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FINAL REPORT

**CHARACTERIZATION OF EMISSIONS FROM CARPET SAMPLES
USING A 10-GALLON AQUARIUM AS THE SOURCE CHAMBER**

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ABSTRACT

As part of Phase I of the carpet bioresponse study sponsored by the U.S. Environmental Protection Agency (EPA), a study was conducted to evaluate the emissions from carpet samples that had previously shown toxic effects on experimental mice as reported by Anderson Laboratories Inc., Dedham, MA in 1992. This document describes the major findings of the chemical characterization work conducted at the Indoor Source Characterization Laboratory of EPA's Air and Energy Engineering Research Laboratory (AEERL). All other results (animal testing, microbial testing, chemical analysis by sample extraction, and pesticide analysis) are reported separately.

The experimental system used in this study was first developed by Anderson Laboratories and was identical to the system EPA's Health Effects Research Laboratory (HERL) used in carpet bioresponse testing. Duplicate tests were conducted for each of three samples received from the Consumer Product Safety Commission (CPSC): two carpet samples plus mock samples (one empty bag and one bag of computer paper).

Toxicologists from HERL evaluated the carpet sample emissions data and concluded that the analytical results did not make a compelling case for a toxic exposure. The emissions characterization team from Acurex Environmental Corporation evaluated the experimental system and concluded that the test system developed by Anderson Laboratories was not suitable for carpet emissions characterization because of poor reproducibility, unusually high thermal conditions, and spurious emissions from the source chamber itself. The 1-h bake cycle prior to the dynamic mode is not typical of indoor air characterization methods.

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SECTION 1

INTRODUCTION

In 1992, researchers at Anderson Laboratories of Dedham, MA distributed data indicating irritancy and toxicity to mice exposed to emissions from heated carpet samples collected from sites with a history of indoor air complaints. The detailed chemical and physical parameters associated with their experiments, however, were unknown. The Indoor Air Branch of the U.S. Environmental Protection Agency's (EPA's) Air and Energy Engineering Research Laboratory (AEERL) collaborated with the EPA Health Effects Research Laboratory (HERL), Pulmonary Toxicology Branch, in an attempt to systematically reproduce Anderson Laboratories' test method and to provide an independent corroboration of Anderson's test results. EPA/AEERL conducted a thorough chemical, physical, and microbial characterization of the test sources (two carpets and an empty chamber), and HERL provided a comprehensive toxicity screen. This report documents the findings of chemical characterization of emissions from source samples tested and the physical characterization of the test method as performed by Acurex Environmental Corporation under EPA Contract No. 68-DO-0141, Technical Directive Nos. 93-170 and 93-111.

Chemical characterization of emissions from carpet samples is an essential step in establishing a correlation between observed toxic effects, if any, and the causative agent or agents. The study goals, reported herein, were limited to answering the following questions:

- Are any known toxic compounds observed as emissions from the carpet samples? And, if so, in what concentrations?

- Are any known toxic compounds observed as emissions from the source chamber itself?

And, if so, in what concentrations?

- Are qualitative and quantitative changes observed over the course of an experiment?

It should be emphasized that the analytical results reported in this document should be considered exploratory and preliminary for the following reasons: (1) the source chamber used in Phase 1 experiments (i.e., the 38-L aquarium) is not a conventional apparatus for source characterization, (2) the source chamber itself is a pollutant emitter, (3) in the source chamber, the carpet samples are heated unevenly to high temperatures not representative of indoor environments, and (4) the experimental conditions are difficult to control. Therefore, the results reported are not likely to occur in normal carpet use,

Because of the limited amount of time given for the analysis of Phase 1 data, only the data that answer the specific questions given in the test plan and restated above have been addressed.

SECTION 2

MATERIALS AND METHODS

2.1 DESCRIPTION OF CARPET SAMPLES TESTED

2.1.1 Sample Source

All tested carpet samples were received by an independent party (Acurex Environmental Task Lead) to ensure that the research teams were not aware of which tests had carpet in the chamber and which did not. Carpet samples were placed in blinded aquariums prior to testing, and the content code that revealed which experiments contained carpet was not given to the research team until after all data had been analyzed. The samples from the Consumer Product Safety Commission included two types of carpets and mock samples (empty bags). The two carpet samples collected by CPSC for EPA were from carpets that had previously been tested by Anderson Laboratories and had been shown to produce biological effects on laboratory animals when they were exposed to the emissions from heated carpet. Each of the samples (carpets and empty bags) were received at least 48 hours prior to testing in the aquarium systems. The carpet samples received from CPSC were packaged in single-layer, heat-sealed Tedlar bags with three, 0.093 m^2 (1-ft²) sections per bag. Each section was tagged with a bag and section number. There were three bags of carpet per box. The first set of samples was used in the EPA/AEERL chamber to determine chemical emissions, and the second set was used in the EPA/HERL chamber for bioresponse testing. The remaining bags' contents were subdivided into separate heat-sealed Tedlar bags for distribution to Research Triangle Institute (RTI)/Analytical and Chemical Sciences (ACS) for pesticide evaluation, RTI/Center for Environmental Analysis (CEA) for

microbial evaluation, and the EPA's Atmospheric Research and Exposure Assessment Laboratory (AREAL) for headspace, supercritical fluid extraction (SFE), and soxhlet extraction.

2.1.2 Installation

One day prior to testing, the carpet samples were removed from the sealed Tedlar bags and each section was weighed and measured before being sealed in the test aquarium. Each section was rolled so that two diagonal corners met and were tied together with a nylon tie. The rolled sections were placed in a clean, background tested aquarium with two sections on the bottom and one section on top of the other two in a pyramidal fashion. Such sample layout created many dead spaces in the source chamber. The aquariums were sealed and covered with duct tape to conceal the contents from the laboratory personnel conducting the experiments. Table 2-1 gives a summary of the test sources as received from CPSC. Each test system was tested for leaks by pulling 7 L/min zero-grade air through the system and measuring the inlet and outlet flow through the system. The difference of these two measurements was not to exceed 10 percent. After the systems were tested for leaks, each aquarium was sealed at both its inlet and outlet with a 14/23 sealed ball and socket joint and placed in the appropriate laboratory on the heating devices (heat off) for testing the next day.

TABLE 2-1. INSTALLATION SUMMARY

Experiment ID	Date Source Received	Date Source Installed	Source/Bag Identifications
1	03/05/93	03/08/93	Sample A ¹ /1292, 1410, 1747
2	03/09/93	03/10/93	Sample B Empty Bag/1768
3	03/19/93	03/22/93	Sample B Empty Bag ² /1399
4	03/23/93	03/24/93	Sample C ³ /1274, 1925, 1488
5	03/26/93	03/29/93	Sample C/1508, 1340, 1916
6	03/30/93	03/31/93	Sample A/1899, 1378 ⁴ , 1499

¹ Sample A: Dark pink, low pile, SBR backing, basically new with some household dirt.

² Experiment 3's empty bag contained computer paper.

³ Sample C: Indoor/outdoor dark blue with gray flecks, urethane backing, glue and plywood on backing, sections marked with an unknown marker source, samples contained large amounts of sand.

⁴ Section 50 in bag 1378 was noted to have an ~200 cm² area of water stain on the backing.

2.1.3 Determination of Sample Area and Volume

The carpet samples received were not exactly squares or rectangles. To calculate the actual sample area with better accuracy, the lengths of each side and one diagonal were measured. The diagonal divides the quadrilateral into two triangles, and the area of each triangle was calculated from Heron's formula:

$$\text{Area of Triangle} = [s (s - a) (s - b) (s - c)]^{0.5}$$

where a, b, and c are the lengths of the three sides and $s = (a + b + c)/2$.

The calculated sample area and volume (area multiplied by thickness) are given in Table 2-2. The approximate area of sample in contact with the heated chamber panel (as measured after loading) in the chamber is also given.

TABLE 2-2. CALCULATED SAMPLE AREA AND VOLUME

Test ID	Sample Bag Number	Section Numbers	Total Area (cm ²)	Contact Area (cm ²)	Total Vol. (cm ³)
1	1410	60, 47, 21	2950	520	3540
2	1768	N/A	N/A	N/A	N/A
3	1399	N/A	N/A	N/A	N/A
4	1925	27, 56, 65	2890	N/A ¹	1730
5	1508	5, 42, 21	2870	370	1720
6	1378	45, 12, 50	2960	520	3550

¹ Not measured.

2.2 EXPERIMENTAL SYSTEM

The experimental system for chemical characterization (Figure 2-1) consisted of four following functional parts: (1) the air supply system, (2) the source chamber (i.e., the aquarium), (3) heating and insulation devices for the source chamber, and (4) the exposure chamber. Teflon tubing with 1-cm ID was used to connect the air supply, the source chamber, and the exposure chamber. The experimental set-up and operational conditions were similar to those used for bio-response studies conducted by

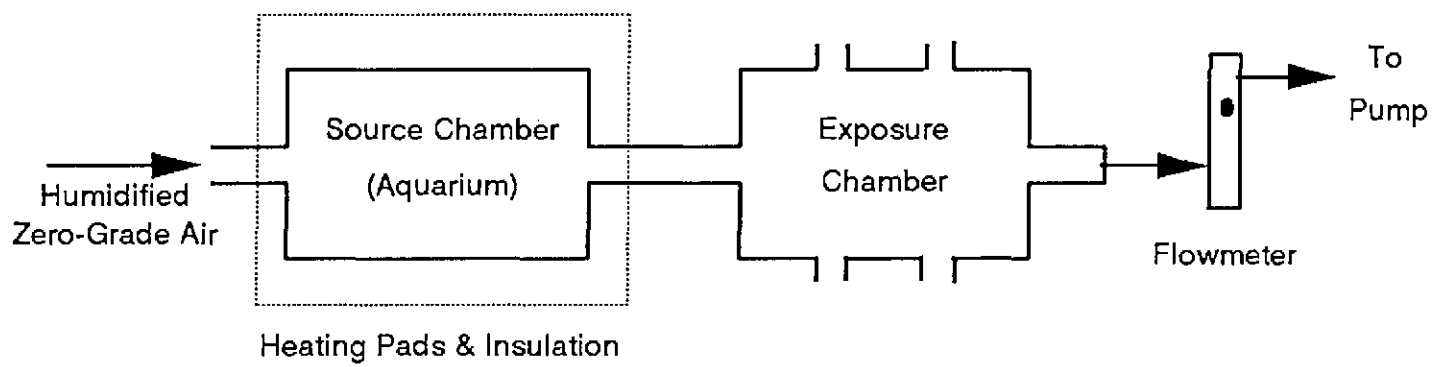


Figure 2-1. The experimental system.

EPA/HERL except that no experimental animals were put in the exposure chamber. Instead, the mouse exposure ports (P1, P2, P6, and P7 in Figure 2-2) were used to collect air samples.

Each of the six tests consisted of four one-hour exposure periods and took two days to complete. The following is a brief discussion of the test procedure:

- Step 1. The day prior to testing, CPSC supplied sources were placed into the source chamber.
- Step 2. On day 1, the initial static mode, the source chamber was sealed, then heated to and maintained in desired temperature ranges. There was no air flow through the chamber during this stage. The samples were baked under such static conditions for about one hour.
- Step 3. During the first one-hour dynamic mode (i.e., the first exposure period), the source chamber was connected to the exposure chamber and humidified zero-grade air was pulled through the system with a vacuum pump for one hour.
- Step 4. During the two-hour static mode between two exposures, after the first exposure, the air flow was cut off and the source chamber was disconnected from the exposure chamber and sealed again. The source chamber was allowed to stay in the static mode for two hours with the heating system on.
- Step 5. During the second one-hour dynamic mode (the second exposure period), the same procedure as in step 3 was followed.
- Step 6. During the experimental pause overnight, after the second exposure, the air flow was again cut off, the source chamber disconnected from the exposure chamber, sealed, and the heating system turned off. On the second day, steps 2 through 5 were repeated to complete the third and fourth exposure periods.

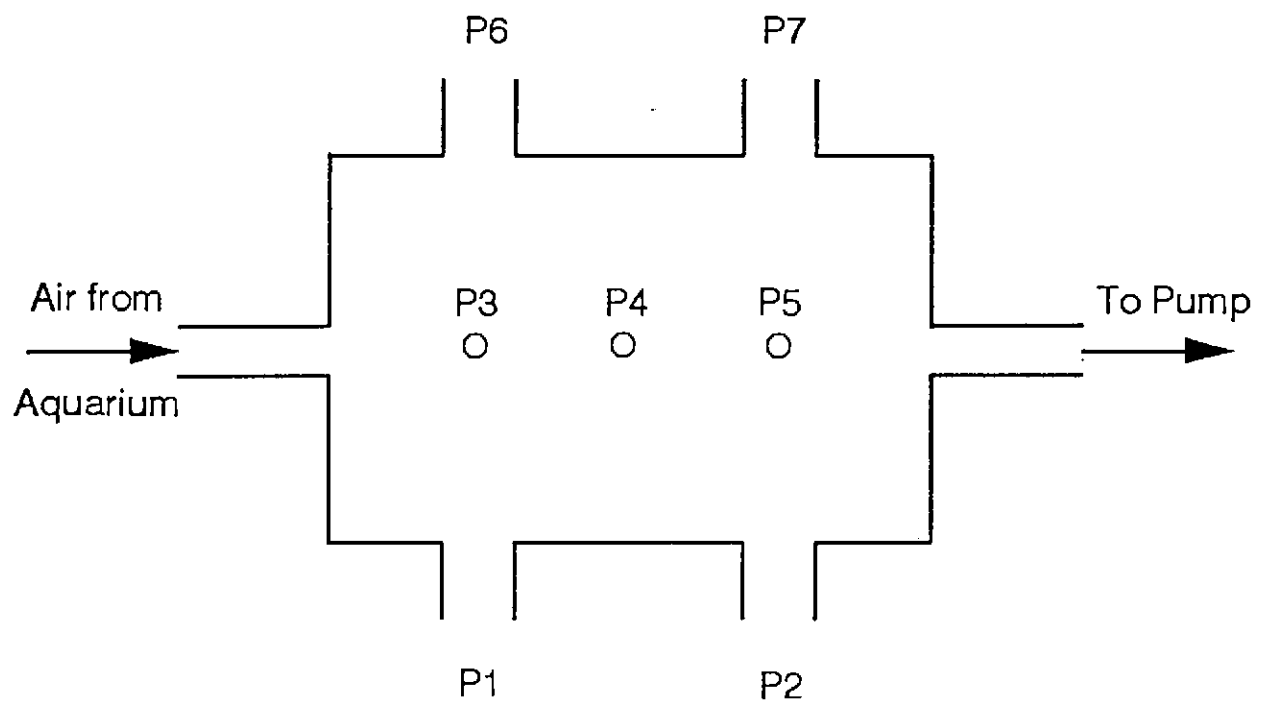


Figure 2-2. Sampling locations in the exposure chamber.

