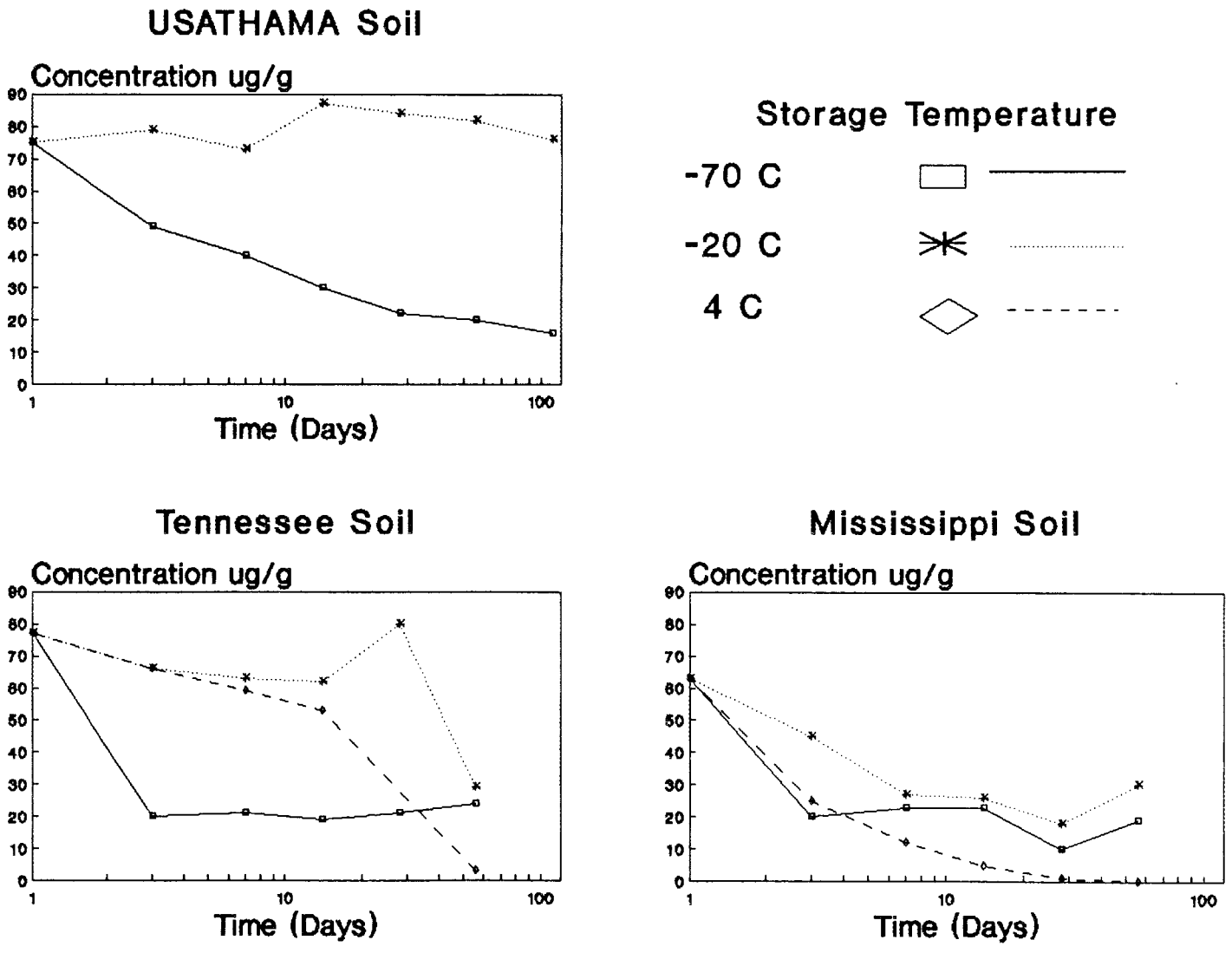


Fig. A.5 1,1-Dichloroethane in soil samples.

Table A.6 Summary concentrations (ug/g) for Chloroform.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 63.7 | . | . | . | . | . | . | 63.7 |
| | | St Dev | 5.6 | . | . | . | . | . | . | 5.6 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 55.0 | 42.8 | 31.4 | 27.9 | 25.2 | 20.7 | 35.3 |
| | | St Dev | . | 12.0 | 10.4 | 4.9 | 4.9 | 2.2 | . | 13.4 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 78.5 | 67.6 | 71.2 | 73.1 | 73.0 | 68.5 | 71.6 |
| | | St Dev | . | 5.3 | 3.7 | 4.8 | 6.1 | 4.1 | 6.2 | 5.7 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 67.7 | . | . | . | . | . | . | 67.7 |
| | | St Dev | 1.5 | . | . | . | . | . | . | 1.5 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 28.1 | 26.1 | 26.2 | 30.1 | 30.6 | . | 28.0 |
| | | St Dev | . | 2.4 | 1.1 | 1.2 | 4.4 | 2.3 | . | 2.7 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 60.2 | 57.9 | 61.1 | 76.5 | 35.7 | . | 56.6 |
| | | St Dev | . | 13.7 | 12.5 | 3.0 | 1.5 | 10.8 | . | 16.4 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 54.2 | 48.4 | 46.4 | . | 2.8 | . | 36.0 |
| | | St Dev | . | 3.9 | 5.9 | 12.2 | . | 1.5 | . | 23.0 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 59.8 | . | . | . | . | . | . | 59.8 |
| | | St Dev | 6.9 | . | . | . | . | . | . | 6.9 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 26.8 | 28.0 | 29.3 | 13.5 | 22.4 | . | 23.4 |
| | | St Dev | . | 2.1 | 1.8 | 0.3 | 3.0 | 5.0 | . | 6.7 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 44.7 | 33.2 | 32.5 | 22.2 | 35.1 | . | 33.3 |
| | | St Dev | . | 7.3 | . | 2.2 | 4.9 | . | . | 8.7 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 29.3 | 16.3 | 9.4 | 2.1 | 0.2 | . | 7.1 |
| | | St Dev | . | . | 11.7 | 9.7 | 1.2 | 0.2 | . | 10.0 |

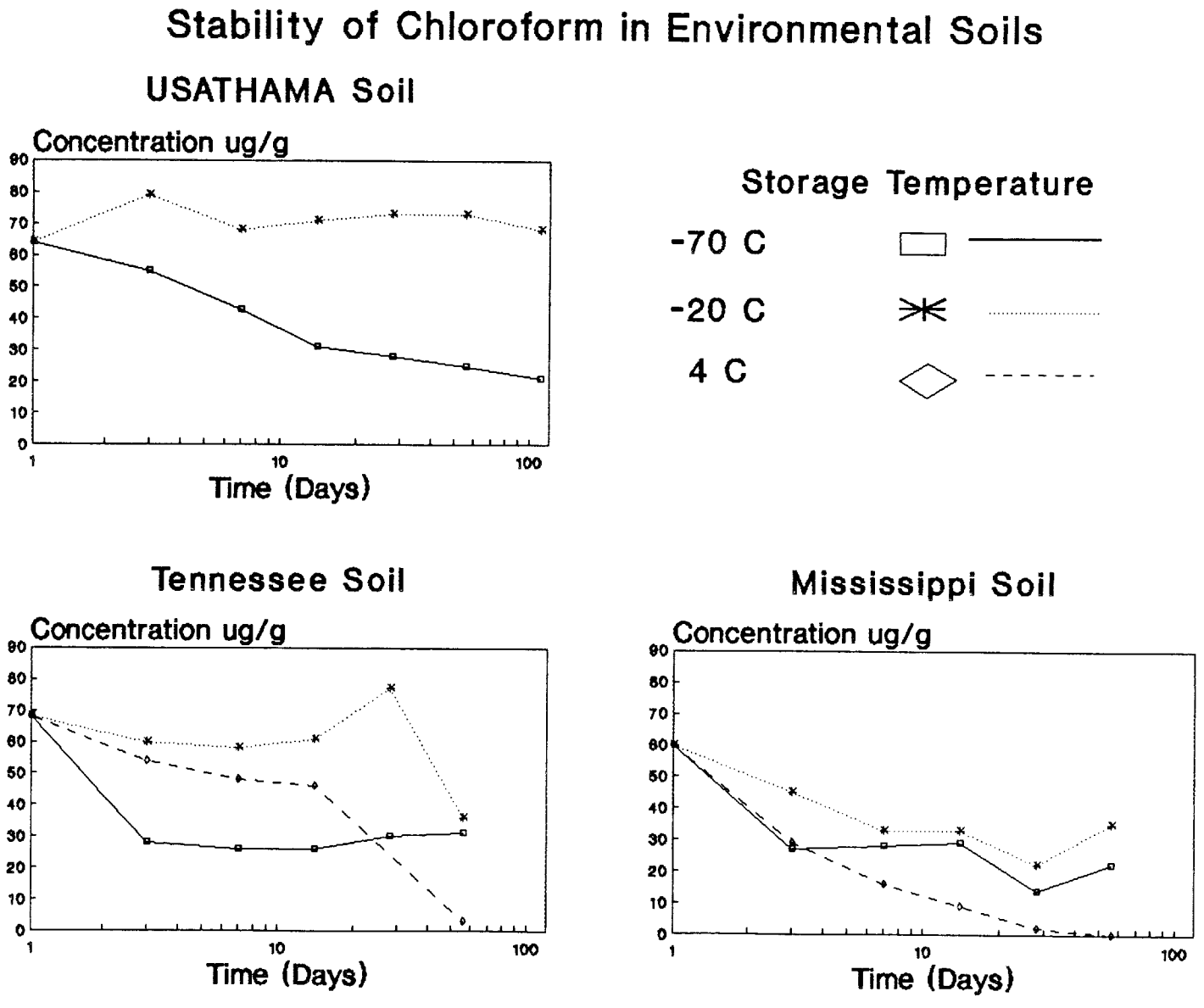


Fig. A.6 Chloroform in soil samples.