2.3 PREPARATION OF THE SOURCE CHAMBER

The source chambers used were 10-gal (38-L) glass aquariums made by All Glass Aquarium Co., Inc., Franklin, WI, and purchased from a local store. Side and bottom panels were 3-mm thick glass plates and the top was 5-mm thick. The outside dimensions of the aquarium were 20 by 10 by 12.5 in (50.8 by 25.4 by 31.8 cm). Four aquariums were used in the six tests.

The aquariums were first prepared by removing the plastic rim using a hot air gun and a knife. Excess silicone adhesive was removed with razor blades and precision knives. The aquariums were then baked overnight at test temperature conditions with a 7 L/min laboratory air flush to remove excess adhesive vapors. Before a test, the aquarium was washed with a Liquinox detergent solution and rinsed with deionized water. The aquarium was air dried or dried with a low lint tissue wipe for immediate use.

In all tests, the aquarium was turned on its side so that the pedestal was facing the wall and designated the "back panel," and the opening was facing the exposure chamber and designated the "front panel." The two largest panels (12.5 by 20 in) then became the top and the bottom. The front panel (i.e., the 10- by 20- by 0.19-in glass cover) has two 0.5-in ID nylon bulkhead fittings in diagonal corners 3 in from each side. An additional 0.25-in ID nylon bulkhead fitting was added for temperature measurement. The cover was attached to the aquarium with duct tape (the Original Brand B-600, Manco Inc., Westlake, OH) and covered with the same type of duct tape to conceal the contents.

2.4 AIR SUPPLY AND HUMIDITY CONTROL

The air supply used in the tests was zero-grade compressed air, which is a synthetic blend of nitrogen and oxygen from Air Products & Chemicals, Inc. (APCI), Research Triangle Park, NC. The Certificate of Zero Grade Air provided by APCI indicated that both total volatile organic compound (TVOC) and water contents were certified to less than 0.1 ppm.

The air humidification was achieved by passing part of the air flow through an impinger—a 1,000-mL flask containing about 600 mL of deionized water (Ion Pure mixed bed/Millipore water system), as seen in Figure 2-3. Two mass flow controllers (Tylan Model FC-260) were used for flow control, and Weathertronics Model 101A humidity probe was used to monitor the relative humidity. The desired humidity of $50\% \pm 10\%$ was achieved by adjusting the ratio of the dry/wet air flows. The actual dry/wet flow ratio was about 1:1.

2.5 AIR FLOW AND PRESSURE CONTROL

The air flow through the experimental system was driven by the positive pressure from the air supply and a vacuum pump downstream from the exposure chamber. To keep the inside pressure close to the ambient atmospheric pressure, the air flow from the gas cylinder was set to a rate higher than the air flow through the exposure chamber. This ensured that excess air was constantly released in the room through a T-tube between the humidifier and the source chamber. A typical flow balance was as follows:

Humidified air from air supply	9-10 L/min
Outlet air flow of exposure chamber	7 L/min
Air flow for two sorbent tube samples	0.3 L/min
Air flow for particle counter	0.7 L/min
Excess air flow	1-2 L/min

For each exposure, the air flow rates were measured at two locations with a precalibrated rotameter: the inlet flow of the source chamber and the outlet flow of the exposure chamber.

The pressure inside the exposure chamber was measured by HERL during all the tests with a magnehelic (Dwyer Instrument Co.) pressure gauge.

2.6 TEMPERATURE CONTROL

The main goal of temperature control was to create temperature profiles in the source chamber as close as possible to those found in Anderson Laboratories' experimental systems. Based on the

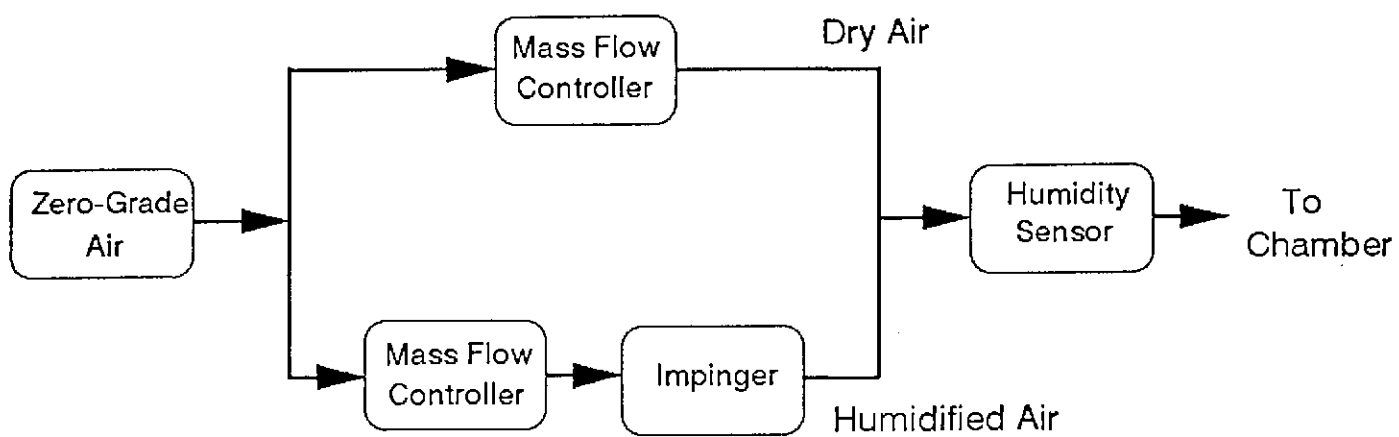


Figure 2-3. The air humidifying system.

actual measurements at Anderson Laboratories (January 4, 1993), the targeted operational parameters were set to the following:

Bottom outside surface of the source chamber	70 ± 5 °C
Air in source chamber	37 ± 3 °C
Air in exposure chamber	24 ± 2 °C

The temperature inside the source chamber was created and maintained by two heating pads outside the aquarium: a Sunbeam model E12107 pad under the bottom and a Sunbeam model HT-1 pad on the top. The heating intensity could be adjusted by the heating pad control systems.

During an experiment the temperature was continuously monitored at 12 locations (Table 2-3). The position descriptions such as "left," "right," "front," and "back" were defined assuming the observer was standing near the exposure chamber and facing the source chamber. Temperature data from the 12 locations were collected by Cole-Palmer Model 92800-00 Scanning Thermocouple Thermometer at a frequency of one reading every minute and logged by a computer.

TABLE 2-3. LOCATIONS OF TEMPERATURE SENSORS

Sensor ID	Sensor Type	Location
1	Air	Inside the aquarium
2	Air	Room air
3	Air	Exposure chamber (front)
4	Air	Exposure chamber (back)
5	Surface	Left glass panel of the aquarium (outside/center)
6	Surface	Right glass panel of the aquarium (outside/center)
7	Surface	Top glass panel of the aquarium (outside/center)
8	Surface	Bottom glass panel of the aquarium (outside/center)
9	Surface	Back glass panel of the aquarium (outside/center)
10	Surface	Front glass panel of the aquarium (outside/center)
11	Surface	Carpet sample (backing side)
12	Surface	Carpet sample (fiber side)

Surface temperatures were measured with K-type, fast-response surface thermocouples and air temperature with Teflon-coated, K-type thermocouples.

2.7 TEST OF AIR MIXING IN THE EXPOSURE CHAMBER

Imperfect air mixing may affect the transport of emissions from the source to each of the experimental animals. To test whether exposures at the four ports were equivalent, the air mixing pattern in the exposure chamber was characterized by using SF₆ tracer gas. The results of this experiment are presented in Section 3.2.1. A calculated amount of SF₆ was injected into the source chamber without a carpet sample. With an air flow rate of 7 L/min, the tracer concentrations were measured at the four exposure ports with a Brüel & Kjær (B&K) Type 1302 Multi-gas Monitor. The optical filter used for measuring SF₆ was UA 0988, which has the center wavelength of 10.6 µm with a half-power bandwidth of approximately 0.4 µm. A detection limit of 5 ppb is reported by the manufacturer.

The results from the analysis of duplicate sorbent tubes collected at different exposure chamber ports help to validate the mixing of the source chamber emissions. These results are found in Sections 3 and 4.

2.8 AIR SAMPLING AND ANALYSIS FOR ORGANIC COMPOUNDS

2.8.1 Sorbents

The relatively low levels of volatile organic compound (VOC) emissions from carpet samples required preconcentration prior to chemical analysis. ST032 multibed sorbent traps were used for this purpose. ST032 traps (T.R. Associates Inc.) are fabricated of 6 mm OD by 203 mm long silanized borosilicate glass tubing sequentially packed with a fritted glass disk, 290 mg of 20/30 mesh silanized glass beads, 85 mg of 20/35 mesh Tenax TA, 170 mg of 35/60 mesh Amborsorb XE-340, and 48 mg of 80/100 mesh activated charcoal. This sampling media allows quantification of volatile and semivolatile compounds ranging from C₄ to C₁₆ with a satisfactory collection and desorption efficiency.¹

Before use, sorbent tubes were conditioned in sets of six using the Envirochem Model 785 Sorbent Tube/Trap Conditioner. The tubes were connected to the conditioner, and a heating sleeve was placed over each respective tube. A purge flow of ca. 50-60 mL/min of UPC nitrogen gas was begun 5 min prior to the heating of the sorbent tubes. The tubes were then heated to 350 °C. The tubes were allowed to remain at this temperature and under the nitrogen purge flow for 20-25 min. After this period, the heat was turned off and the tubes were allowed to cool to 50 °C at which time they were removed from the desorber using lint-free nylon gloves and placed in their respective vials. The vials were then placed in their appropriately labelled PTFE bags and sealed using an impulse bag sealer. To evaluate the "cleanliness" of the conditioned tubes, one tube from each set of six was randomly chosen and desorbed/run on the GC under the same conditions as a sample would be. If the total mass resulting from the GC run was less than 40 ng, then that tube and the other five tubes in the set of six were considered as having passed QC.

2.8.2 Sampling Procedure

Immediately before each one-hour exposure period, three air samples were drawn directly from the source chamber to determine the accumulated organic pollutant concentrations that resulted from heating under the static mode. For tests 1-3, static chamber samples were collected at a volume of 0.1 L using a Samplair vacuum pump. This volume was determined to be inadequate for quantitative analysis by gas chromatography, since the peaks were below the detection limit of the instrument. Therefore, 1-L samples were collected using mass flow controllers for tests 4-6. This volume was the largest that could be justified without jeopardizing the integrity of the static chamber emissions. The dilution of

the static air containing the accumulated carpet emissions in the source chamber was less than 10 percent.

During each exposure, the time-concentration dependence was monitored by sequentially taking samples from the mouse ports in the exposure chamber by using an air pump at a flow rate of either 50 mL/min or 150 mL/min to collect sorbent trap samples. The sampling flow rate was controlled by a mass flow controller.

When the sampling was completed, the trap was put back in the glass vial and sealed in Teflon bags. The samples were stored in a freezer at approximately -10 °C until analysis (up to 21 days).

2.8.3 Analytical Instruments

Two gas chromatograph (GC) systems were used in this work. Table 2-4 describes both analytical systems.

TABLE 2-4. DESCRIPTION OF ANALYTICAL SYSTEMS

Analytical Systems	Envirochem I (EC I)	Envirochem II (EC II)
GC	Hewlett-Packard (HP) model 5890, series II	HP model 5890
GC column	J&W Megabore DB-5	J&W Capillary 0.32 mm DB-5
Multitube desorber	Envirochem model 8916	Envirochem model 8916
Concentrator	Unacon model 810	Unacon model 810
Flame ionization detector (FID)	HP model 19231-60010	HP model 19231-60010
Mass spectrometric detector	Not equipped	HP model 597 (MSD)

2.8.4 Analysis

The primary identification of individual compounds from the sources used in this study was the responsibility of RTI/ACS. However, Acurex Environmental provided backup identifications from samples collected for analysis using the HP MSD on EC II. Compound identification was done using an electronic database search of the NBS 43K mass spectral library and the NIST/EPA/NIH mass spectral library for personal computers. Further manual review of the data was performed using the Alderman 8-Peak Compound Index EPA/NIH Spectra Data Base to verify computer library searches and to identify compounds not found during the search.

The analytical results for specific VOCs will be reported at the following three levels:

- Level 1. A compound in samples that has previously been analyzed as a standard by the laboratory, and a retention time and mass spectra exist that match the sample compound as reviewed by the analyst.
- Level 2. A compound that has not been previously identified by a match with retention time of a known standard but has a good library match as reviewed by the analyst. The concentration will be reported "as toluene."
- Level 3. The compound is not individually identified by MS but is confirmed as a class such as alkanes or isomers of a compound. These compounds will be reported as the class name and the concentration reported "as toluene."

The initial list of identifications from RTI was given to the HERL to mark any compounds that had known toxic effects. The following compounds were requested by HERL for quantitation and evaluation of their behavior over the course of the experiments:

1. Methylene chloride

2. Perchloroethylene
3. Benzaldehyde
4. Methylnaphthalene
5. Acetic acid
6. Benzene
7. Naphthalene
8. Butylatedhydroxytoluene (BHT)

All of these compounds were evaluated first for their maximum concentrations and feasibility of quantitation over the duration of the study. Methylene chloride was noted as a system contaminant and therefore was eliminated from the list. Perchloroethylene was found only in trace amounts in Sample A. Benzaldehyde coeluted with another compound and was difficult to identify and quantify. Methylnaphthalene was identified by RTI; however, this particular compound could not be identified in the retention time range by Acurex Environmental. 4-phenylcyclohexane (4-PCH) was also added to this list of compounds because it is a known carpet emission. On the MSD system, 4-PCH was identified by ion extraction from a coeluting siloxane compound and found in trace amounts in both carpet samples and none in the empty chamber. Acetic acid, benzene, naphthalene, and BHT were all found in quantifiable concentrations throughout all of this study. These compounds comprise the major focus of the individual compound analysis for this study.