Table A.7 Summary concentrations (ug/g) for Carbon Tetrachloride.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 69.7 | . | . | . | . | . | . | 69.7 |
| | | St Dev | 7.4 | . | . | . | . | . | . | 7.4 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 47.4 | 27.4 | 17.6 | 9.9 | 9.2 | 7.0 | 21.3 |
| | | St Dev | . | 17.2 | 11.6 | 5.7 | 2.5 | 2.0 | . | 17.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 85.3 | 66.5 | 71.6 | 78.7 | 76.4 | 69.2 | 73.8 |
| | | St Dev | . | 6.1 | 5.9 | 7.8 | 8.6 | 4.5 | 8.6 | 8.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 73.1 | . | . | . | . | . | . | 73.1 |
| | | St Dev | 3.3 | . | . | . | . | . | . | 3.3 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 7.6 | 8.6 | 8.3 | 8.4 | 9.1 | . | 8.4 |
| | | St Dev | . | 1.5 | 0.4 | 0.4 | 2.1 | 1.8 | . | 1.1 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 66.8 | 52.6 | 65.1 | 74.5 | 15.2 | . | 52.5 |
| | | St Dev | . | 14.8 | 18.4 | 1.3 | 4.6 | 6.2 | . | 25.1 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 59.5 | 55.9 | 52.0 | . | 3.9 | . | 40.7 |
| | | St Dev | . | 5.6 | 4.5 | 17.9 | . | 2.0 | . | 26.0 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 58.8 | . | . | . | . | . | . | 58.8 |
| | | St Dev | 8.3 | . | . | . | . | . | . | 8.3 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 6.1 | 5.8 | 6.2 | 2.7 | 4.4 | . | 4.9 |
| | | St Dev | . | 0.3 | 0.3 | 0.1 | 1.3 | 0.8 | . | 1.6 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 35.4 | 9.2 | 7.7 | 4.7 | 7.5 | . | 13.3 |
| | | St Dev | . | 15.7 | . | 1.2 | 1.1 | . | . | 13.8 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 15.9 | 6.6 | 2.1 | 0.2 | 0.0 | . | 2.6 |
| | | St Dev | . | . | 7.1 | 2.6 | 0.1 | 0.0 | . | 5.0 |

# Stability of Carbon Tetrachloride in Environmental Soils

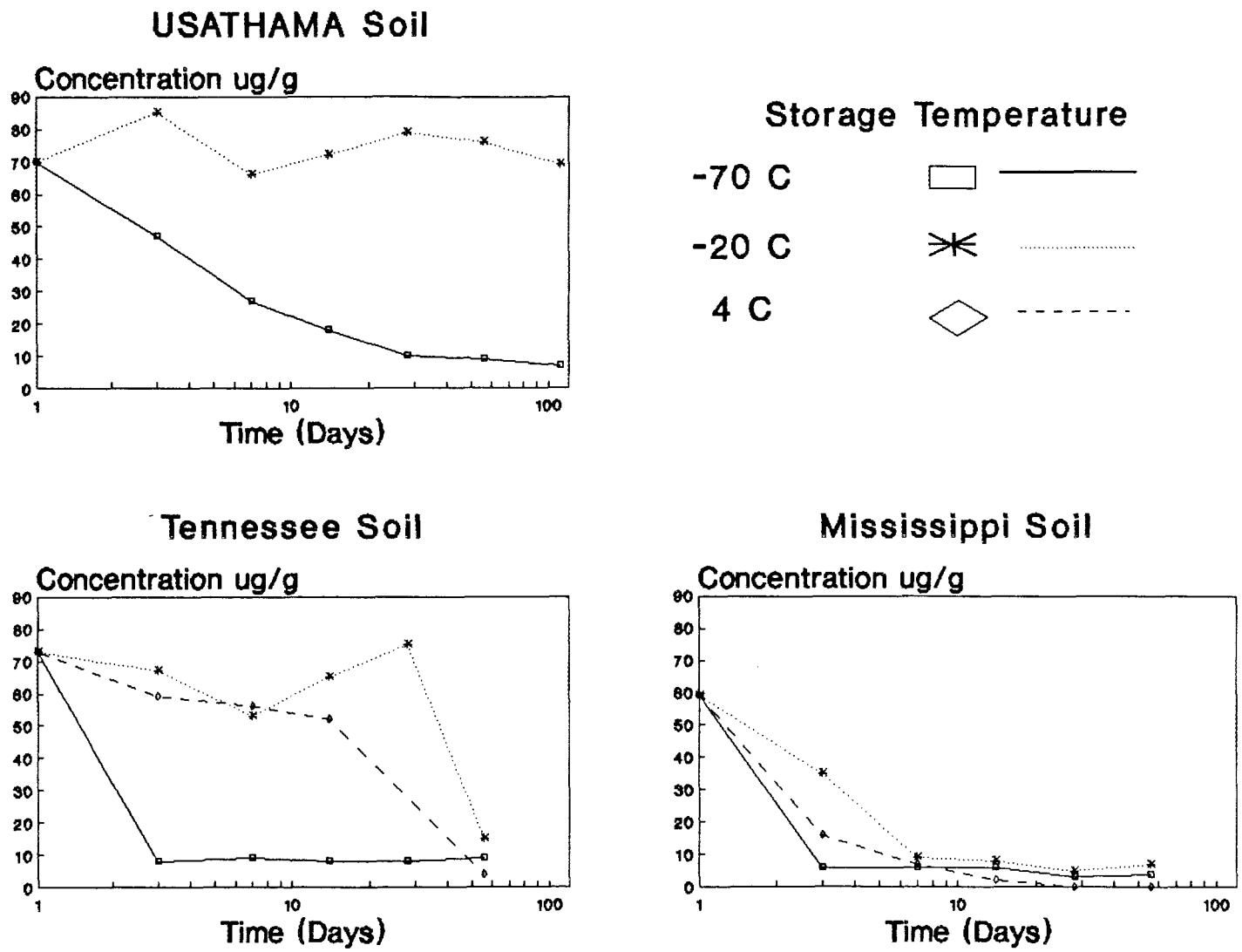


Fig. A.7 Carbon Tetrachloride in soil samples.

Table A.8 Summary concentrations (ug/g) for 1,2-Dichloropropane.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Stogage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 58.3 | . | . | . | . | . | . | 58.3 |
| | | St Dev | 6.8 | . | . | . | . | . | . | 6.8 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 54.3 | 46.8 | 39.8 | 38.7 | 32.1 | 27.3 | 41.4 |
| | | St Dev | . | 6.3 | 5.9 | 3.6 | 4.3 | 2.2 | . | 9.3 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 68.3 | 59.1 | 71.8 | 62.4 | 63.7 | 64.8 | 65.0 |
| | | St Dev | . | 6.0 | 8.1 | 3.5 | 6.9 | 2.9 | 5.0 | 6.6 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 57.2 | . | . | . | . | . | . | 57.2 |
| | | St Dev | 0.8 | . | . | . | . | . | . | 0.8 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 34.7 | 34.9 | 30.4 | 33.5 | 34.8 | . | 33.4 |
| | | St Dev | . | 0.2 | 1.2 | 2.3 | 4.0 | 2.4 | . | 2.7 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 46.7 | 56.9 | 54.5 | 68.6 | 39.9 | . | 51.7 |
| | | St Dev | . | 13.2 | 8.5 | 1.4 | 1.4 | 11.0 | . | 12.8 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 48.0 | 43.7 | 41.0 | . | 2.2 | . | 32.0 |
| | | St Dev | . | 2.7 | 4.9 | 9.6 | . | 1.1 | . | 20.4 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 57.6 | . | . | . | . | . | . | 57.6 |
| | | St Dev | 6.6 | . | . | . | . | . | . | 6.6 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 34.6 | 33.3 | 34.3 | 16.9 | 25.8 | . | 28.1 |
| | | St Dev | . | 2.3 | 1.9 | 0.6 | 2.8 | 6.0 | . | 7.8 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 46.1 | 37.4 | 36.0 | 25.9 | 40.1 | . | 36.6 |
| | | St Dev | . | 4.0 | . | 1.7 | 4.4 | . | . | 7.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 31.9 | 19.7 | 11.7 | 2.8 | 0.3 | . | 8.5 |
| | | St Dev | . | . | 11.4 | 10.4 | 1.9 | 0.2 | . | 11.1 |

## Stability of 1,2-Dichloropropane in Environmental Soils

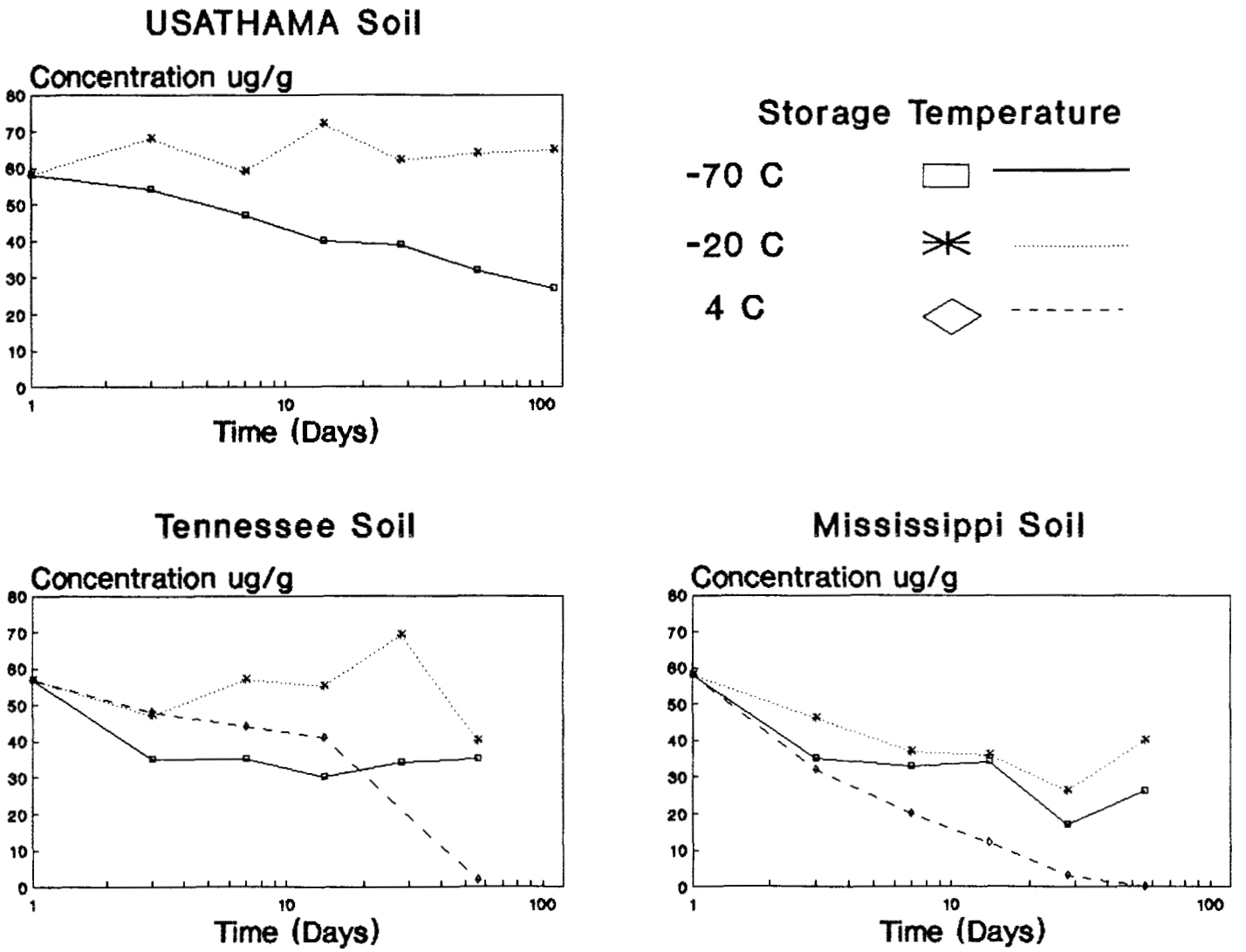


Fig. A.8 1,2-Dichloropropane in soil samples.