Quantitative analysis was made by using both GC systems with FID. All concentrations were based on the response factor for toluene and reported at the following three levels: (1) TVOCs; (2) TVOCs divided into three groups—molecular weight $<C_8$, C_8-C_{12} , and $>C_{12}$; and (3) selected individual organic compounds.

Quantification of individual compounds required retention time correlation of known marker compounds (toluene and alkanes) between the MSD/FID output from EC II and the FID output from EC I. Manual review of the chromatograms for matching peak shapes and patterns was also performed to ensure the best match to the identified compound.

2.9 AIR SAMPLING FOR ANALYSIS OF CARBONYL COMPOUNDS

DNPH-Silica Sep-Pak cartridges purchased from Waters were used to sample formaldehyde and other carbonyl compounds from chamber air. The effective reagent, 2,4-dinitrophenylhydrazine (DNPH), in the cartridge reacts with the aldehydes and ketones to form hydrazone derivatives. DNPH samples were taken from one of the mouse ports of the exposure chamber at a sampling flow rate of 400 mL/min for the duration of the dynamic mode.

The DNPH cartridge samples collected were then sent to RTI/ACS for extraction and subsequent reverse-phase HPLC analysis. The analytical results will be reported separately by RTI in a Final Report.

2.10 SAMPLING AND ANALYSIS OF AEROSOLS

The instrument used for monitoring particle concentration was a model 8010 PortaCount portable counter (TSI, Inc.). Designed to count individual airborne particles, this instrument is based on a miniature, continuous-flow condensation nucleus counter (CNC) and is sensitive to particles having diameters as small as 0.02 μm , but insensitive to variations in particle size, shape, composition, and refractive index.² In this work, the instrument was operated in the "Count Mode," in which the instrument directly counts the aerosol drawn through the sample port and gives the concentration in particles per cubic centimeter (P/cm^3). The instrument can measure particle concentration between 0 and $5 \times 10^5 \text{ P}/\text{cm}^3$. The

counting results can be taken either manually from its display or automatically through a computer.

Air samples were taken from the top of the exposure chamber through sampling port P4 (see Figure 2-2). The sampling flow rate was 0.7 L/min. For comparison purposes, the particle concentrations in the laboratory air were also measured before and after each exposure period.

SECTION 3

RESULTS

3.1 EXPERIMENTAL SUMMARY

A total of six tests were conducted for the three samples received from CPSC with each being tested in duplicate. Four aquariums were used in the six tests. This was necessary because of the test schedule and breakage. An experimental summary is given in Table 3-1.

TABLE 3-1. EXPERIMENTAL SUMMARY

	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
Experiment ID	1	2	3	4	5	6
Chamber ID	AQ2	AQ4	AQ4	AQ3	AQ4	AQ1
Sample ID	A	B	B	C	C	A
Test start date	03/09	03/11	03/23	03/25	03/30	04/01
Test finish date	03/10	03/12	03/24	03/26	03/31	04/02

A total of 148 ST032 sorbent traps and 30 DNPH cartridges were taken during the six tests for chemical analysis. This number excludes field blanks, laboratory blanks and background samples. A detailed sampling scheme is given in Appendix A. Table 3-2 shows the distribution of ST032 traps. All the DNPH cartridges were sent to RTI for analysis of carbonyl compounds and the results will be reported by RTI in a separate report.

TABLE 3-2. DISTRIBUTION OF ST032 SORBENT TRAPS¹

Number of Traps	Purpose	Analyzed by
14	Qualitative analysis	RTI
26	Qualitative analysis	Acurex Environmental
99	Quantitative analysis	Acurex Environmental

¹ Nine traps were lost before or during analysis.

3.2 MONITORING RESULTS OF EXPERIMENTAL CONDITIONS

3.2.1 Air Mixing in the Exposure Chamber

The air mixing in the exposure chamber was determined prior to Test 1. Normal air flow was maintained during the test. SF₆ tracer gas was introduced into the source chamber at a constant rate. After equilibrium was established, the tracer concentrations were measured at five locations, and the results are given in Table 3-3. The difference of average concentrations between the two mouse ports was less than 10 percent.

TABLE 3-3. CONCENTRATIONS OF SF₆ TRACER GAS MEASURED IN DIFFERENT LOCATIONS (IN mg/m³)

Sampling Location	Mean	RSD (%)
Manifold between aquarium and exposure chamber	9.94	2
Inside the aquarium	9.72	2
Mouse port 1 of exposure chamber ¹	8.56	4
Mouse port 2 of exposure chamber ²	9.29	2
Center of exposure chamber ³	9.72	1

¹ Mouse port 1 is marked "P1" in Figure 2-2.

² Mouse port 2 is marked "P2" in Figure 2-2.

³ This sampling port is marked "P4" in Figure 2-2.

Sampling of duplicate volume sorbent traps at different mouse ports confirms the mixing of the effluent from the source chamber in the exposure chamber. Table 3-4 gives a summary of the

results from three selected duplicate analyses. A complete report of all duplicate samples is presented in Section 4 of this document.

TABLE 3-4. TVOC CONCENTRATIONS AT DIFFERENT MOUSE PORT
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Sample ID	Mouse Port	TVOC Conc.	Mean	RSD
3105	Port 1	164		
3106	Port 2	200	182	14%
3315	Port 1	2470		
3316	Port 4 ¹	2750	2610	8%
3522	Port 1	2320		
3523	Port 4 ¹	2070	2200	8%

¹ Mouse port 4 is marked "P7" in Figure 2-2.

3.2.2 Air Pressure in the Exposure Chamber

The air pressure inside the exposure chamber was measured before the first sample testing under experimental air flow condition (7 L/min). The source chamber was empty and unheated. Slight negative pressure was observed inside the exposure chamber. The pressure difference between laboratory air and exposure chamber was in the range of 0.050-0.075 in of water (0.09-0.14 mm Hg). This measurement was performed during all mouse exposures by the bioassay laboratory and remained constant throughout the study.

3.2.3 Temperature

The temperature ranges for all the six tests are given in Table 3-5. An example of temperature profiles for a complete test is graphically shown in Figures 3-1 through 3-6. The average temperatures measured at 12 locations (see Section 2.6 for description) during each test are given in Appendix B.

TABLE 3-5. TEMPERATURE RANGES AT 12 LOCATIONS FOR ALL THE TESTS (IN °C)

Thermocouple Location	Mean	Range
Air in source chamber	41	36 ~ 46
Laboratory air	23	21 ~ 24
Air in exposure chamber (front port ¹)	27	23 ~ 29
Air in exposure chamber (back port ²)	25	23 ~ 26
Left panel of source chamber	44	39 ~ 50
Right panel of source chamber	43	38 ~ 48
Top panel of source chamber	48	44 ~ 54
Bottom panel of source chamber	72	66 ~ 76
Back panel of source chamber	46	37 ~ 52
Front panel of source chamber	32	29 ~ 36
Sample backing (inward) ³	50	40 ~ 57
Sample fiber (outward) ^{3,4}	68	59 ~ 72

¹ Marked "P3" in Figure 2-2.

² Marked "P5" in Figure 2-2.

³ Excluding Test 2 and Test 3 (no carpet sample).

⁴ Temperature of sample surface in contact with heated chamber bottom.

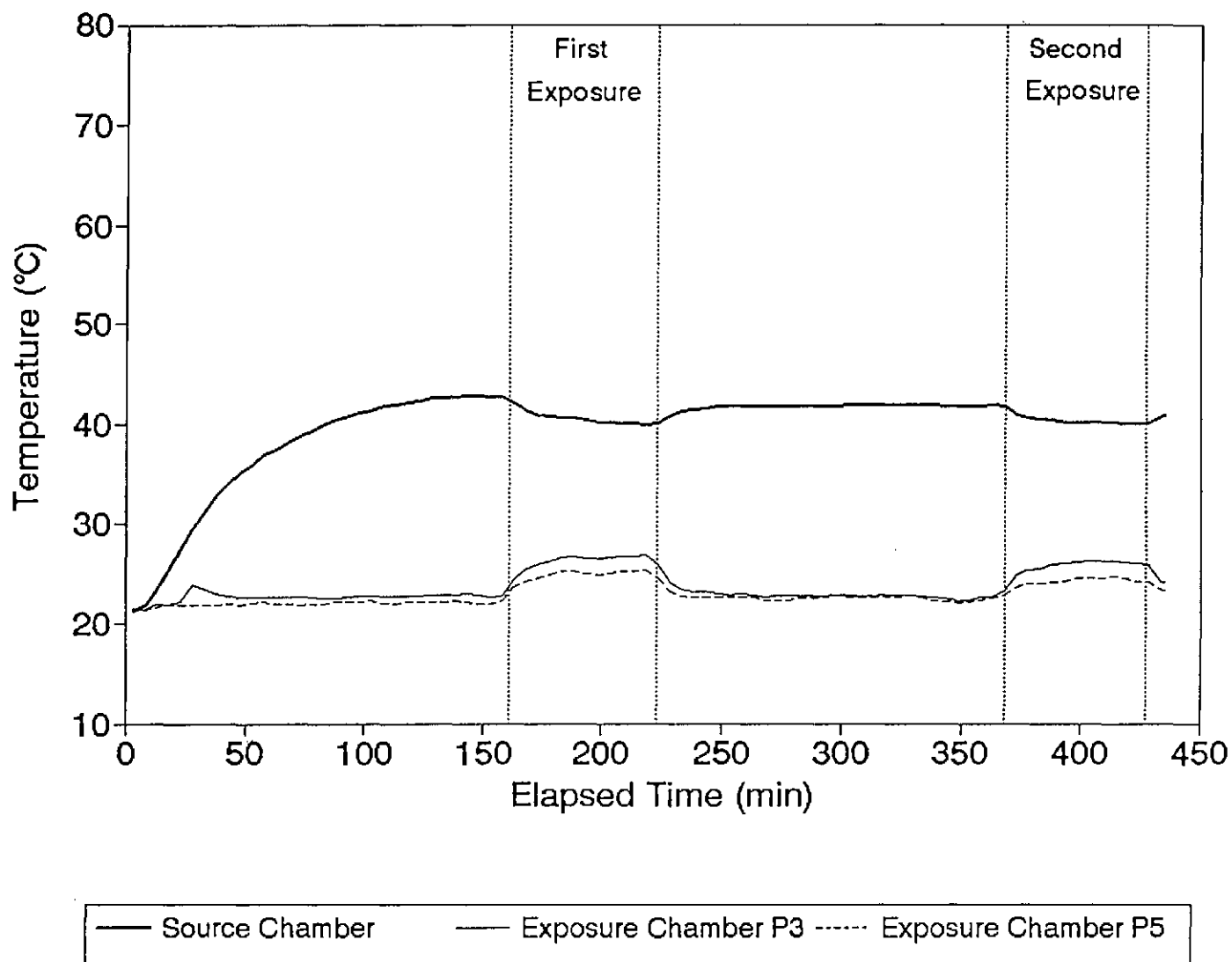


Figure 3-1. Temperature profiles for Test 4; Air temperature on Day 1.

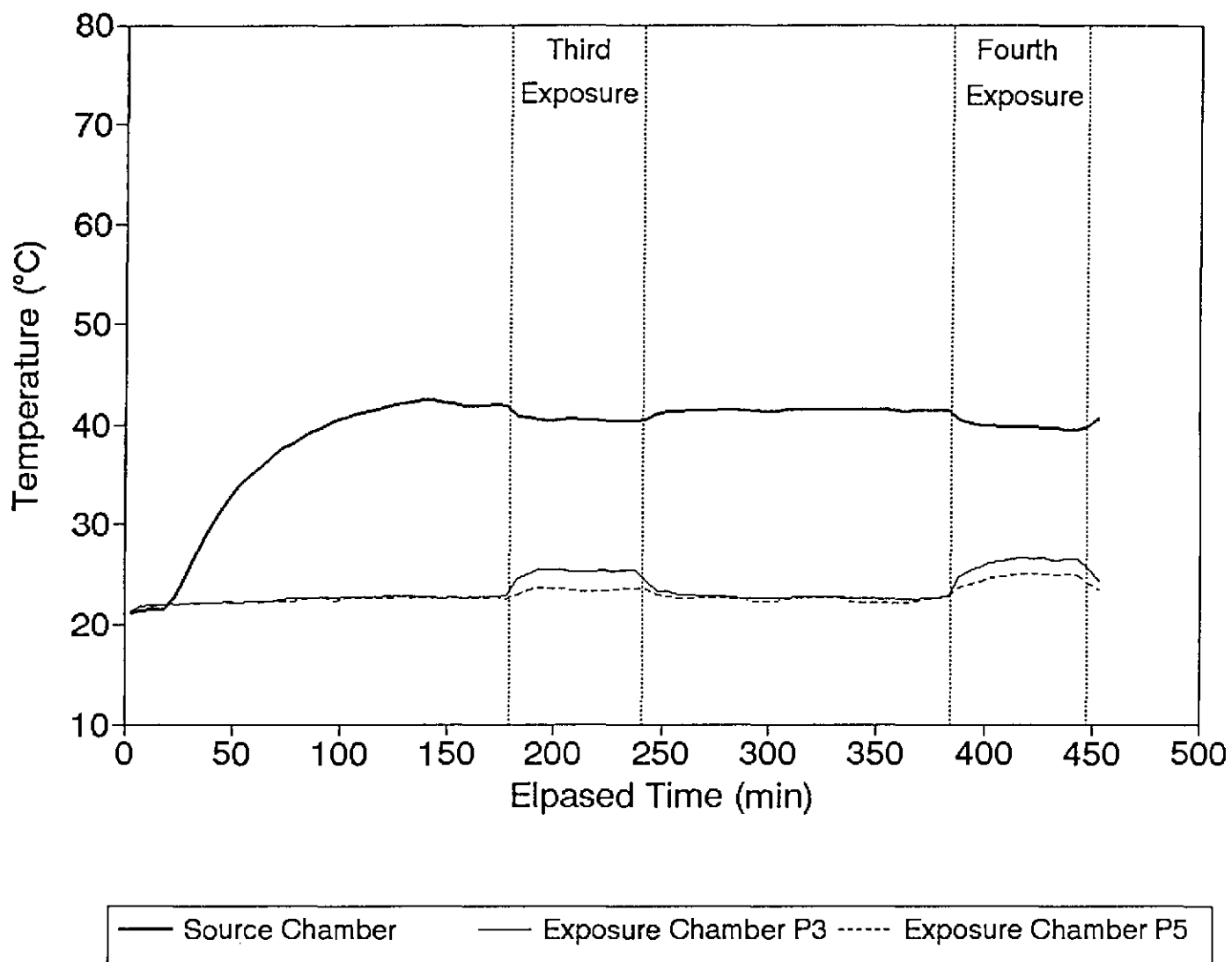


Figure 3-2. Temperature profiles for Test 4; Air temperature on Day 2.

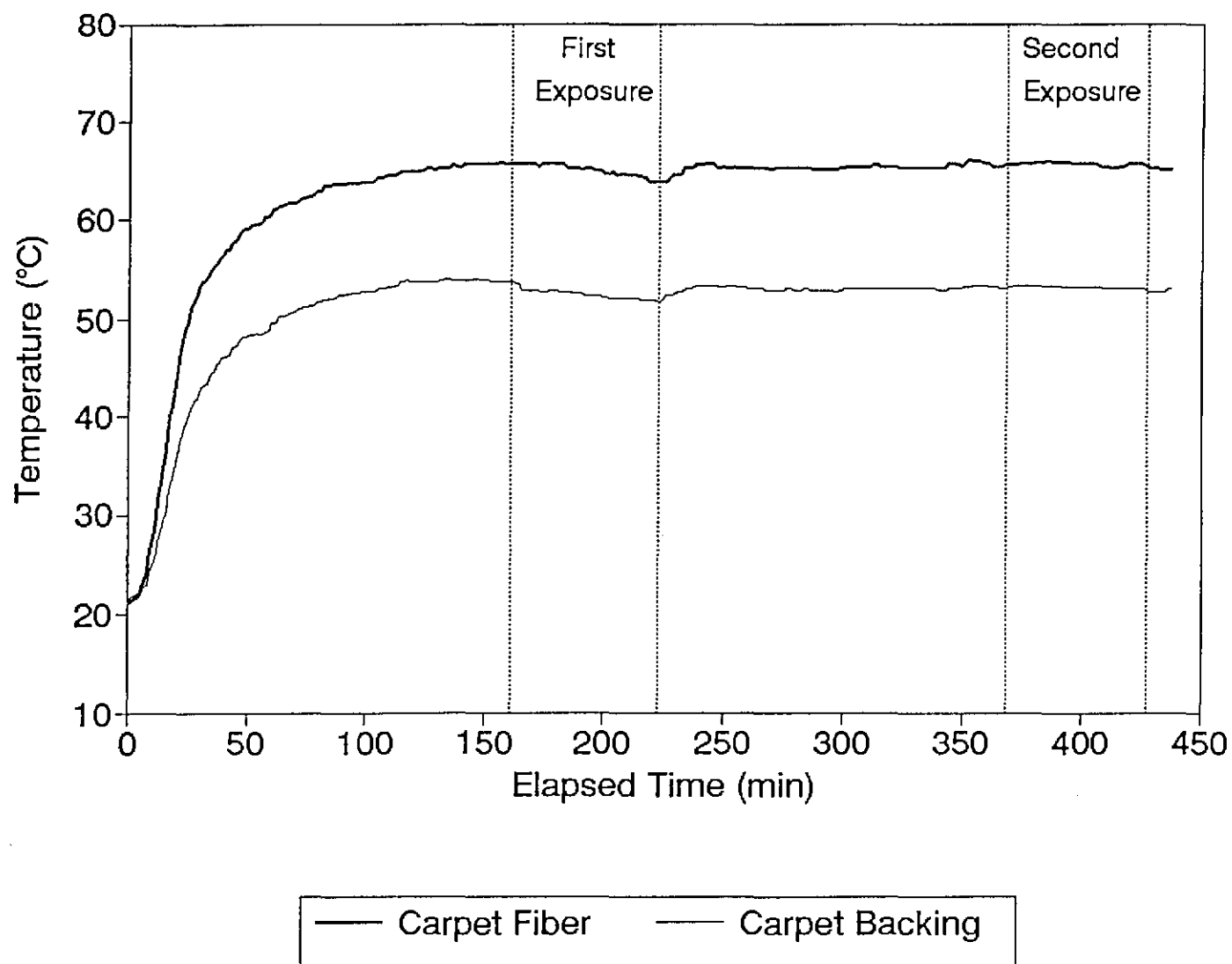


Figure 3-3. Temperature profiles for Test 4; Sample surface temperature on Day 1.

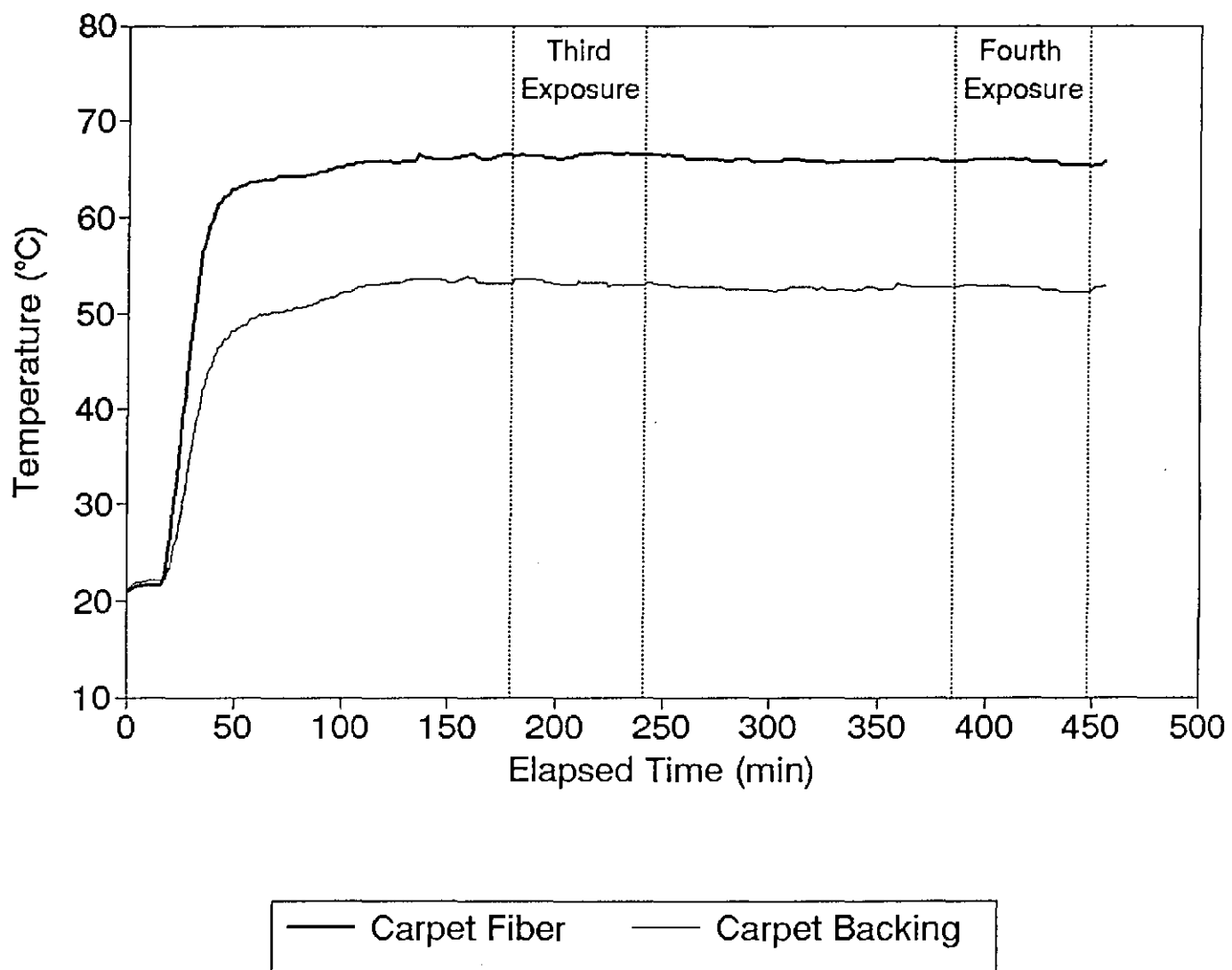


Figure 3-4. Temperature profiles for Test 4; Sample surface temperature on Day 2.

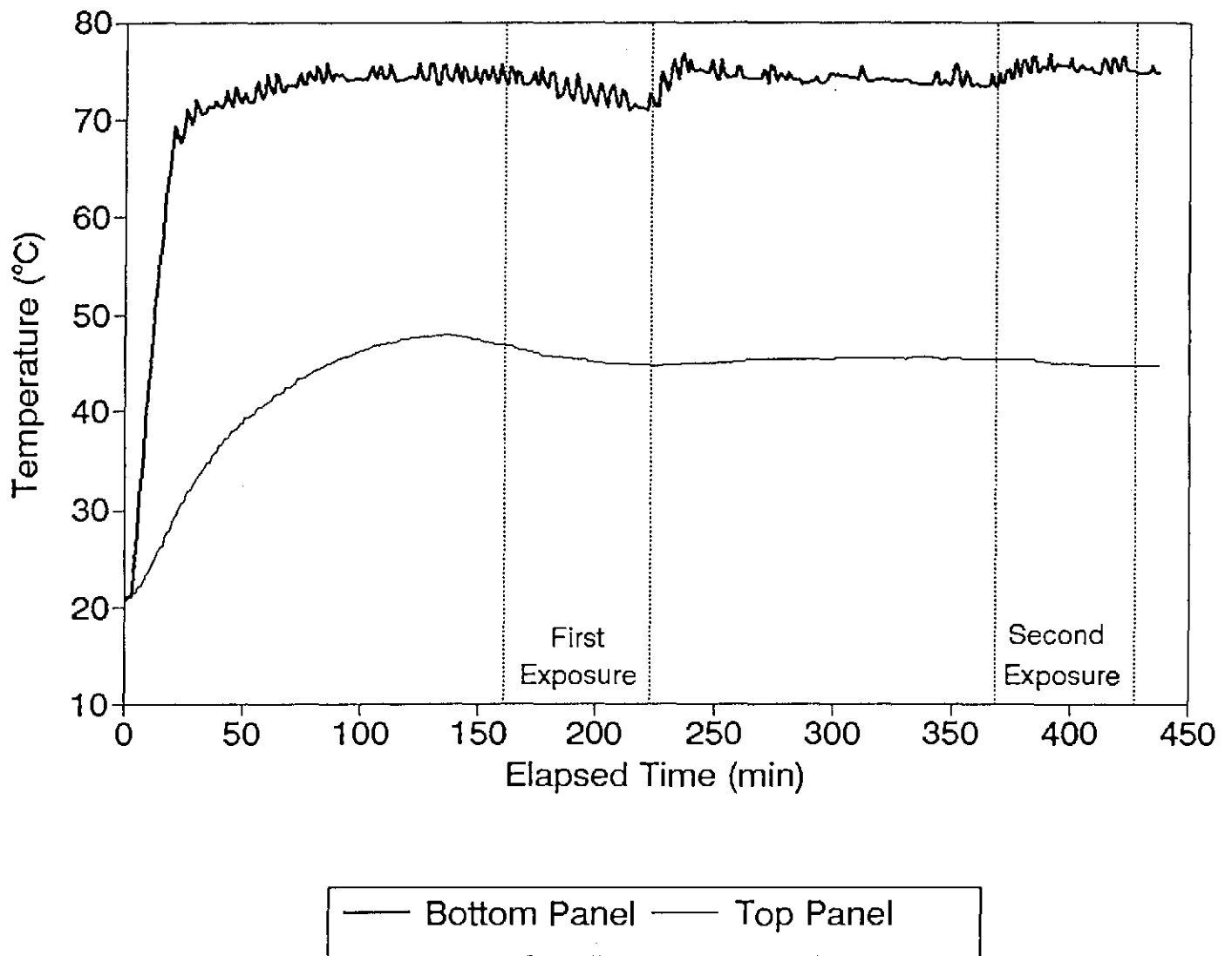


Figure 3-5. Temperature profiles for Test 4; Chamber panel temperature on Day 1.