Table A.9 Summary concentrations (ug/g) for Trichloroethene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 60.7 | . | . | . | . | . | . | 60.7 |
| | | St Dev | 8.9 | . | . | . | . | . | . | 8.9 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 51.5 | 35.2 | 24.8 | 22.0 | 18.3 | 15.6 | 29.4 |
| | | St Dev | . | 12.7 | 8.5 | 4.3 | 4.2 | 1.3 | . | 14.1 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 76.2 | 61.2 | 63.7 | 61.7 | 55.6 | 57.3 | 62.4 |
| | | St Dev | . | 5.2 | 5.3 | 4.6 | 7.2 | 2.5 | 5.8 | 7.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 54.9 | . | . | . | . | . | . | 54.9 |
| | | St Dev | 2.8 | . | . | . | . | . | . | 2.8 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 19.9 | 19.2 | 20.5 | 22.1 | 22.1 | . | 20.7 |
| | | St Dev | . | 1.0 | 0.2 | 1.8 | 4.1 | 0.7 | . | 2.0 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 51.5 | 45.3 | 50.6 | 59.0 | 25.7 | . | 45.1 |
| | | St Dev | . | 8.3 | 7.9 | 1.8 | 1.1 | 8.2 | . | 13.6 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 40.2 | 31.0 | 29.7 | . | 1.8 | . | 24.2 |
| | | St Dev | . | 2.7 | 2.8 | 8.3 | . | 1.2 | . | 15.9 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 48.8 | . | . | . | . | . | . | 48.8 |
| | | St Dev | 6.0 | . | . | . | . | . | . | 6.0 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 16.7 | 15.8 | 16.8 | 6.8 | 11.3 | . | 13.0 |
| | | St Dev | . | 0.5 | 1.2 | 0.9 | 2.2 | 2.2 | . | 4.4 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 34.3 | 19.0 | 17.8 | 11.5 | 16.3 | . | 20.0 |
| | | St Dev | . | 10.2 | . | 1.2 | 2.5 | . | . | 9.3 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 18.3 | 12.0 | 8.2 | 2.8 | 0.7 | . | 5.8 |
| | | St Dev | . | . | 6.8 | 5.5 | 1.5 | 0.5 | . | 6.2 |

# Stability of Trichloroethene in Environmental Soils

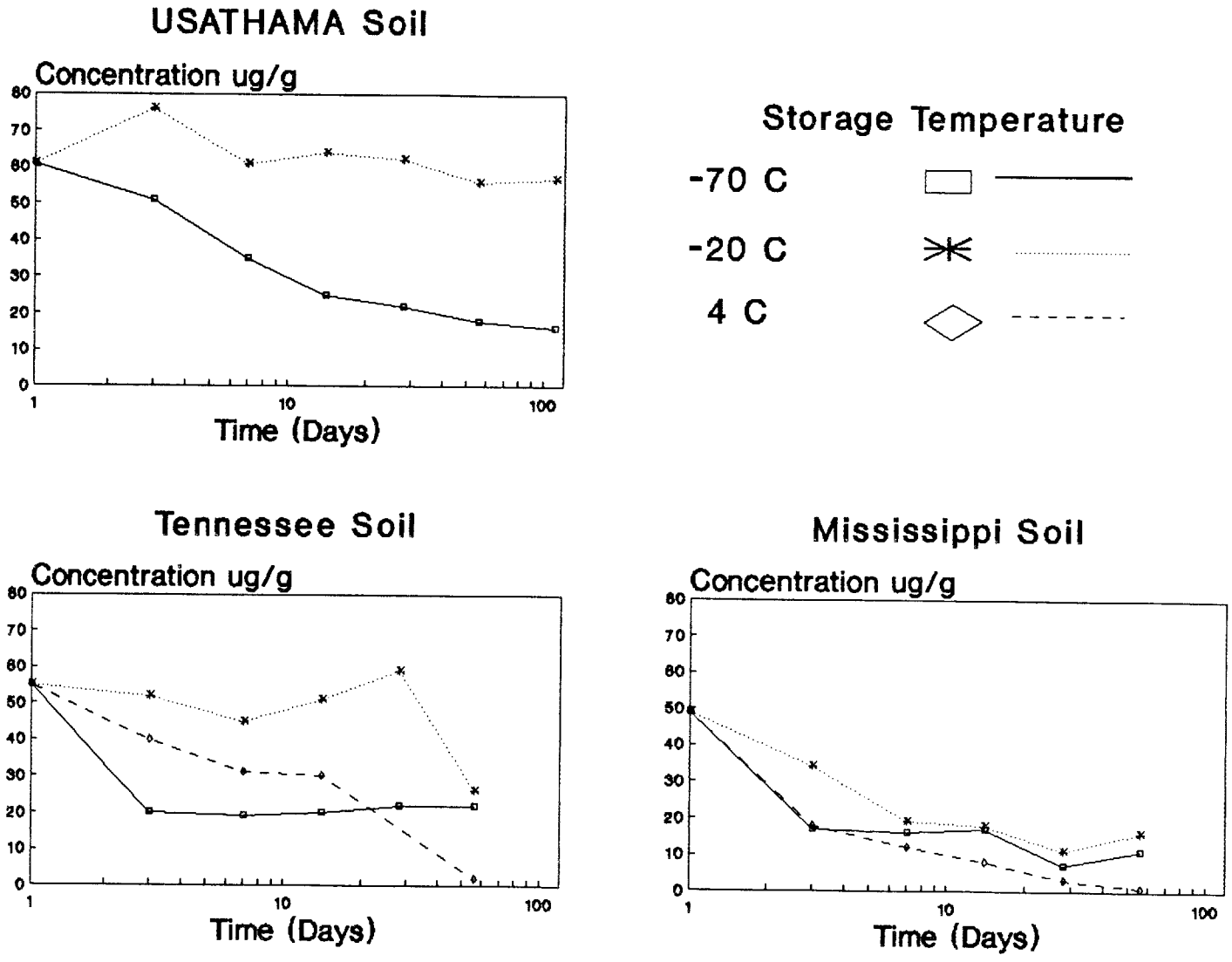


Fig. A.9 Trichloroethene in soil samples.

Table A.10 Summary concentrations (ug/g) for Benzene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 59.9 | . | . | . | . | . | . | 59.9 |
| | | St Dev | 8.2 | . | . | . | . | . | . | 8.2 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 49.2 | 37.9 | 26.0 | 20.2 | 17.7 | 16.6 | 29.1 |
| | | St Dev | . | 13.4 | 10.2 | 4.2 | 3.0 | 1.8 | . | 14.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 72.7 | 65.8 | 65.8 | 62.5 | 54.7 | 60.8 | 63.8 |
| | | St Dev | . | 3.4 | 4.4 | 5.5 | 5.7 | 2.2 | 6.1 | 6.8 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 62.6 | . | . | . | . | . | . | 62.6 |
| | | St Dev | 2.6 | . | . | . | . | . | . | 2.6 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 25.0 | 22.7 | 22.4 | 27.6 | 26.1 | . | 24.6 |
| | | St Dev | . | 2.5 | 1.8 | 2.0 | 4.1 | 1.3 | . | 2.8 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 56.7 | 52.3 | 56.5 | 71.1 | 31.3 | . | 52.0 |
| | | St Dev | . | 10.6 | 11.7 | 1.2 | 3.0 | 10.8 | . | 15.7 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 45.5 | 38.1 | 34.2 | . | 2.6 | . | 28.5 |
| | | St Dev | . | 4.0 | 4.0 | 8.5 | . | 1.7 | . | 18.1 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 49.7 | . | . | . | . | . | . | 49.7 |
| | | St Dev | 7.9 | . | . | . | . | . | . | 7.9 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 19.9 | 19.7 | 21.0 | 9.9 | 16.0 | . | 16.8 |
| | | St Dev | . | 1.8 | 1.3 | 0.6 | 2.9 | 3.3 | . | 4.7 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 39.0 | 23.7 | 22.9 | 16.2 | 24.8 | . | 25.3 |
| | | St Dev | . | 12.8 | . | 1.4 | 3.6 | . | . | 9.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 18.4 | 13.4 | 6.6 | 1.9 | 0.3 | . | 5.3 |
| | | St Dev | . | . | 11.6 | 6.4 | 0.9 | 0.3 | . | 7.2 |

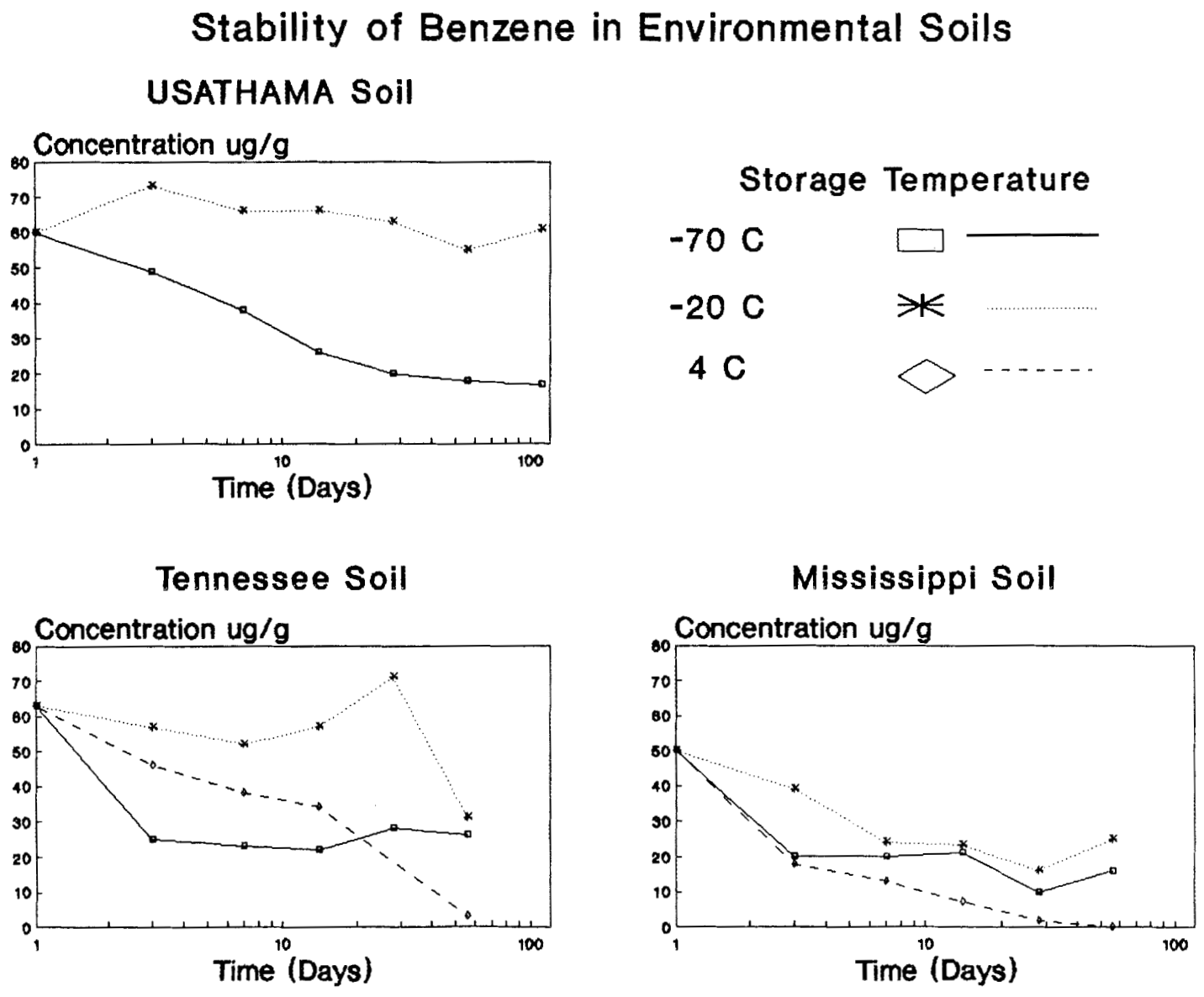


Fig. A.10 Benzene in soil samples.