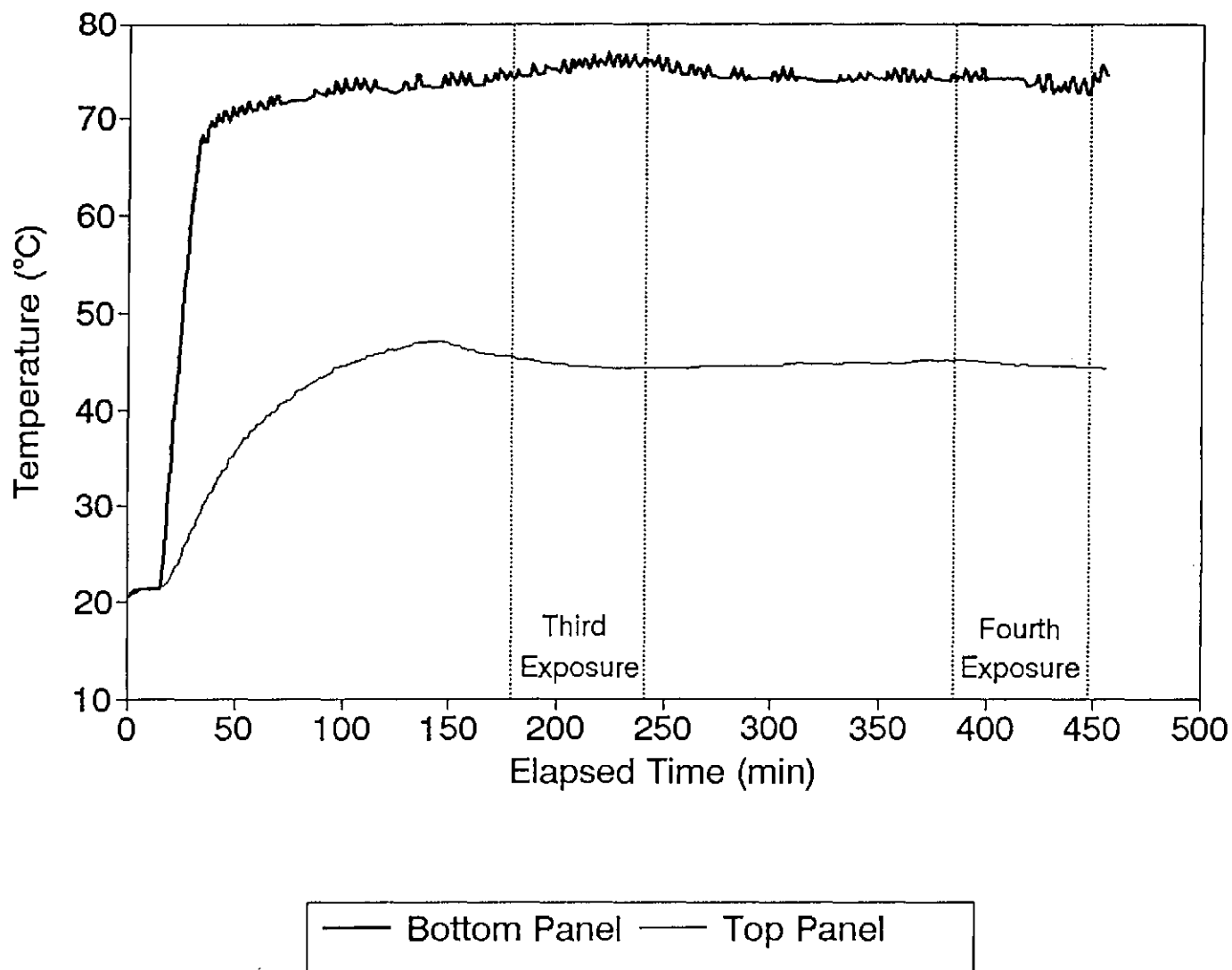


Figure 3-6. Temperature profiles for Test 4; Chamber panel temperature on Day 2.

3.2.4 Humidity

The desired relative humidity for humidified air was 50 percent. The actual measured humidity varied between 41 and 59 percent (Table 3-6). Deviations from the target value are all within 10 percent with an average deviation of 2.3 percent.

TABLE 3-6. MEASURED RELATIVE HUMIDITY FOR INLET AIR

Date	Time	Test ID	Exposure ID	Measured RH (%)
03/09/93	08:45	1	1	43.1
03/09/93	14:06	1	2	48.6
03/10/93	09:00	1	3	51.5
03/10/93	14:09	1	4	45.3
03/11/93	08:55	2	1	49.7
03/11/93	13:59	2	2	48.8
03/12/93	06:39	2	3	52.4
03/12/93	13:22	2	4	47.6
03/23/93	08:30	3	1	51.3
03/23/93	13:38	3	2	59.3
03/24/93	08:56	3	3	50.2
03/24/93	13:30	3	4	49.9
03/25/93	08:15	4	1	50.2
03/25/93	13:15	4	2	50.2
03/26/93	14:00	4	3	47.9
03/26/93	07:50	4	4	52.4
03/30/93	08:48	5	1	52.4
03/30/93	14:27	5	2	48.8
03/31/93	08:36	5	3	52.0
03/31/93	15:02	5	4	54.5
04/01/93	08:12	6	1	50.6
04/01/93	13:12	6	2	49.3
04/02/93	07:26	6	3	54.5
04/02/93	12:33	6	4	49.5

3.2.5 Air Flow Rate

Before the start of each experiment, the inlet and outlet flow rates were measured to make sure the difference between the two flow rates was within 10 percent (Table 3-7).

During an experiment, the outlet flow was checked prior to each exposure. Results in Table 3-8 shows that the outlet flow was well controlled. The inlet flow, however, had more substantial changes during the experiment (Table 3-9). This may have been caused by the increased leakage in the source chamber as a result of continuous heating. Experiment 4 was an extreme case, in which the inlet flow was reduced by 68 percent after four exposures.

TABLE 3-7. THE INLET AND OUTLET FLOW RATES BEFORE TESTING STARTED (L/min)

Experiment ID	Outlet Flow (L/min)	Inlet Flow (L/min)	Percent Difference
1	7.20	6.70	6.9
2	7.28	6.73	7.6
3	7.25	6.70	7.6
4	7.20	6.75	6.3
5	7.14	6.90	3.3
6	6.90	6.35	8.0

TABLE 3-8. THE OUTLET FLOW RATES MEASURED DURING THE EXPERIMENT (L/min)

Experiment ID	Exposure 1	Exposure 2	Exposure 3	Exposure 4
1	7.08	7.08	6.45	6.95
2	6.98	6.95	7.04	6.84
3	7.09	6.95	7.01	6.88
4	6.98	6.99	6.95	6.90
5	6.76	6.65	7.14	7.01
6	6.83	6.84	7.07	7.11

TABLE 3-9. THE CHANGE OF INLET AIR FLOW DURING AN EXPERIMENT (L/min)

Experiment ID	Flow After Exposure 2	Percent Reduction ¹	Flow After Exposure 4	Percent Reduction ¹
1	N/A	N/A	6.11	9
2	6.49	4	5.95	12
3	N/A	N/A	5.79	14
4	N/A	N/A	2.17	68
5	6.03	13	5.9	14
6	5.04	21	4.85	24

¹ Percent reduction was calculated based on the initial inlet flow rate (Table 3-7) to 100%.
N/A = data not available.

3.3 SAMPLE WEIGHT LOSS AFTER EXPERIMENT

The samples were weighed before and after the test. The change of sample weights is shown in Table 3-10.

TABLE 3-10. THE WEIGHT CHANGES OF SAMPLES AFTER TEST

Test ID	Weight Before Test (g)	Weight After Test (g)	Weight Change (g)
1	764	742	-22
4	544	539	-5
5	533	525	-8
6	807	804	-3

3.4 QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS

The qualitative analysis of the heated emissions from each of the sources tested in this study proved to be a challenge. More than two hundred compounds existed in the carpet emissions. Automated library searches with the NBS 43K mass spectral library were of limited success for most of the samples because of the number of compounds (up to 300) detected in each of the sources tested. Therefore, each ion chromatogram was manually scanned and evaluated for the major emissions from each of the study sources. The specific compounds requested by HERL were given the highest

priority for in-depth evaluations. A comprehensive identification of the emissions from each of the sources will be reported by RTI/ACS in a separate report. The correlation of findings from both laboratories (RTI/Acurex Environmental) were ensured by the analysis of an even n-alkane standard that was prepared by Acurex Environmental for daily QC checks for all three analytical systems. An early evaluation of each laboratory's identifications of the source emissions showed no disagreement. Among more than 200 peaks in the chromatograms, about 15% have been identified and confirmed by interlaboratory comparison so far, another 70% were tentatively identified, and the remaining 15% remained unknown. Figure 3-7 shows a typical chromatogram from each of the tested sources. These chromatograms are typical of static chamber emissions from the sources used in this study. Test 2, Sample B shows a typical emissions spectrum from an empty aquarium with a TVOC value of 1,200 $\mu\text{g}/\text{m}^3$ from a 0.1-L volume sample. Tests 5 and 6 show representative emissions from the carpet sources with TVOC values greater than 6,000 $\mu\text{g}/\text{m}^3$ from 1-L samples.

Table 3-11 gives the list of compounds identified by Acurex Environmental from each of the test sources. All compounds listed in this table are considered Level 2 identifications with the exceptions of toluene and styrene which are Level 1. Table 3-12 shows the classes of compounds found in the emissions from each of the sources.

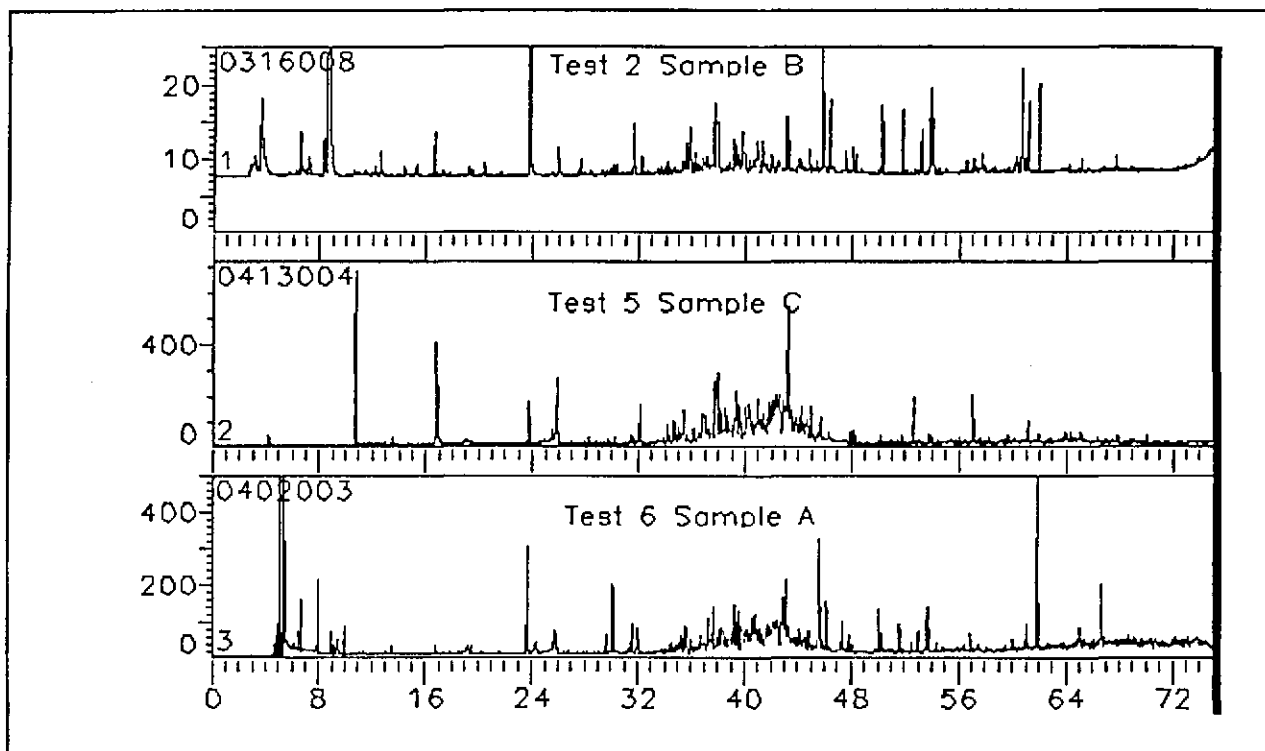


Figure 3-7. Representative chromatograms from test sources.

TABLE 3-11. INDIVIDUAL COMPOUNDS IDENTIFIED IN THE THREE SAMPLES

Compound Name	Sample A	Sample B	Sample C
Acetone	+	+	+
Isopropanol	+	+	+
Benzene	-	-	+
Acetic acid	+	-	+
Toluene	+	+	+
Hexanal	+	-	+
Ethylbenzene	+	-	+
m,p-Xylene	+	-	+
N,N-Dimethyl-acetamide	+	-	+
Styrene	+	-	+
o-Xylene	+	-	-
α -Pinene	+	-	+
Benzaldehyde	+	-	+
Decane	+	-	-
Trimethylbenzene	+	-	-
Limonene	+	-	+
Acetophenone	-	-	+
Terpene	+	-	+
Undecane	+	-	-
n-Dodecene	-	-	+
Camphor	+	-	-
Naphthalene	+	-	-
Dodecane	+	-	+
Dodecamethylcyclhexasiloxane	+	+	+
4-Phenylcyclohexene	+	-	+
Butylatedhydroxytoluene	+	+	+
Hexadecane	+	-	-
Butanoic acid	+	-	-
2,3-Dihydro-1,1,3-trimethyl-3-phenyl-1H-indene	-	-	+

TABLE 3-12. CLASSES OF COMPOUNDS IDENTIFIED IN THE THREE SAMPLES

Class Name	Sample A	Sample B	Sample C
Alkanes	+	-	+
Alkenes	+	-	+
Cycloalkanes	+	-	+
Cycloalkenes	+	-	+
Oxygenated hydrocarbons	+	+	+
Substituted benzene	+	+	+
Siloxanes	+	+	+
Substituted phenol	+	-	+

3.5 QUANTITATIVE ANALYSIS OF ORGANIC COMPOUNDS

3.5.1 TVOCs and Grouped VOCs

TVOC concentration data are presented in Tables 3-13 through 3-18. Before the start of air flow through the source chamber for each exposure of each experiment, static chamber samples were collected to determine the maximum concentration for that exposure. For any given experiment, during exposures 1 and 3, the time-concentration dependence was monitored by sequentially collecting samples at 0-5 min, 5-20 min, 20-40 min, and 20-60 min. The sampling flow rate was consistent at 150 mL/min for all six experiments. During exposures 2 and 4, simultaneous 60-min samples were collected to represent the average concentration of the VOCs from the carpet emissions. The flow rate for the 60-min samples varied from 150 mL/min for experiments 1, 2 (exposure 2), 3, and 4 (exposure 2) to 50 mL/min for experiments 2 (exposure 2), 4 (exposure 4), 5 and 6. After analysis of the first set of samples, the sample volume of the 60-min sample was determined to be too large for the amount of volatiles detected on the sorbent. Therefore, the flow rate was reduced to 50 mL/min. The mass flow controllers used for air sampling were calibrated individually. The flow rates used were close to each other but not exactly the same; this was not considered a problem. In addition, TVOCs in each sample is divided into three groups based on the retention times of alkane markers. Group 1 includes the light compounds with retention time less than octane, group 2 includes the

compounds in the intermediate range with a retention time between and including octane through dodecane, and group 3 includes the heavier compounds with retention times greater than dodecane. Quantification limits and detection limits are given in Section 4.5.1.