Table A.11 Summary concentrations (ug/g) for 1,1,2-Trichloroethane.

| Soil Type | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 44.1 | . | . | . | . | . | . | 44.1 |
| | | St Dev | 3.8 | . | . | . | . | . | . | 3.8 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 51.8 | 46.1 | 44.0 | 40.3 | 42.3 | 40.2 | 44.6 |
| | | St Dev | . | 5.3 | 3.1 | 3.3 | 1.7 | 3.5 | . | 5.2 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 62.3 | 54.2 | 60.9 | 52.6 | 55.0 | 57.9 | 57.2 |
| | | St Dev | . | 1.2 | 1.6 | 1.7 | 2.7 | 0.8 | 2.6 | 3.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 45.1 | . | . | . | . | . | . | 45.1 |
| | | St Dev | 1.3 | . | . | . | . | . | . | 1.3 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 37.1 | 36.6 | 41.0 | 41.4 | 44.0 | . | 40.1 |
| | | St Dev | . | 7.1 | 1.7 | 1.5 | 1.7 | 0.1 | . | 3.7 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 35.1 | 42.2 | 51.0 | 58.0 | 48.8 | . | 46.2 |
| | | St Dev | . | 15.6 | 2.3 | 0.9 | 0.9 | 8.9 | . | 11.3 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 34.5 | 32.6 | 34.0 | . | 1.4 | . | 24.5 |
| | | St Dev | . | 2.6 | 3.6 | 6.0 | . | 0.6 | . | 15.6 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 52.8 | . | . | . | . | . | . | 52.8 |
| | | St Dev | 3.2 | . | . | . | . | . | . | 3.2 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 48.7 | 45.4 | 48.3 | 29.6 | 38.4 | . | 41.1 |
| | | St Dev | . | 4.3 | 2.5 | 1.9 | 3.6 | 15.8 | . | 10.2 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 46.1 | 47.2 | 46.5 | 38.3 | 51.6 | . | 45.2 |
| | | St Dev | . | 1.5 | . | 1.9 | 3.7 | . | . | 4.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 35.9 | 29.7 | 23.8 | 9.0 | 1.4 | . | 14.9 |
| | | St Dev | . | . | 5.7 | 11.1 | 6.5 | 1.5 | . | 13.6 |

# Stability of 1,1,2-Trichloroethane in Environmental Soils

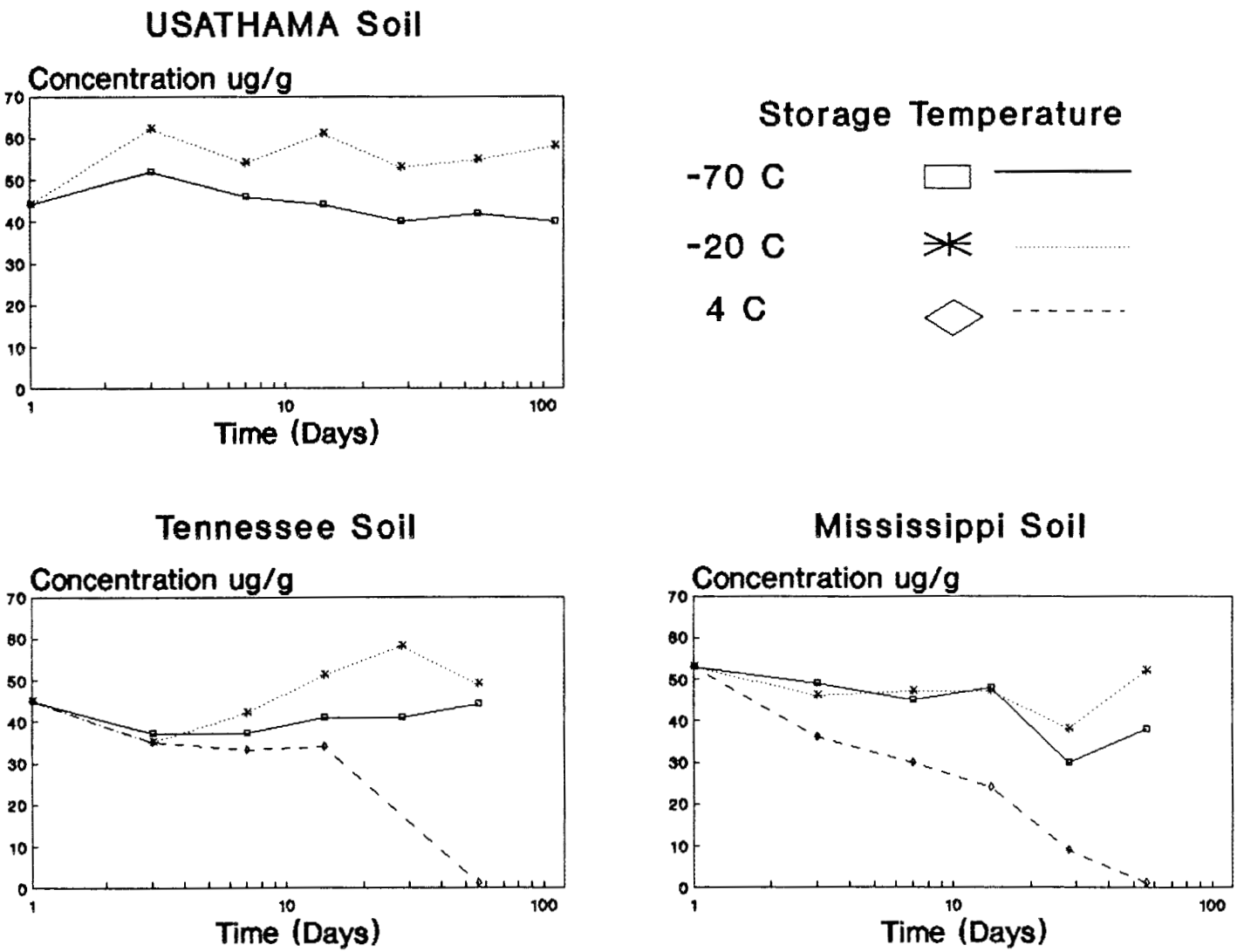


Fig. A.11 1,1,2-Trichloroethane in soil samples.

Table A.12 Summary concentrations (ug/g) for Bromoform.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 34.1 | . | . | . | . | . | . | 34.1 |
| | | St Dev | 2.6 | . | . | . | . | . | . | 2.6 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 38.6 | 31.9 | 29.9 | 31.8 | 33.1 | 33.3 | 33.1 |
| | | St Dev | . | 4.6 | 3.2 | 2.4 | 1.9 | 3.8 | . | 4.2 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 47.1 | 37.8 | 44.4 | 39.7 | 41.2 | 45.0 | 42.5 |
| | | St Dev | . | 2.0 | 2.4 | 1.6 | 1.4 | 0.2 | 1.8 | 3.7 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 26.2 | . | . | . | . | . | . | 26.2 |
| | | St Dev | 0.8 | . | . | . | . | . | . | 0.8 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 22.3 | 22.7 | 30.2 | 28.5 | 31.7 | . | 27.4 |
| | | St Dev | . | 4.1 | 1.5 | 1.0 | 1.2 | 1.1 | . | 4.3 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 19.7 | 25.7 | 34.8 | 36.4 | 31.8 | . | 29.0 |
| | | St Dev | . | 9.3 | 0.2 | 0.2 | 0.0 | 4.1 | . | 7.9 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 19.4 | 18.3 | 15.5 | . | 0.1 | . | 12.5 |
| | | St Dev | . | 2.1 | 1.4 | 2.4 | . | 0.1 | . | 8.4 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 38.2 | . | . | . | . | . | . | 38.2 |
| | | St Dev | 2.0 | . | . | . | . | . | . | 2.0 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 2.0 | . | 12.0 |
| | | Avg | . | 36.9 | 37.7 | 38.1 | 24.3 | 42.4 | . | 35.1 |
| | | St Dev | . | 4.2 | 3.2 | 1.8 | 3.4 | 0.6 | . | 7.2 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 33.0 | 37.3 | 33.9 | 30.6 | 41.1 | . | 34.1 |
| | | St Dev | . | 0.8 | . | 1.4 | 2.8 | . | . | 3.5 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 25.0 | 23.4 | 17.8 | 8.3 | 1.5 | . | 11.7 |
| | | St Dev | . | . | 2.2 | 7.1 | 6.1 | 2.3 | . | 9.9 |

# Stability of Bromoform in Environmental Soils

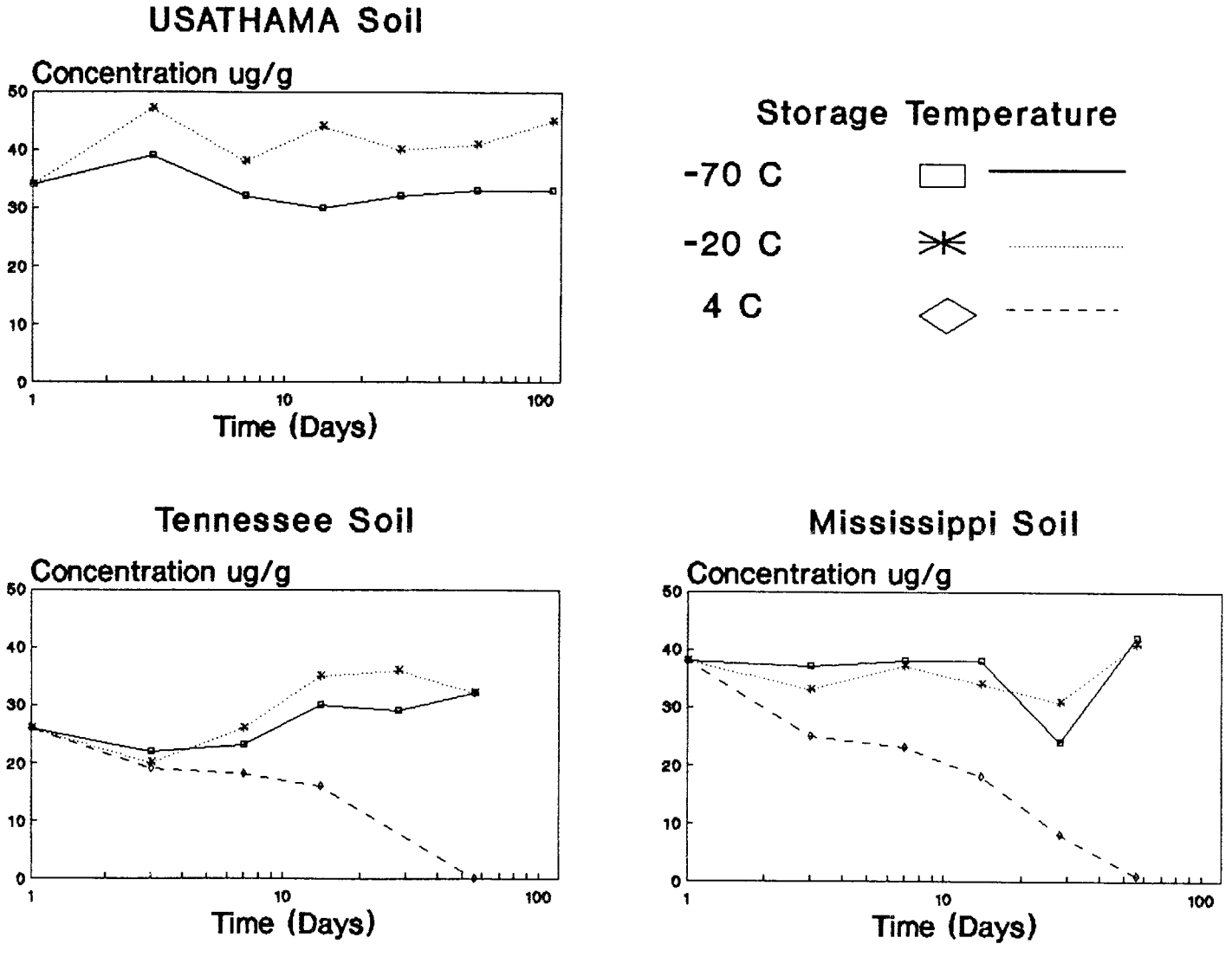


Fig. A.12 Bromoform in soil samples.