Figure 3-8 is an example of decaying pattern of TVOC concentrations during an exposure. The dotted line in the figure is the simple first-order decay (i.e., exponential decay) curve.³ The difference between the two curves is an indication of continuous TVOC emissions during the exposure period (see Section 5.4 for further discussion).

TABLE 3-13. CONCENTRATIONS OF TVOCs AND GROUPED VOCs FOR TEST 1
(SAMPLE A)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	>C ₁₂
1	2977	0.10	static	8790	7310	1070	434
1	2979	0.75	0 to 5	7610	5200	1730	681
1	2980	2.20	5 to 20	3010	1290	1060	665
1	2982	2.98	20 to 40	1820	437	737	644
1	2985	5.80	20 to 60	1380	281	526	569
2	2999	0.10	static	2810	BQL	918	665
2	3001	0.10	static	BQL	BQL	424	456
2	2995	9.05	0 to 60	1890	261	715	915
2	2997	8.76	0 to 60	1580	222	618	744
3	3055	0.10	static	2300	BQL	503	721
3	3058	0.10	static	2040	BQL	582	509
3	3059	0.80	0.2 to 5.3	2570	841	1030	698
3	3047	2.20	5.3 to 20.3	1730	292	706	728
3	3048	3.00	20.3 to 40.3	1320	120	456	742
3	3049	6.00	20.3 to 60.3	1190	86.9	404	696
4	3033	0.10	static	BQL	ND	589	734
4	3034	9.20	0 to 60.3	1650	90.7	566	992
4	3065	8.90	0 to 60.3	1560	81.5	552	921

ND = Not detected

BQL = Below quantification limit

TABLE 3-14. CONCENTRATIONS OF TVOCs AND GROUPED VOCs FOR TEST 2 (SAMPLE B)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	>C ₁₂
1	3030	0.10	static	BQL	BQL	ND	42.2
1	3006	3.00	20.2 to 45.2	86.8	BQL	14.3	42.4
1	3007	5.90	20.2 to 60.2	90.8	BQL	17.1	46.7
2	3101	0.10	static	BQL	ND	BQL	BQL
2	3103	0.10	static	BQL	ND	BQL	BQL
2	3105	9.00	1 to 61	164	43.6	31.4	87.8
2	3106	8.80	1 to 61	201	54.5	39.4	106
3	3147	0.10	static	BQL	ND	BQL	BQL
3	3129	0.76	2.8 to 7.8	1030	475	294	251
3	3118	2.20	7.8 to 22.8	243	83.0	57.7	99.2
3	3117	2.96	22.8 to 42.8	78.6	ND	15.3	57.4
3	3115	6.00	22.8 to 62.8	83.9	BQL	16.1	54.7
4	3109	0.10	static	BQL	ND	ND	ND
4	3112	3.30	0.6 to 67.4	162	BQL	32.4	100
4	3113	3.40	0.6 to 67.4	129	BQL	28.5	75.5

ND = Not detected

BQL = Below quantification limit

TABLE 3-15. CONCENTRATIONS OF TVOCs AND GROUPEd VOCs FOR TEST 3 (SAMPLE B)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	>C ₁₂
1	3233	0.10	static	BQL	BQL	346	2040
1	3229	0.76	1 to 6	1400	679	341	377
1	3231	2.27	6 to 21	466	227	114	125
1	3209	2.99	21 to 41	BQL	BQL	BQL	BQL
1	3210	6.02	21 to 61	110	33.9	19.1	57.7
2	3237	0.10	static	BQL	ND	ND	BQL
2	3221	0.10	static	BQL	ND	ND	152
2	3222	9.05	0.3 to 60.3	112	31.6	18.4	61.8
2	3225	8.76	0.3 to 60.3	117	38.5	19.4	59.8
3	3256	0.10	static	BQL	ND	BQL	242
3	3257	0.10	static	BQL	ND	BQL	ND
3	3261	0.74	0.2 to 5.3	210	BQL	38.1	79.5
3	3249	2.25	5.3 to 20.2	74.4	BQL	10.7	30.2
3	3250	2.99	20.2 to 40.2	27.7	ND	ND	12.4
3	3252	5.95	20.2 to 60.2	55.3	BQL	10.1	24.7
4	3253	0.10	static	BQL	ND	ND	ND
4	3240	8.90	1.3 to 61.3	75.5	18.5	13.4	43.8
4	3228	8.90	1.3 to 61.3	71.5	20.5	11.4	39.8

ND = Not detected

BQL = Below quantification limit

TABLE 3-16. CONCENTRATIONS OF TVOCs AND GROUPED VOCs FOR TEST 4
(SAMPLE C)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	>C ₁₂
1	3287	1.00	static	12000	2730	8270	1030
1	3289	0.75	1.5 to 6.5	9240	1820	6660	765
1	3291	2.26	6.5 to 21.5	5590	609	4140	843
1	3305	2.97	21.5 to 41.5	4070	218	2960	892
1	3306	6.02	21.5 to 61.5	3180	161	2180	836
2	3281	1.00	static	7580	367	5630	1590
2	3282	0.99	static	6100	435	4820	844
2	3285	8.60	0.7 to 60.7	1890	148	1530	218
3	3318	1.00	static	7220	836	5430	954
3	3319	0.99	static	7300	797	5400	1110
3	3327	5.96	20.3 to 60.3	1180	121	808	260
4	3330	1.00	static	5840	304	4550	985
4	3315	3.09	0.4 to 61	2470	89.0	1760	619
4	3316	3.03	0.4 to 61	2750	92.9	1810	844

TABLE 3-17. CONCENTRATIONS OF TVOCs AND GROUPED VOCs FOR TEST 5
(SAMPLE C)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	>C ₁₂
1	3369	1.00	static	6500	723	4780	986
1	3372	0.74	0.3 to 5.3	7040	728	5420	879
1	3373	2.24	5.3 to 20.3	4260	304	3310	634
1	3383	2.97	20.3 to 40.3	3220	139	2430	646
1	3384	6.02	20.3 to 60.3	2770	88.5	2000	680
2	3387	0.99	static	7070	532	5680	841
2	3388	0.98	static	7730	600	6010	1100
2	3354	3.02	0.3 to 60.2	3180	90.1	2370	719
2	3355	2.89	0.3 to 60.2	1010	695	261	54.3
3	3501	1.02	static	4950	485	3680	777
3	3502	1.01	static	5030	518	3770	731
3	3505	0.75	0.4 to 5.4	4100	339	3070	680
3	3507	2.29	5.4 to 20.4	2740	93.3	2000	642
3	3508	3.02	20.4 to 40.4	2210	46.1	1560	606
3	3509	5.99	20.4 to 60.4	1880	29.6	1220	627
4	3349	0.98	static	4250	205	3300	729
4	3510	2.83	0.3 to 60.3	2020	30.5	1350	629
4	3358	2.78	0.3 to 60.3	2030	44.4	1420	569

TABLE 3-18. CONCENTRATIONS OF TVOCs AND GROUPEd VOCs FOR TEST 6
(SAMPLE A)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	TVOC	<C ₈	C ₈ ~C ₁₂	C ₁₂
1	3549	1.00	static	9220	2210	5600	1420
1	3552	0.80	0 to 5	5690	1140	3450	1090
1	3553	2.27	5.8 to 20.8	3500	503	2060	932
1	3555	2.98	20.8 to 40.8	2390	191	1370	829
1	3556	6.04	20.6 to 60.7	2280	176	1160	935
2	3558	0.99	static	4740	531	3060	1140
2	3559	0.99	static	4920	423	3150	1340
2	3522	3.23	0.4 to 60.4	2320	180	1110	1030
2	3523	2.89	0.4 to 60.4	2070	161	1030	879
3	3587	1.00	static	3460	515	1750	1180
3	3586	1.00	static	3350	457	1730	1150
3	3584	0.76	0.2 to 5.2	2390	358	1110	920
3	3593	2.25	5.2 to 20.2	1930	134	871	918
3	3594	2.99	20.2 to 40.2	1910	102	815	988
3	3595	5.97	20.2 to 60.2	1820	73.0	749	994
4	3569	0.99	static	4150	550	2180	1410
4	3600	3.27	2.2 to 62.2	2030	143	750	1130
4	3601	3.06	2.2 to 62.2	1970	155	748	1060

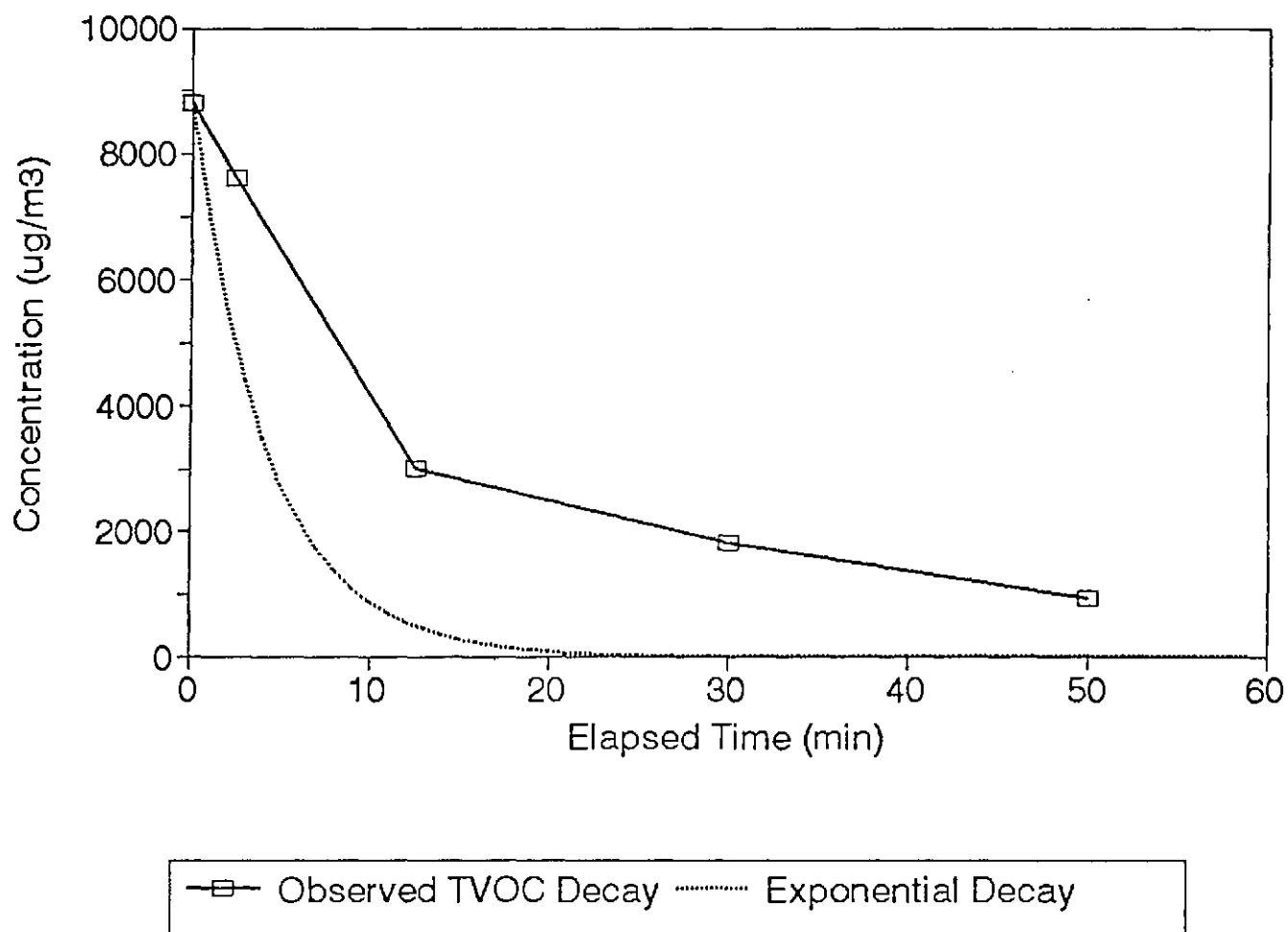


Figure 3-8. Observed TVOC concentrations vs. exponential decay (Exposure 1, Test 1).

3.5.2 Individual Compounds

The quantitative analysis of individual compounds for the source emissions was limited to the specific compounds selected by HERL as possible irritants or toxicants.

Four individual compounds were quantified for all the samples. They are as follows:

Acetic acid (AA)

Benzene (Benz)

Naphthalene (Naph)

Butylatedhydroxytoluene (BHT)

The results are presented in Tables 3-19 through 3-24. Note that the abbreviation BQL in the tables means that compound was identified in the sample but its quantity is below quantification limit, whereas ND means that compound was not detected in the sample.