Table A.13 Summary concentrations (ug/g) for 1,1,2,2-Tetrachloroethane.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 34.4 | . | . | . | . | . | . | 34.4 |
| | | St Dev | 2.8 | . | . | . | . | . | . | 2.8 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 39.2 | 34.6 | 39.7 | 31.1 | 34.0 | 34.4 | 35.7 |
| | | St Dev | . | 3.5 | 2.8 | 3.6 | 2.1 | 3.9 | . | 4.4 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 47.0 | 40.3 | 54.5 | 38.0 | 42.2 | 47.4 | 45.2 |
| | | St Dev | . | 1.3 | 2.0 | 2.0 | 1.1 | 1.0 | 1.5 | 5.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 31.5 | . | . | . | . | . | . | 31.5 |
| | | St Dev | 0.6 | . | . | . | . | . | . | 0.6 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 28.4 | 25.1 | 32.4 | 32.0 | 37.0 | . | 31.1 |
| | | St Dev | . | 6.2 | 2.2 | 1.5 | 1.4 | 0.7 | . | 4.6 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 24.5 | 29.2 | 37.4 | 40.8 | 40.3 | . | 34.1 |
| | | St Dev | . | 9.7 | 1.1 | 0.2 | 0.6 | 5.3 | . | 8.5 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 26.9 | 24.6 | 26.0 | . | 0.7 | . | 18.7 |
| | | St Dev | . | 2.8 | 2.0 | 3.7 | . | 0.4 | . | 12.0 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 44.1 | . | . | . | . | . | . | 44.1 |
| | | St Dev | 2.1 | . | . | . | . | . | . | 2.1 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 2.0 | . | 12.0 |
| | | Avg | . | 44.2 | 42.5 | 43.1 | 33.7 | 53.6 | . | 42.6 |
| | | St Dev | . | 5.2 | 3.5 | 0.7 | 4.4 | 2.3 | . | 7.3 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 40.3 | 42.4 | 40.7 | 40.9 | 49.1 | . | 41.7 |
| | | St Dev | . | 1.7 | . | 1.6 | 3.5 | . | . | 3.2 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 33.7 | 30.3 | 25.9 | 14.6 | 2.9 | . | 17.2 |
| | | St Dev | . | . | 1.8 | 8.9 | 10.8 | 4.3 | . | 13.2 |

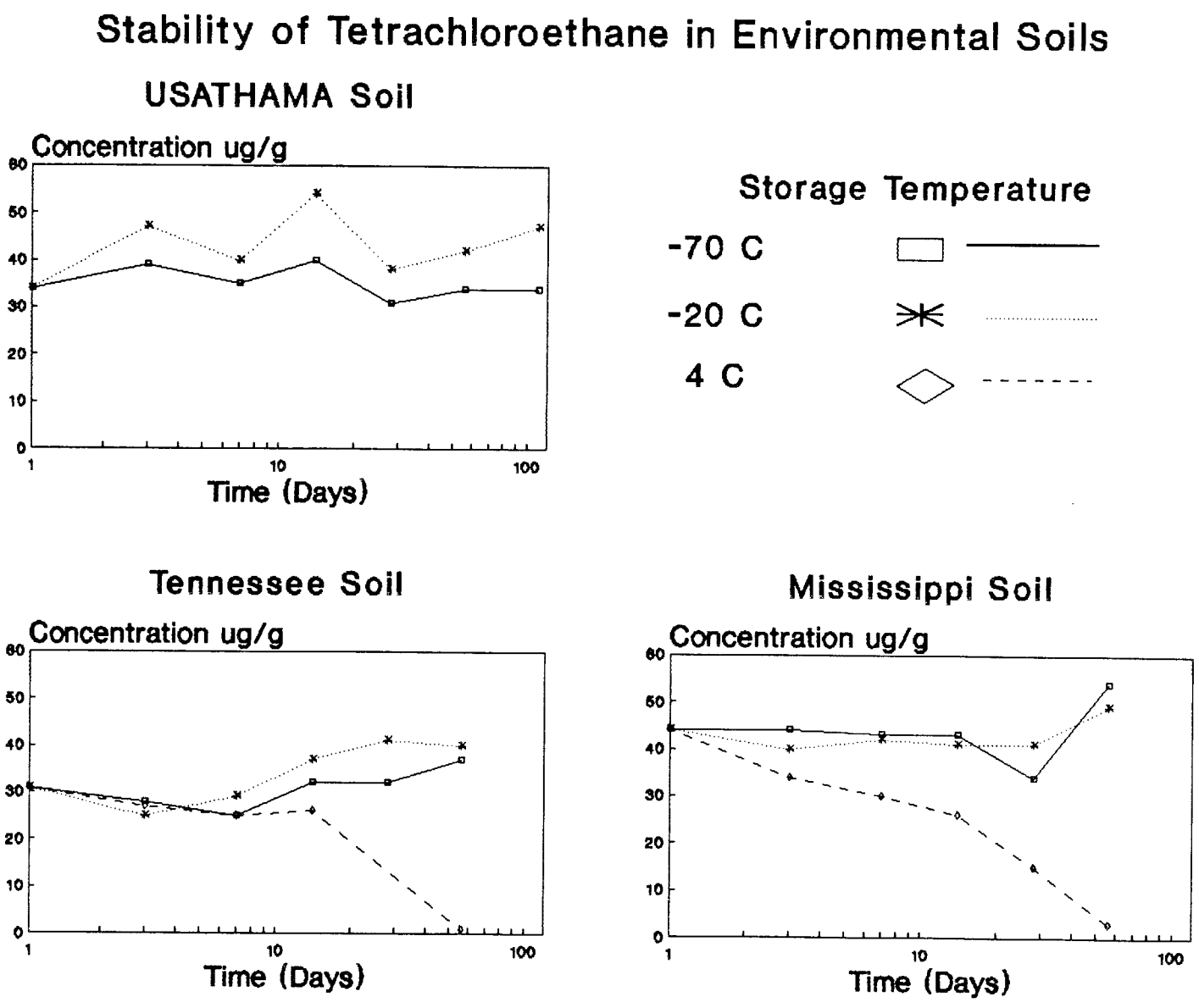


Fig. A.13 1,1,2,2-Tetrachloroethane in soil samples.

Table A.14 Summary concentrations (ug/g) for Tetrachloroethene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 52.2 | . | . | . | . | . | . | 52.2 |
| | | St Dev | 6.2 | . | . | . | . | . | . | 6.2 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 40.2 | 30.2 | 20.2 | 20.0 | 16.6 | 13.8 | 24.6 |
| | | St Dev | . | 8.6 | 5.3 | 3.0 | 3.3 | 1.4 | . | 10.1 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 56.7 | 44.4 | 44.6 | 43.2 | 39.4 | 35.9 | 43.7 |
| | | St Dev | . | 6.0 | 4.3 | 4.0 | 5.8 | 1.0 | 3.6 | 7.4 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 51.1 | . | . | . | . | . | . | 51.1 |
| | | St Dev | 7.1 | . | . | . | . | . | . | 7.1 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 20.4 | 17.8 | 19.8 | 21.3 | 19.9 | . | 19.9 |
| | | St Dev | . | 4.3 | 0.0 | 2.2 | 4.6 | 0.8 | . | 2.5 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 46.3 | 39.4 | 47.0 | 51.5 | 23.4 | . | 40.4 |
| | | St Dev | . | 8.5 | 6.5 | 2.6 | 0.3 | 7.6 | . | 12.1 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 35.8 | 27.7 | 26.7 | . | 1.9 | . | 21.8 |
| | | St Dev | . | 3.9 | 1.9 | 7.3 | . | 1.4 | . | 14.1 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 44.3 | . | . | . | . | . | . | 44.3 |
| | | St Dev | 6.1 | . | . | . | . | . | . | 6.1 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 17.2 | 12.9 | 13.1 | 5.8 | 8.2 | . | 10.9 |
| | | St Dev | . | 2.7 | 0.5 | 1.2 | 2.3 | 1.7 | . | 4.3 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 33.5 | 15.9 | 13.8 | 8.9 | 12.2 | . | 17.2 |
| | | St Dev | . | 11.0 | . | 1.1 | 1.6 | . | . | 10.4 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 16.8 | 12.0 | 7.3 | 1.8 | 0.3 | . | 5.1 |
| | | St Dev | . | . | 7.5 | 6.0 | 0.8 | 0.3 | . | 6.3 |

# Stability of Tetrachloroethene in Environmental Soils

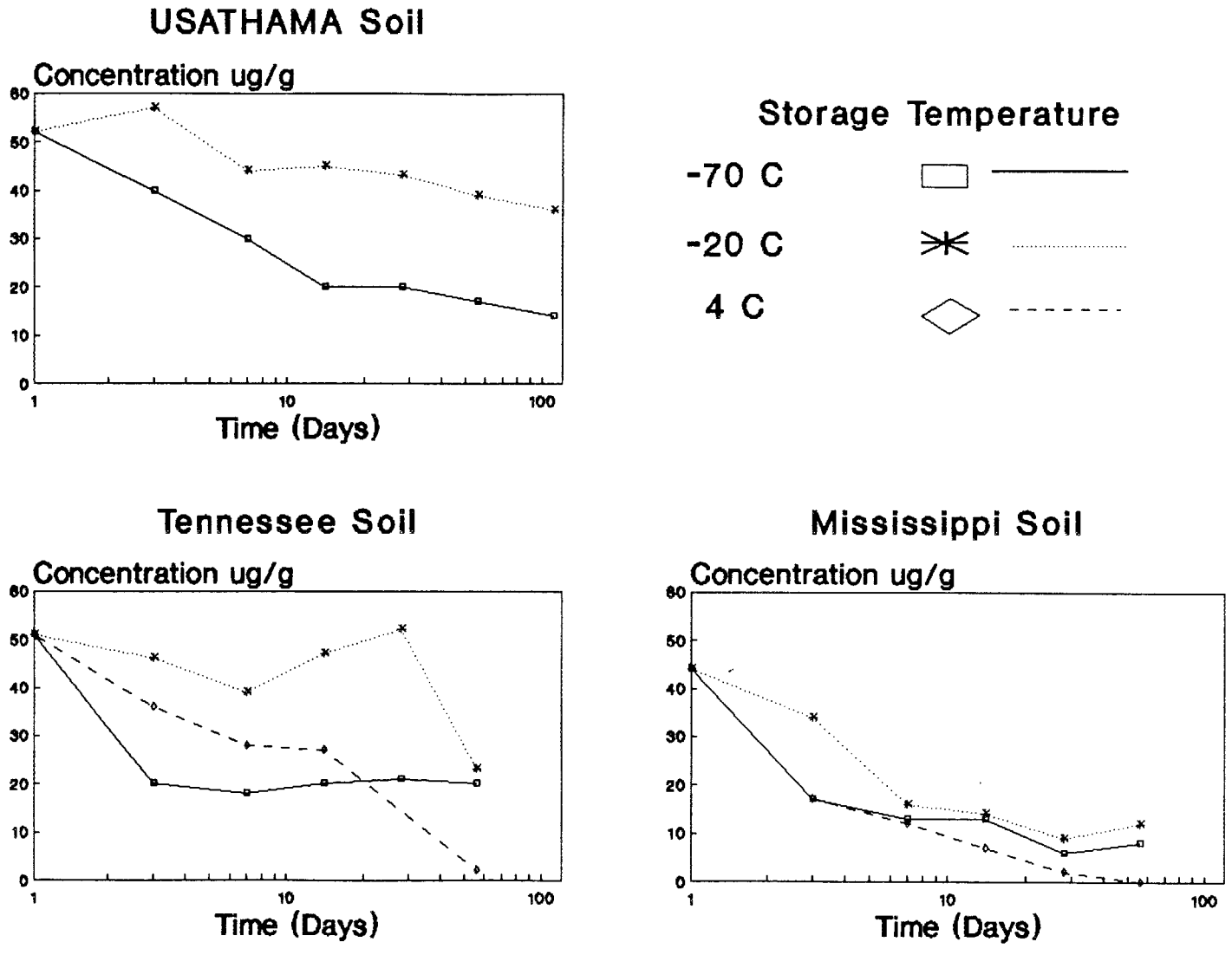


Fig. A.14 Tetrachloroethene in soil samples.

Table A.15 Summary concentrations (ug/g) for Toluene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 57.2 | . | . | . | . | . | . | 57.2 |
| | | St Dev | 4.6 | . | . | . | . | . | . | 4.6 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 51.0 | 37.5 | 30.8 | 28.2 | 22.4 | 20.3 | 33.1 |
| | | St Dev | . | 5.6 | 2.7 | 4.1 | 3.4 | 2.4 | . | 11.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 67.1 | 54.1 | 54.0 | 51.9 | 49.9 | 50.9 | 54.4 |
| | | St Dev | . | 4.6 | 5.0 | 15.2 | 5.4 | 1.6 | 5.1 | 8.8 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 88.7 | . | . | . | . | . | . | 88.7 |
| | | St Dev | 8.6 | . | . | . | . | . | . | 8.6 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 54.0 | 40.8 | 26.8 | 51.9 | 22.6 | . | 38.1 |
| | | St Dev | . | 0.4 | 10.0 | 24.8 | 9.6 | 31.8 | . | 20.5 |
| | -20°C | Num | . | 3.0 | 2.0 | 1.0 | 2.0 | 3.0 | . | 11.0 |
| | | Avg | . | 72.0 | 72.3 | 84.4 | 97.7 | 0.1 | . | 38.1 |
| | | St Dev | . | 15.5 | 11.9 | . | 17.5 | 0.0 | . | 20.5 |
| | 4°C | Num | . | 3.0 | 3.0 | 3.0 | . | 3.0 | . | 12.0 |
| | | Avg | . | 44.5 | 34.8 | 38.9 | . | 1.4 | . | 29.9 |
| | | St Dev | . | 7.9 | 5.3 | 4.0 | . | 0.4 | . | 18.1 |
| Mississippi | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 45.8 | . | . | . | . | . | . | 45.8 |
| | | St Dev | 9.1 | . | . | . | . | . | . | 9.1 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 38.5 | 15.6 | 23.8 | 6.7 | 18.9 | . | 19.1 |
| | | St Dev | . | 18.9 | 13.4 | 2.5 | 11.5 | 6.1 | . | 14.0 |
| | -20°C | Num | . | 1.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 8.0 |
| | | Avg | . | 42.0 | 26.2 | 28.1 | 27.9 | 25.7 | . | 29.2 |
| | | St Dev | . | . | . | 15.4 | 3.7 | . | . | 9.8 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 27.7 | 34.1 | 31.0 | 11.1 | 2.1 | . | 17.3 |
| | | St Dev | . | . | 26.0 | 27.6 | 5.8 | 1.9 | . | 18.9 |

# Stability of Toluene in Environmental Soils

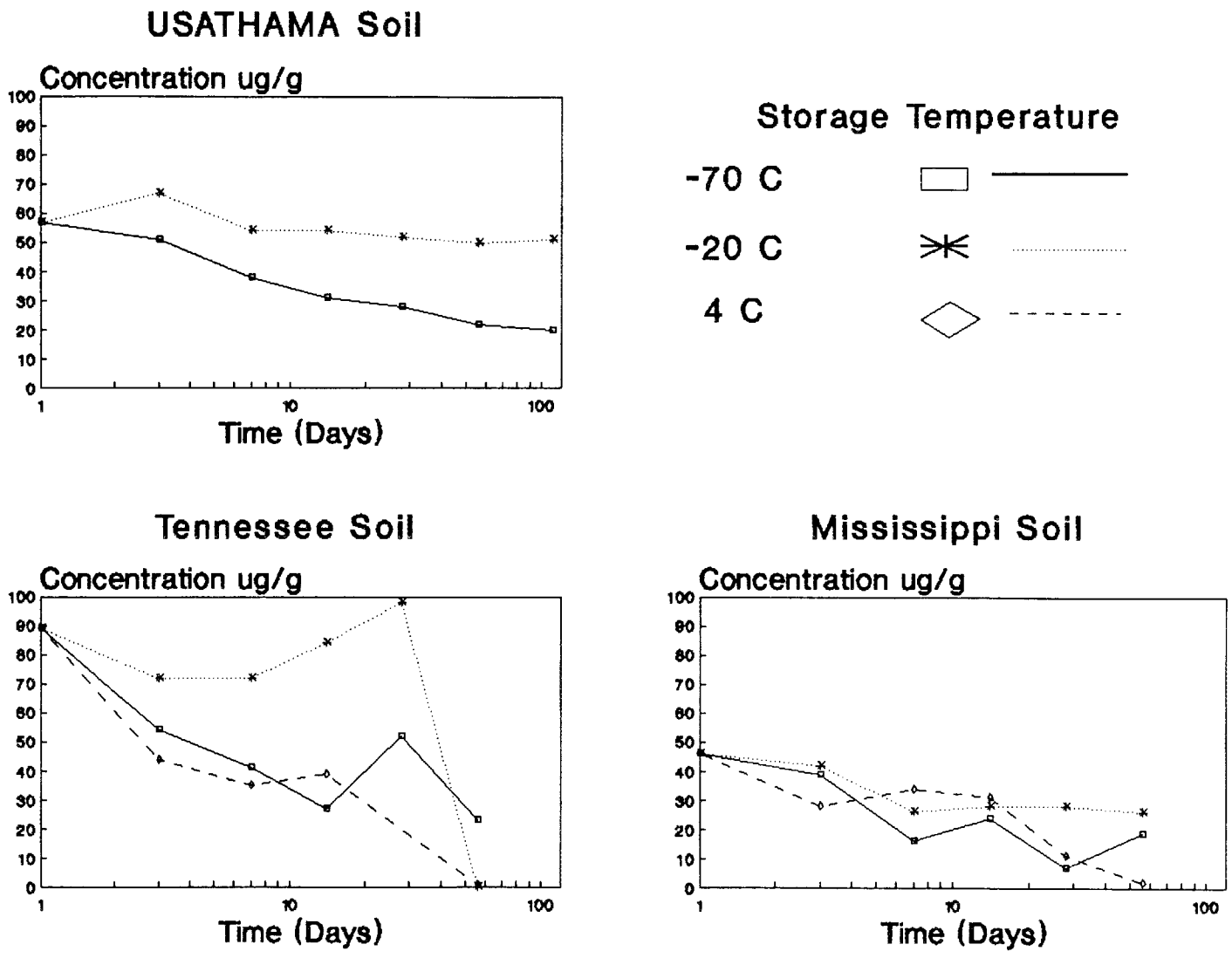


Fig. A.15 Toluene in soil samples.

Table A.16 Summary concentrations (ug/g) for Chlorobenzene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 42.7 | . | . | . | . | . | . | 42.7 |
| | | St Dev | 3.7 | . | . | . | . | . | . | 3.7 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 38.9 | 31.0 | 27.3 | 29.6 | 29.3 | 28.1 | 31.1 |
| | | St Dev | . | 4.6 | 3.4 | 3.4 | 2.1 | 1.4 | . | 5.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 47.2 | 38.6 | 44.4 | 38.7 | 40.1 | 40.3 | 41.5 |
| | | St Dev | . | 2.9 | 2.9 | 2.2 | 3.4 | 0.8 | 2.4 | 3.8 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 32.6 | . | . | . | . | . | . | 32.6 |
| | | St Dev | 1.8 | . | . | . | . | . | . | 1.8 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 26.4 | 23.0 | 28.4 | 30.1 | 28.0 | . | 27.3 |
| | | St Dev | . | 2.9 | 0.4 | 2.5 | 2.3 | 1.4 | . | 3.0 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 25.5 | 26.9 | 33.3 | 37.6 | 26.2 | . | 29.2 |
| | | St Dev | . | 6.3 | 0.6 | 0.5 | 0.2 | 5.5 | . | 6.0 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 22.5 | 16.8 | 16.5 | . | 0.6 | . | 13.3 |
| | | St Dev | . | 1.5 | 1.3 | 3.2 | . | 0.5 | . | 8.8 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 32.6 | . | . | . | . | . | . | 32.6 |
| | | St Dev | 2.5 | . | . | . | . | . | . | 2.5 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 28.7 | 26.1 | 27.0 | 11.1 | 17.7 | . | 21.2 |
| | | St Dev | . | 2.5 | 1.2 | 4.2 | 2.3 | 4.6 | . | 7.5 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 28.3 | 24.9 | 23.4 | 15.4 | 23.3 | . | 22.8 |
| | | St Dev | . | 3.1 | . | 1.3 | 2.0 | . | . | 4.9 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 15.5 | 13.3 | 10.9 | 3.2 | 0.8 | . | 6.5 |
| | | St Dev | . | . | 3.6 | 5.8 | 2.3 | 0.7 | . | 6.2 |