TABLE 3-19. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 1
(SAMPLE A)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	2977	0.10	static	90.2	ND	BQL	242
1	2979	0.75	0 to 5	58.3	ND	25.7	362
1	2980	2.20	5 to 20	27.7	ND	21.1	316
1	2982	2.98	20 to 40	16.6	ND	17.5	322
1	2985	5.80	20 to 60	7.33	ND	15.5	295
2	2999	0.10	static	ND	ND	BQL	332
2	3001	0.10	static	ND	ND	ND	239
2	2995	9.05	0 to 60	14.8	ND	20.0	413
2	2997	8.76	0 to 60	12.9	ND	17.3	360
3	3055	0.10	static	ND	ND	ND	344
3	3058	0.10	static	ND	ND	ND	290
3	3059	0.80	0.2 to 5.3	BQL	ND	17.8	373
3	3047	2.20	5.3 to 20.3	8.30	ND	16.8	369
3	3048	3.00	20.3 to 40.3	10.0	ND	15.4	363
3	3049	6.00	20.3 to 60.3	8.32	ND	14.5	347
4	3033	0.10	static	ND	ND	ND	431
4	3034	9.20	0 to 60.3	11.1	ND	15.2	401
4	3065	8.90	0 to 60.3	4.87	ND	15.4	411

ND = Not detected

BQL = Below quantification limit

TABLE 3-20. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 2
(SAMPLE B)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	3030	0.10	static	ND	ND	ND	BQL
1	3006	3.00	20.2 to 45.2	ND	ND	ND	13.0
1	3007	5.90	20.2 to 60.2	ND	ND	ND	14.9
2	3101	0.10	static	ND	ND	ND	108
2	3103	0.10	static	ND	ND	ND	BQL
2	3105	9.00	1 to 61	BQL	ND	ND	26.5
2	3106	8.80	1 to 61	1.0	ND	ND	31.5
3	3147	0.10	static	ND	ND	ND	BQL
3	3129	0.76	2.8 to 7.8	ND	ND	ND	60.5
3	3118	2.20	7.8 to 22.8	ND	ND	ND	27.5
3	3117	2.96	22.8 to 42.8	ND	ND	ND	11.9
3	3115	6.00	22.8 to 62.8	ND	ND	ND	14.7
4	3109	0.10	static	ND	ND	ND	ND
4	3112	3.30	0.6 to 67.4	ND	ND	ND	30.6
4	3113	3.40	0.6 to 67.4	ND	ND	ND	20.8

ND = Not detected

BQL = Below quantification limit

TABLE 3-21. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 3
(SAMPLE B)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	3233	0.10	static	ND	ND	ND	ND
1	3229	0.76	1 to 6	ND	ND	ND	121
1	3231	2.27	6 to 21	ND	ND	ND	95.7
1	3209	2.99	21 to 41	ND	ND	ND	ND
1	3210	6.02	21 to 61	ND	ND	ND	22.6
2	3237	0.10	static	ND	ND	ND	BQL
2	3221	0.10	static	ND	ND	ND	BQL
2	3222	9.05	0.3 to 60.3	ND	ND	ND	19.3
2	3225	8.76	0.3 to 60.3	ND	ND	ND	17.0
3	3256	0.10	static	ND	ND	ND	BQL
3	3257	0.10	static	ND	ND	ND	BQL
3	3261	0.74	0.2 to 5.3	ND	ND	ND	21.5
3	3249	2.25	5.3 to 20.2	ND	ND	ND	11.4
3	3250	2.99	20.2 to 40.2	ND	ND	ND	4.02
3	3252	5.95	20.2 to 60.2	ND	ND	ND	9.34
4	3253	0.10	static	ND	ND	ND	BQL
4	3240	8.90	1.3 to 61.3	ND	ND	ND	14.2
4	3228	8.90	1.3 to 61.3	ND	ND	ND	13.4

ND = Not detected

BQL = Below quantification limit

TABLE 3-22. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 4
(SAMPLE C)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	3287	1.00	static	BQL	245	BQL	14.9
1	3289	0.75	1.5 to 6.5	29.8	161	BQL	14.4
1	3291	2.26	6.5 to 21.5	46.9	51.0	BQL	19.7
1	3305	2.97	21.5 to 41.5	20.1	14.1	BQL	21.9
1	3306	6.02	21.5 to 61.5	7.92	8.77	BQL	21.9
2	3281	1.00	static	ND	ND	BQL	14.9
2	3282	0.99	static	ND	BQL	BQL	BQL
2	3285	8.60	0.7 to 60.7	ND	BQL	BQL	5.70
3	3318	1.00	static	16.7	BQL	BQL	10.4
3	3319	0.99	static	16.2	BQL	BQL	11.3
3	3327	5.96	20.3 to 60.3	3.60	BQL	BQL	2.64
4	3330	1.00	static	13.9	BQL	BQL	14.6
4	3315	3.09	0.4 to 61	3.93	ND	BQL	16.2
4	3316	3.03	0.4 to 61	9.18	ND	BQL	20.1

ND = Not detected

BQL = Below quantification limit

TABLE 3-23. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 5
(SAMPLE C)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	3369	1.00	static	ND	212	13.1	19.8
1	3372	0.74	0.3 to 5.3	ND	170	BQL	14.9
1	3373	2.24	5.3 to 20.3	ND	52.2	5.00	12.8
1	3383	2.97	20.3 to 40.3	ND	25.9	4.50	17.0
1	3384	6.02	20.3 to 60.3	ND	BQL	4.20	17.9
2	3387	0.99	static	ND	BQL	7.80	11.0
2	3388	0.98	static	10.5	BQL	9.80	20.5
2	3354	3.02	0.3 to 60.2	ND	ND	5.00	21.2
2	3355	2.89	0.3 to 60.2	5.10	ND	1.20	ND
3	3501	1.02	static	ND	ND	7.30	10.8
3	3502	1.01	static	ND	ND	BQL	11.9
3	3505	0.75	0.4 to 5.4	8.90	ND	BQL	BQL
3	3507	2.29	5.4 to 20.4	ND	ND	4.70	16.0
3	3508	3.02	20.4 to 40.4	4.10	ND	4.00	14.2
3	3509	5.99	20.4 to 60.4	ND	ND	2.10	15.1
4	3349	0.98	static	BQL	ND	BQL	10.5
4	3510	2.83	0.3 to 60.3	ND	ND	3.70	15.8
4	3358	2.78	0.3 to 60.3	ND	ND	5.50	12.1

ND = Not detected

BQL = Below quantification limit

TABLE 3-24. CONCENTRATIONS OF SELECTED INDIVIDUAL COMPOUNDS FOR TEST 6
(SAMPLE A)
(Concentration unit: $\mu\text{g}/\text{m}^3$)

Exposure ID	Sample ID	Sampling Vol. (L)	Sampling Period (min)	AA	Benz	Naph	BHT
1	3549	1.00	static	58.4	ND	43.6	597
1	3552	0.80	0 to 5	108	ND	30.5	470
1	3553	2.27	5.8 to 20.8	99.9	ND	23.9	404
1	3555	2.98	20.8 to 40.8	44.8	ND	27.0	356
1	3556	6.04	20.6 to 60.7	24.8	ND	24.0	337
2	3558	0.99	static	49.4	ND	35.1	515
2	3559	0.99	static	57.2	ND	36.2	591
2	3522	3.23	0.4 to 60.4	30.8	ND	24.6	428
2	3523	2.89	0.4 to 60.4	24.1	ND	20.0	386
3	3587	1.00	static	28.0	ND	27.5	457
3	3586	1.00	static	29.2	ND	26.7	460
3	3584	0.76	0.2 to 5.2	18.7	ND	21.2	395
3	3593	2.25	5.2 to 20.2	17.0	ND	18.7	383
3	3594	2.99	20.2 to 40.2	10.2	ND	20.7	381
3	3595	5.97	20.2 to 60.2	11.8	ND	20.4	379
4	3569	0.99	static	24.2	ND	30.5	586
4	3600	3.27	2.2 to 62.2	20.9	ND	18.2	434
4	3601	3.06	2.2 to 62.2	20.4	ND	18.0	446

ND = Not detected

3.6 PARTICLE COUNTING

The average particle concentrations during exposure periods are summarized in Table 3-25. An example of particle concentration profile is shown in Figure 3-9. Without carpet sample in the source chamber, the particle concentration was about $30 \text{ P}/\text{cm}^3$ (Test 3). The particle concentration with carpet samples were not much higher than the empty chamber except Test 4, which had the highest concentration of $740 \text{ P}/\text{cm}^3$. This high reading, however, may have been caused by the

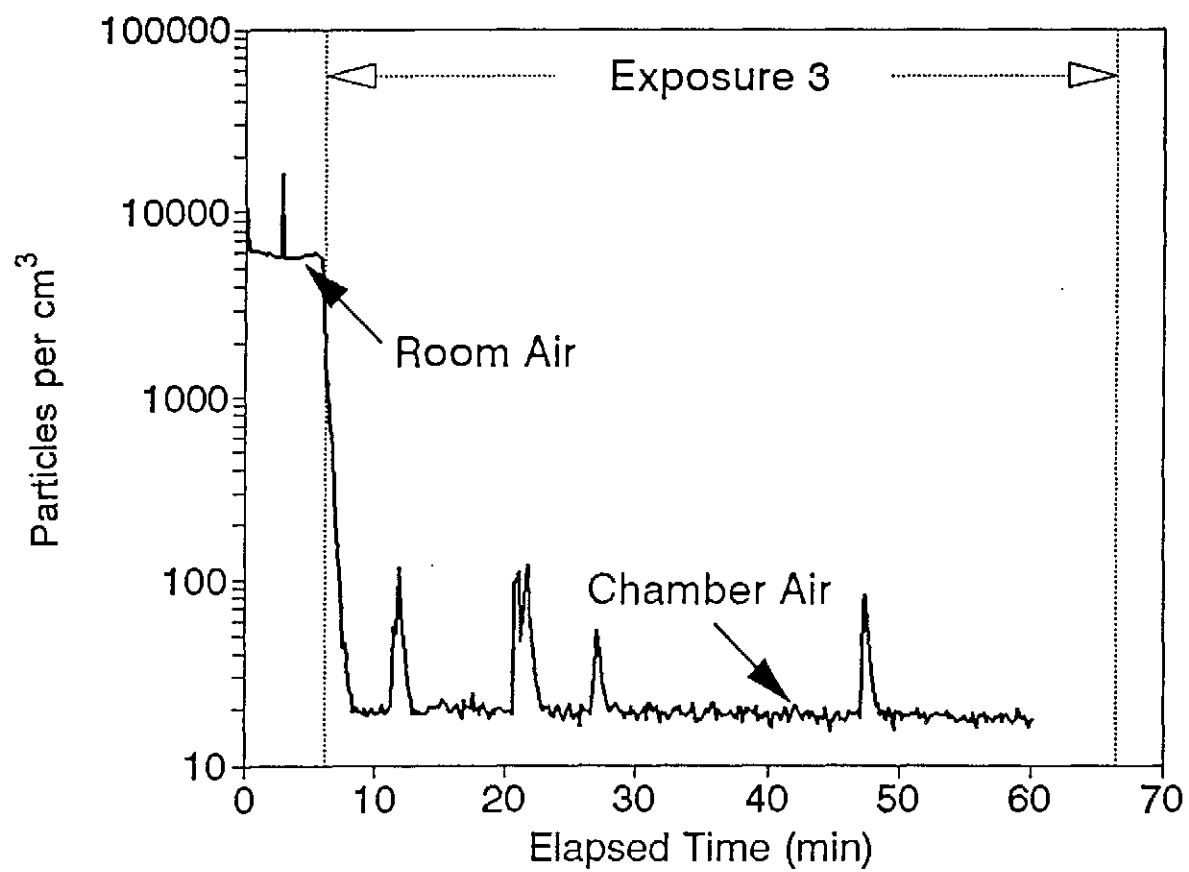


Figure 3-9. Particle counting data for Sample A in Test 1.

intrusion of laboratory air. During that test, the most serious air leak occurred—the humidified air flow entering the source chamber reduced by 68 percent by the end of the test (see air flow data in Table 3-5).

TABLE 3-25. PARTICLE CONCENTRATIONS IN THE EXPOSURE CHAMBER (IN P/cm³)

Test ID	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Test 1	50	N/A ¹	20	N/A ¹
Test 2	N/A ²	N/A ¹	N/A ³	N/A ¹
Test 3	30	30	40	20
Test 4	60	630	740	70
Test 5	30	60	50	40
Test 6	N/A ³	60	30 140	

¹ Not measured.

² Data lost due to computer problem.

³ Instrument flooding problem.

N/A = data not available.

For comparison purposes, the particle concentrations in the laboratory air were also measured during each test, and they varied from 1,000 to 10,000 P/cm³.

3.7 MISCELLANEOUS OBSERVATIONS

Sample A, the dark pink low pile carpet, was noted to have a variety of dirt spots on the samples used for Test 1. The Sample A subset of carpet used for Test 6 was observed to have a large yellow water stain on one section. During this test, condensation filled the exposure chamber during the first exposure. This phenomenon was not observed by HERL during animal testing.

Sample C, the indoor/outdoor dark blue carpet, was adhered to the inside of the Tedlar bag from the glue on the backing of the carpet. Slivers of plywood were attached to the glue. The Tedlar bags contained about 2.0 mL of sand in one bag. The carpet sections were also marked with an ID No. on the carpet. The source of these makings was not identified but became a part of the test. Condensation was again observed in the AEERL system and not in the HERL system during Test 4.

The bottom of the chamber cracked from the heat during the first exposure of Test 5. The chamber was repaired with duct tape and the sampling continued.

SECTION 4
DATA QUALITY REVIEW

4.1 DATA QUALITY OBJECTIVES

Data quality objectives as outlined in the test plan are summarized in Table 4-1. In addition, objectives for temperature control were described in Section 2.6.

TABLE 4-1. DATA QUALITY OBJECTIVES

Measurement	Accuracy	Precision	Completeness
Temperature	$\pm 1^{\circ}\text{C}$	N/A	85%
Air flow	10%	15%	85%
Relative humidity	10%	15%	85%
Carpet area	10%	15%	85%
Sampling period	5%	N/A	90%
GC analysis	15%	15%	90%
Aerosol zero-checking	$<200 \text{ P/cm}^3$	N/A	90%

4.2 TEMPERATURE

4.2.1 Sensor Calibration

The thermocouples were calibrated before Test 1 and after Test 6. The temperature medias used were as follows: ice/water mixture (0°C), boiling water (100°C), and warm water (temperature was determined by an NIST standard thermometer). The calibration data are given in Tables C-1 through C-12 in Appendix C. The absolute errors varies from 0 to 0.9°C —all are within the $\pm 1^{\circ}\text{C}$

accuracy objective. The standard deviations for repeated measurements are also satisfactory—ranging from 0.0 to 0.11 °C.

4.2.2 Temperature Control

Figures 4-1 through 4-3 show the temperature control results for the three key locations in the experimental system—the outside surface of the source chamber bottom, the air in the source chamber, and the air in the exposure chamber. All data are the average temperature during the exposure periods. The air temperature in the exposure chamber is the average of two sampling locations.

For chamber bottom temperature measurements, three out of 24 data points exceeded the 70 ± 5 °C range, 71 percent of average temperature for chamber air exceeded the 37 ± 3 °C range. Overall, the air temperature was 4 °C higher than the 37 °C target.

For the temperature measurements in the exposure chamber, nine out of 24 data points exceeded the 24 ± 2 °C range.

4.3 RELATIVE HUMIDITY

4.3.1 Calibration of Humidity Probes

Humidity probes were calibrated before and after the testing period. Two humidity standards were used: saturated NaCl solution and saturated LiCl solution. The calibration data are given in Appendix C (Tables B-13 and B-14). Results show that the responses of the two sensors shifted only 0.5 percent relative humidity (RH).