# Stability of Chlorobenzene in Environmental Soils

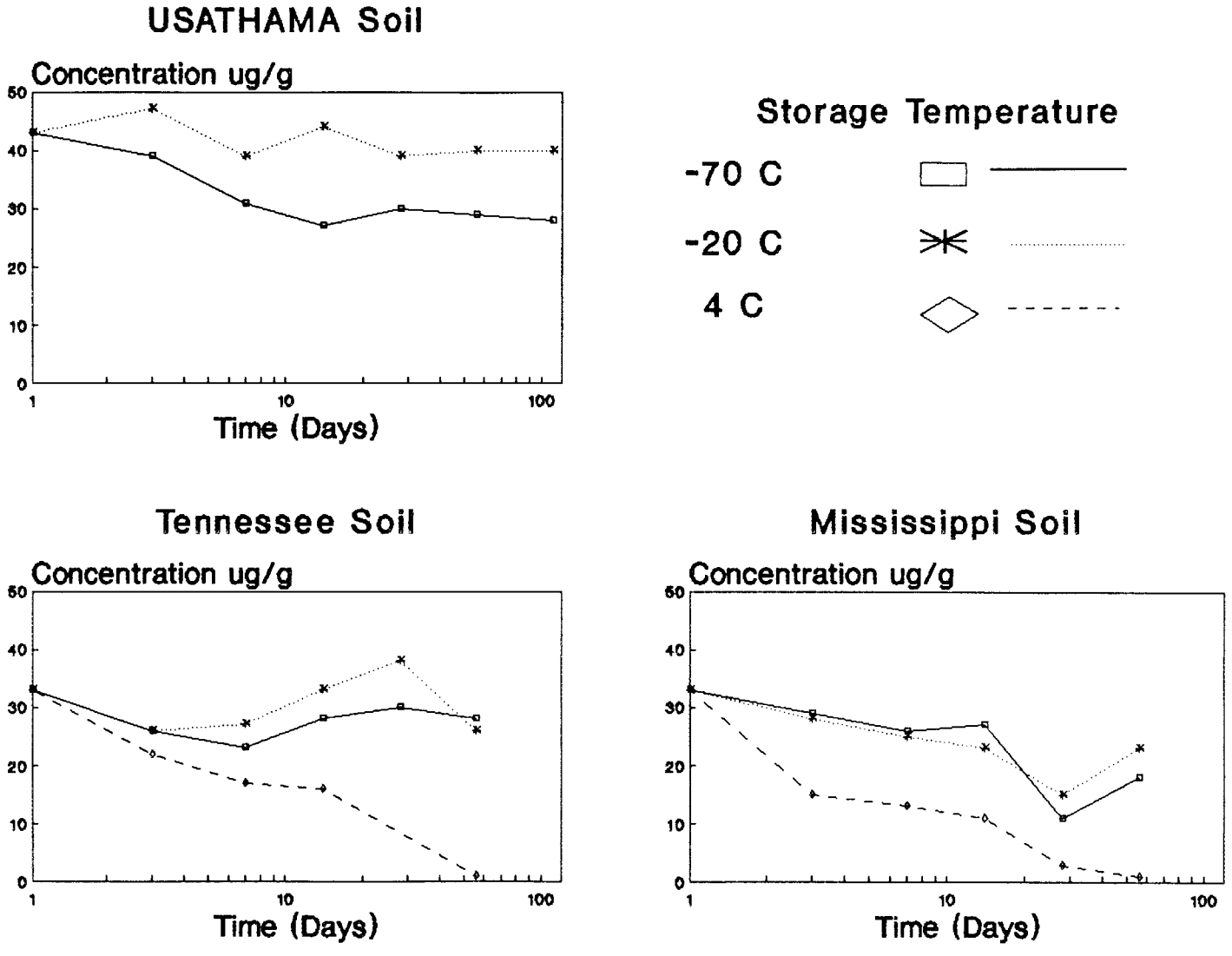


Fig. A.16 Chlorobenzene in soil samples.

Table A.17 Summary concentrations (ug/g) for Ethylbenzene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 54.7 | . | . | . | . | . | . | 54.7 |
| | | St Dev | 4.9 | . | . | . | . | . | . | 4.9 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 50.1 | 42.2 | 32.2 | 35.6 | 32.9 | 33.2 | 38.2 |
| | | St Dev | . | 4.8 | 2.3 | 4.7 | 2.8 | 2.6 | . | 7.7 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 59.3 | 51.7 | 53.5 | 46.9 | 48.0 | 44.4 | 50.5 |
| | | St Dev | . | 4.7 | 4.8 | 2.8 | 4.8 | 1.8 | 4.2 | 6.0 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 43.2 | . | . | . | . | . | . | 43.2 |
| | | St Dev | 3.0 | . | . | . | . | . | . | 3.0 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 33.4 | 27.8 | 29.2 | 30.7 | 26.4 | . | 29.5 |
| | | St Dev | . | 5.2 | 0.8 | 4.8 | 3.1 | 0.4 | . | 3.8 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 34.8 | 31.9 | 38.2 | 44.0 | 26.3 | . | 34.3 |
| | | St Dev | . | 6.1 | 2.6 | 0.4 | 0.4 | 6.9 | . | 7.4 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 27.9 | 20.3 | 12.9 | . | 1.2 | . | 14.4 |
| | | St Dev | . | 2.3 | 1.6 | 8.9 | . | 0.4 | . | 11.1 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 36.4 | . | . | . | . | . | . | 36.4 |
| | | St Dev | 4.2 | . | . | . | . | . | . | 4.2 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 31.0 | 23.2 | 23.9 | 9.1 | 14.1 | . | 19.2 |
| | | St Dev | . | 3.0 | 1.1 | 6.0 | 2.6 | 2.7 | . | 8.3 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 30.9 | 21.7 | 13.7 | 13.1 | 0.0 | . | 16.8 |
| | | St Dev | . | 5.4 | . | 11.9 | 2.1 | . | . | 11.6 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 17.1 | 13.4 | 9.8 | 2.6 | 2.9 | . | 6.8 |
| | | St Dev | . | . | 5.6 | 6.6 | 1.5 | 0.8 | . | 6.1 |

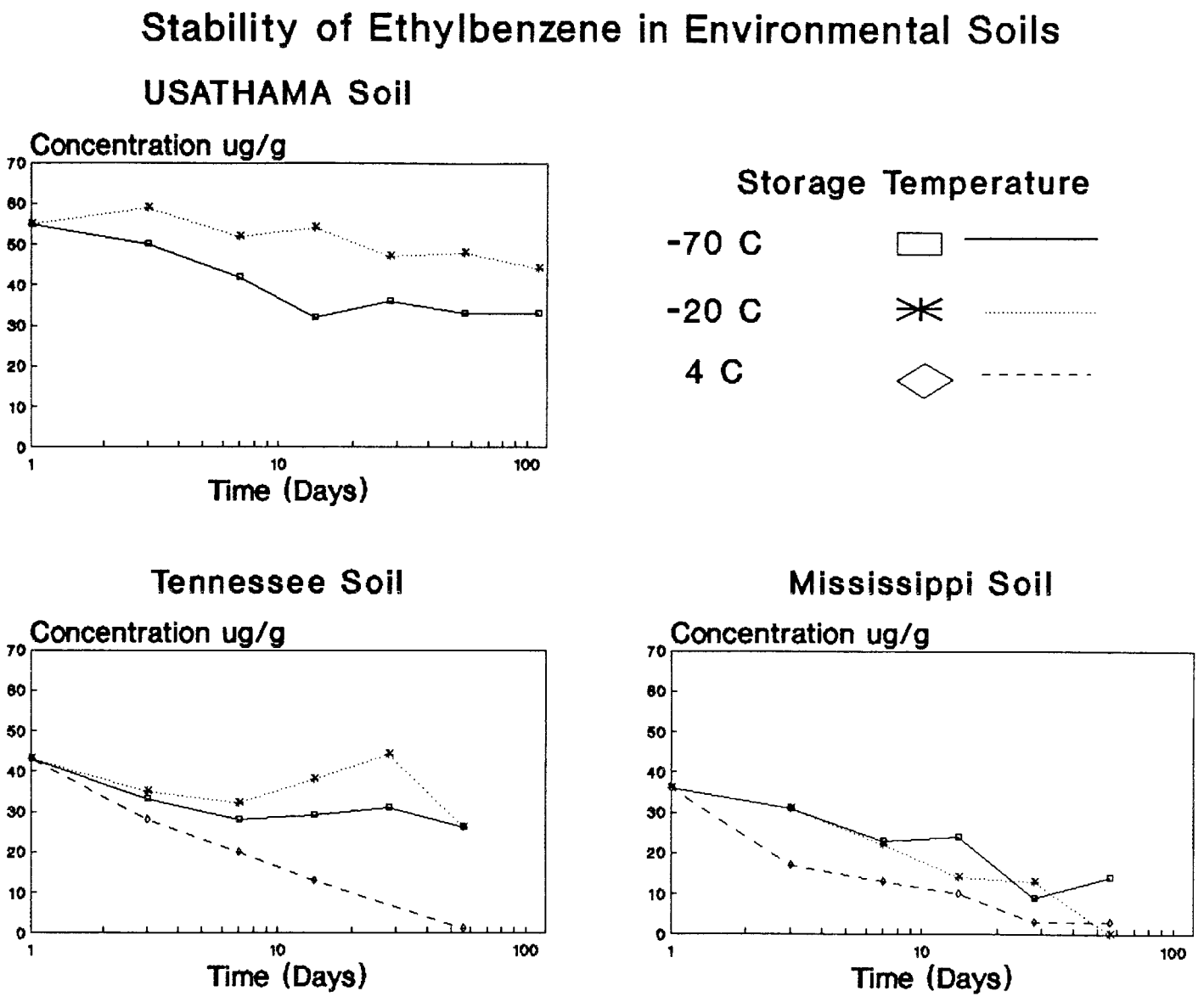


Fig. A.17 Ethylbenzene in soil samples.

Table A.18 Summary concentrations (ug/g) for Styrene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 36.7 | . | . | . | . | . | . | 36.7 |
| | | St Dev | 1.8 | . | . | . | . | . | . | 1.8 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 35.7 | 30.6 | 25.5 | 33.3 | 30.0 | 30.0 | 31.0 |
| | | St Dev | . | 2.6 | 1.2 | 2.4 | 2.9 | 1.1 | . | 4.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 36.6 | 31.3 | 30.1 | 33.6 | 33.9 | 31.9 | 32.6 |
| | | St Dev | . | 4.3 | 3.6 | 2.8 | 2.6 | 0.9 | 2.0 | 3.3 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 17.6 | . | . | . | . | . | . | 17.6 |
| | | St Dev | 3.6 | . | . | . | . | . | . | 3.6 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 19.0 | 14.8 | 22.8 | 23.5 | 14.3 | . | 19.2 |
| | | St Dev | . | 3.1 | 1.3 | 2.4 | 2.0 | 3.4 | . | 4.5 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 11.2 | 9.0 | 18.4 | 16.3 | 11.0 | . | 12.8 |
| | | St Dev | . | 2.9 | 0.3 | 3.1 | 0.3 | 2.2 | . | 4.0 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 7.8 | 4.4 | 0.9 | . | 0.0 | . | 2.9 |
| | | St Dev | . | 0.5 | 1.6 | 1.0 | . | 0.1 | . | 3.2 |
| Mississippi | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 16.1 | . | . | . | . | . | . | 16.1 |
| | | St Dev | 4.4 | . | . | . | . | . | . | 4.4 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 28.4 | 28.8 | 34.1 | 10.5 | 17.9 | . | 22.8 |
| | | St Dev | . | 4.2 | 1.0 | 7.0 | 2.1 | 5.2 | . | 9.5 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 19.4 | 20.3 | 15.1 | 12.5 | 0.0 | . | 14.4 |
| | | St Dev | . | 1.0 | . | 13.1 | 0.8 | . | . | 9.0 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 7.7 | 8.0 | 6.0 | 1.7 | 0.4 | . | 3.6 |
| | | St Dev | . | . | 3.6 | 4.9 | 1.0 | 0.5 | . | 3.8 |

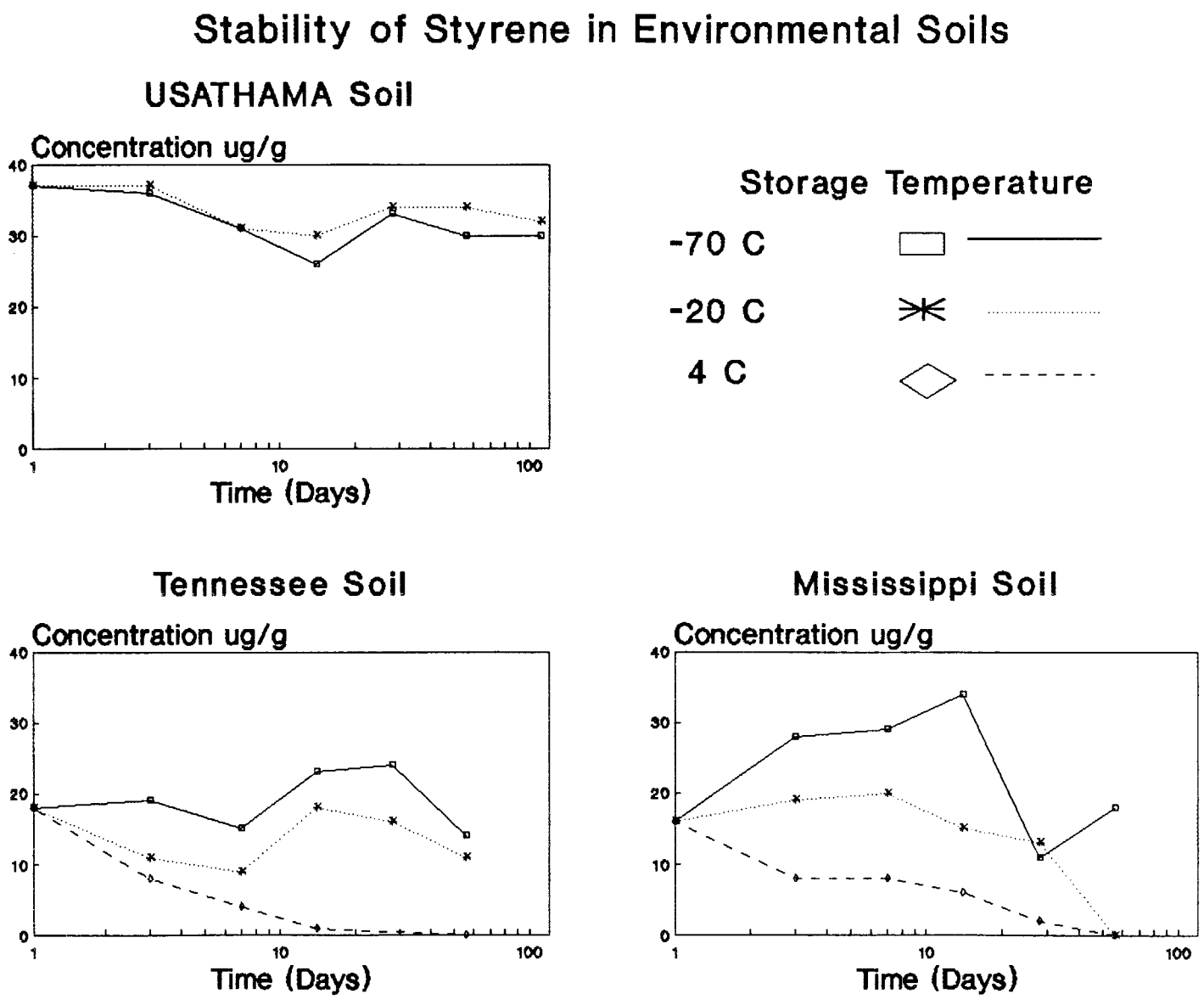


Fig. A.18 Styrene in soil samples.

Table A.19 Summary concentrations (ug/g) for o-Xylene.

| Soil Storage | | Num Avg St Dev | Day | | | | | | | All Days |
|---|---|---|---|---|---|---|---|---|---|---|
| Soil Type | Storage | | 0 | 3 | 7 | 14 | 28 | 56 | 111 | |
| USATHAMA | None | Num | 4.0 | . | . | . | . | . | . | 4.0 |
| | | Avg | 53.2 | . | . | . | . | . | . | 53.2 |
| | | St Dev | 3.9 | . | . | . | . | . | . | 3.9 |
| | -70°C | Num | . | 4.0 | 3.0 | 4.0 | 4.0 | 4.0 | 1.0 | 20.0 |
| | | Avg | . | 50.8 | 42.1 | 38.0 | 37.9 | 38.0 | 37.7 | 41.2 |
| | | St Dev | . | 3.6 | 3.4 | 4.8 | 3.1 | 2.6 | . | 6.0 |
| | -20°C | Num | . | 3.0 | 4.0 | 4.0 | 3.0 | 3.0 | 4.0 | 21.0 |
| | | Avg | . | 57.5 | 49.5 | 55.5 | 47.2 | 49.7 | 46.9 | 51.0 |
| | | St Dev | . | 2.8 | 3.6 | 2.4 | 4.4 | 1.2 | 3.2 | 4.9 |
| Tennessee | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 39.1 | . | . | . | . | . | . | 39.1 |
| | | St Dev | 1.8 | . | . | . | . | . | . | 1.8 |
| | -70°C | Num | . | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | . | 11.0 |
| | | Avg | . | 34.8 | 29.6 | 34.9 | 37.1 | 32.2 | . | 33.8 |
| | | St Dev | . | 3.1 | 0.4 | 3.3 | 2.4 | 0.3 | . | 3.3 |
| | -20°C | Num | . | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | . | 12.0 |
| | | Avg | . | 32.2 | 31.9 | 41.1 | 45.3 | 32.3 | . | 35.8 |
| | | St Dev | . | 6.6 | 1.1 | 2.3 | 1.5 | 6.9 | . | 7.0 |
| | 4°C | Num | . | 3.0 | 3.0 | 4.0 | . | 4.0 | . | 14.0 |
| | | Avg | . | 28.5 | 23.2 | 9.5 | . | 2.4 | . | 14.5 |
| | | St Dev | . | 2.9 | 1.0 | 10.9 | . | 0.4 | . | 12.0 |
| Mississippi | None | Num | 3.0 | . | . | . | . | . | . | 3.0 |
| | | Avg | 38.3 | . | . | . | . | . | . | 38.3 |
| | | St Dev | 3.7 | . | . | . | . | . | . | 3.7 |
| | -70°C | Num | . | 2.0 | 3.0 | 2.0 | 3.0 | 3.0 | . | 13.0 |
| | | Avg | . | 35.8 | 31.6 | 36.5 | 12.9 | 18.0 | . | 25.6 |
| | | St Dev | . | 3.5 | 1.8 | 8.6 | 2.6 | 4.8 | . | 10.7 |
| | -20°C | Num | . | 2.0 | 1.0 | 3.0 | 2.0 | 1.0 | . | 9.0 |
| | | Avg | . | 34.6 | 28.1 | 20.5 | 17.1 | 0.2 | . | 21.5 |
| | | St Dev | . | 5.0 | . | 17.8 | 2.5 | . | . | 13.9 |
| | 4°C | Num | . | 1.0 | 2.0 | 3.0 | 4.0 | 4.0 | . | 14.0 |
| | | Avg | . | 20.5 | 17.9 | 13.9 | 3.9 | 2.8 | . | 8.9 |
| | | St Dev | . | . | 5.6 | 7.6 | 2.2 | 0.6 | . | 7.8 |

# Stability of o-Xylene in Environmental Soils

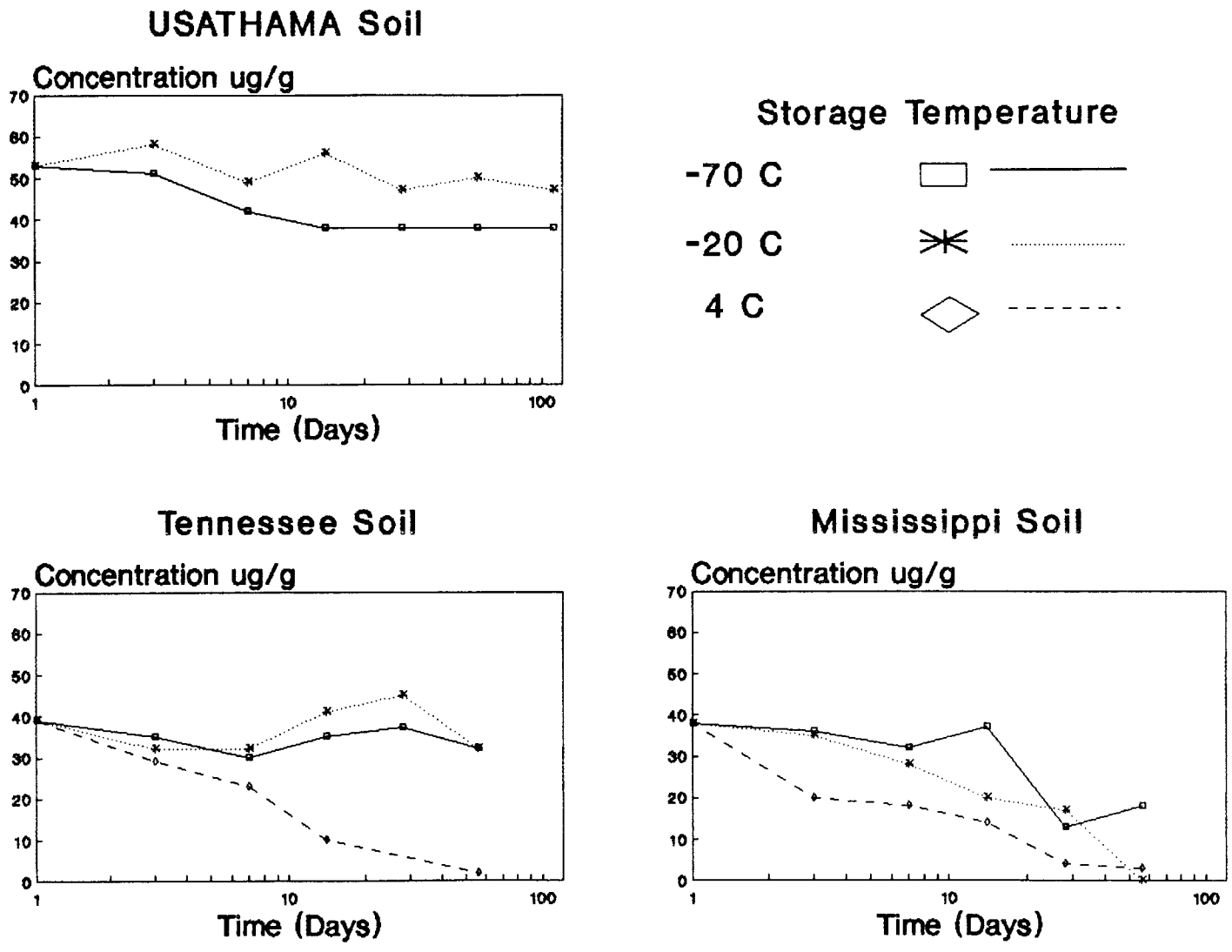


Fig. A.19 o-Xylene in soil samples.

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