4.3.2 Humidity Measurement

The desired RH for humidified air was 50 percent. The actual measured RH varied between 41 and 59 percent (Table 4-2). The deviations from 50 percent RH are all within 10 percent.

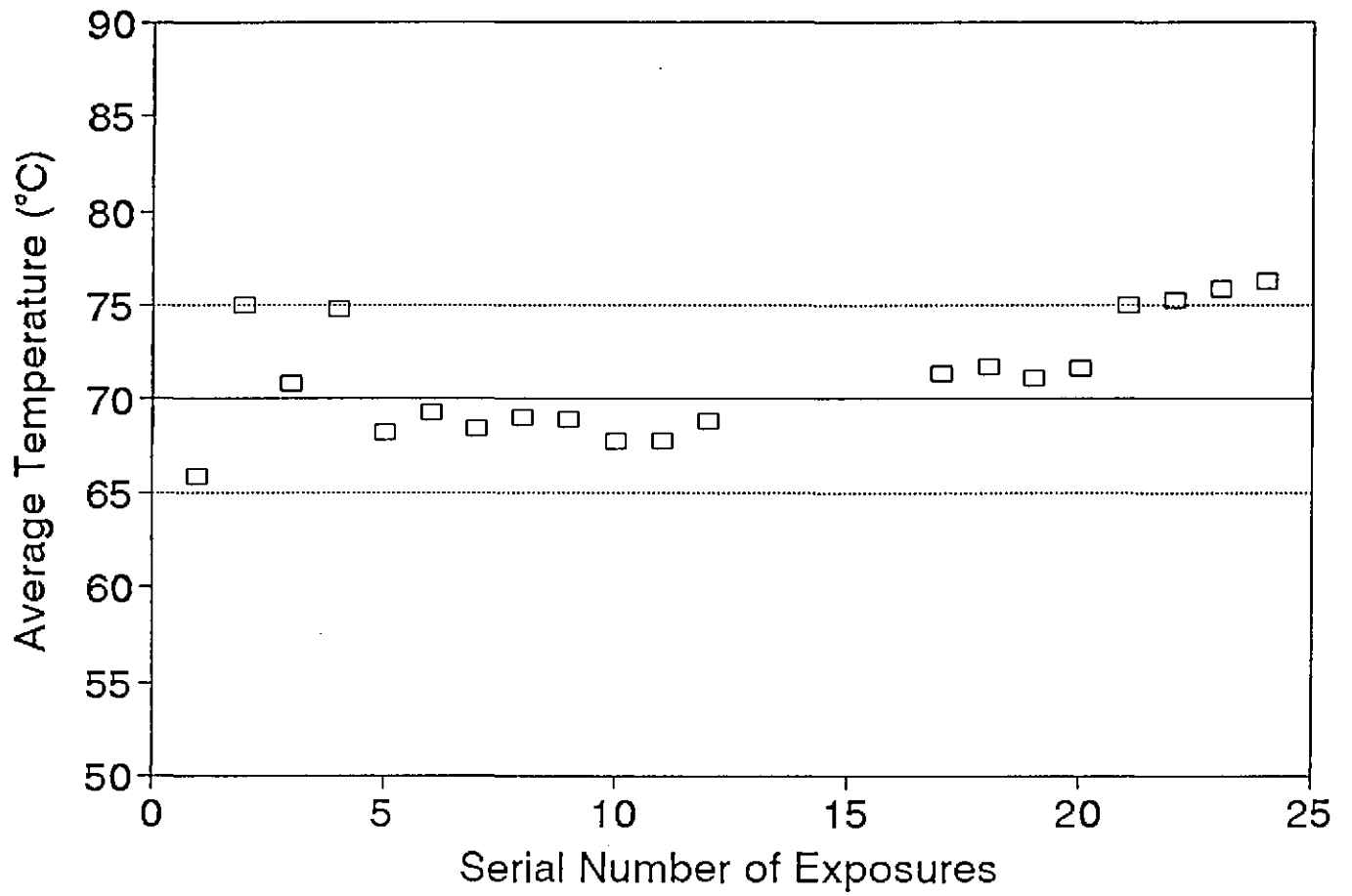


Figure 4-1. Results of temperature control; Chamber bottom panel.

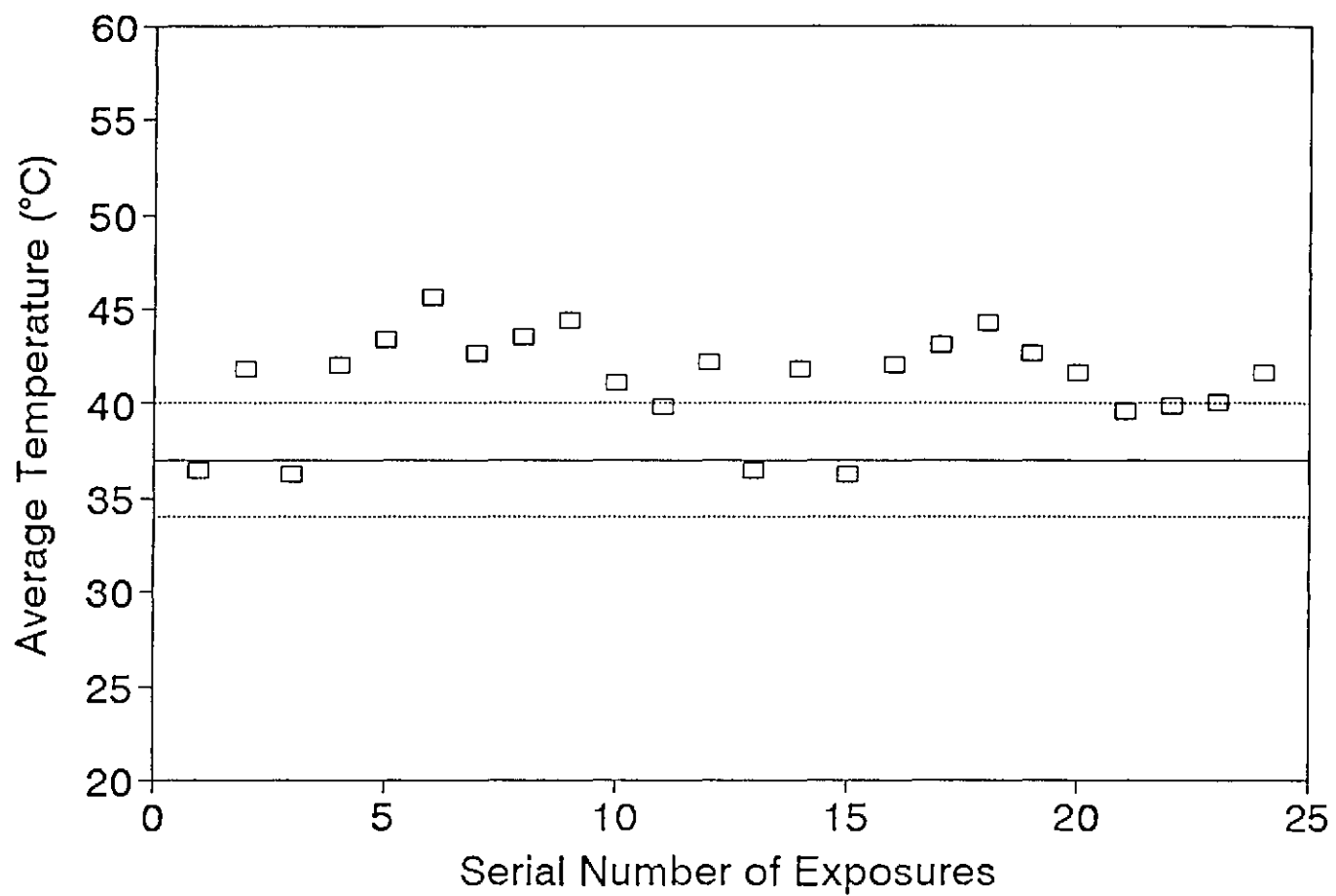


Figure 4-2. Results of temperature control; Source chamber air.

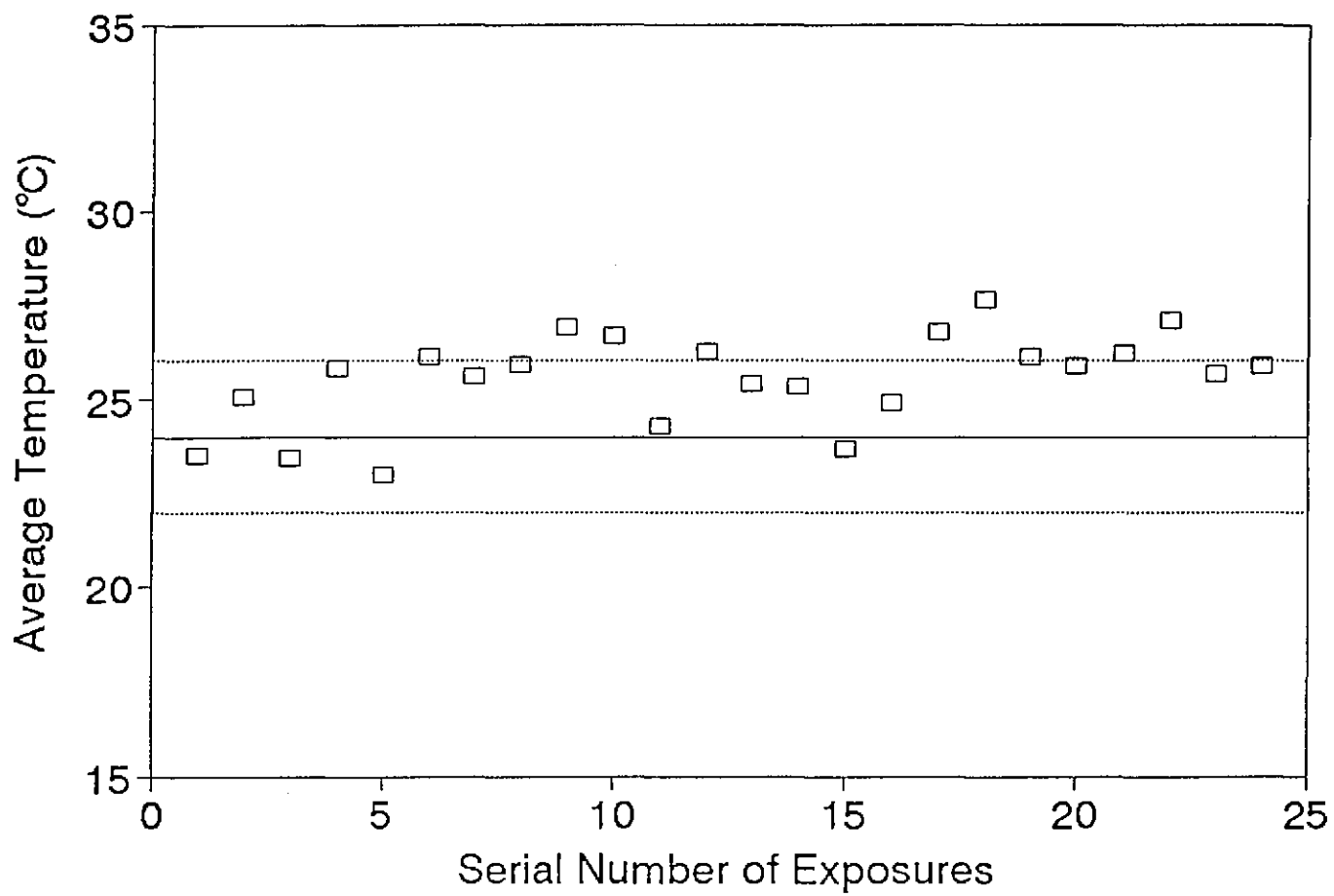


Figure 4-3. Results of temperature control; Air in the exposure chamber.

TABLE 4-2. MEASURED RELATIVE HUMIDITY FOR INLET AIR

Date	Measured RH (%)	Deviation from 50% RH
03/09/93	43.1	+6.9%
03/10/93	51.5	+1.5%
03/10/93	45.3	-4.7%
03/11/93	49.7	-0.3%
03/11/93	48.8	-1.2%
03/12/93	52.4	+2.4%
03/12/93	47.6	-2.4%
03/23/93	51.3	+1.3%
03/23/93	59.3	+9.3%
03/24/93	50.2	+0.2%
03/25/93	50.2	+0.2%
03/25/93	50.2	+0.2%
03/26/93	47.9	-2.1%
03/26/93	52.4	+2.4%
03/30/93	52.4	+2.4%
03/30/93	48.8	-1.2%
03/31/93	52.0	+2.0%
03/31/93	54.5	+4.5%
04/01/93	50.6	+0.6%
04/01/93	49.3	-0.7%
04/02/93	54.5	+4.5%
04/02/93	49.5	-0.5%
Average ¹		2.3%

¹ Average was made on absolute values.

4.4 AIR FLOW RATE MEASUREMENTS

4.4.1 Sampling Flow Rate

Three sampling flow rates requiring mass flow controllers were used in the experiment: 50, 150, and 400 mL/min. Mass flow controllers were fully calibrated and checked daily. Figures 4-4 and 4-5 show the calibration results for the seven mass flow controllers that had been used to sample at only one rate setting, and Figure 4-6 shows the results for the three mass flow controllers that had been used at varied sampling flow rates. All the flow rates were within 10 percent of the target rates. There were only three flow rates exceeding 5 percent of the desired rate.

4.4.2 Chamber Air Flow Rate

The inlet/outlet flows were all well balanced before the start of each test, and the differences of the two flow rates were all within 10 percent (see Table 3-7).

The outlet flow rates measured during the tests were also satisfactory (see Table 3-8). The difference between the desired rate (7 L/min) and those observed ranged from 0.1 to 8 percent.

Because of the continuous heating, the chamber became progressively leakier during the test period. In two out of six cases, the inlet flow decreased by more than 15 percent at the end of the test. The worst case occurred in Test 4, in which the flow reduction was as high as 68 percent. Note that this may not be a data quality problem but rather a problem with the experimental method.

To evaluate the effect of laboratory air intrusion on pollutant concentrations in the experimental system, laboratory air samples were taken during the testing period. These samples have been analyzed, and the analytical results were not considered an important factor to the final results of this study.

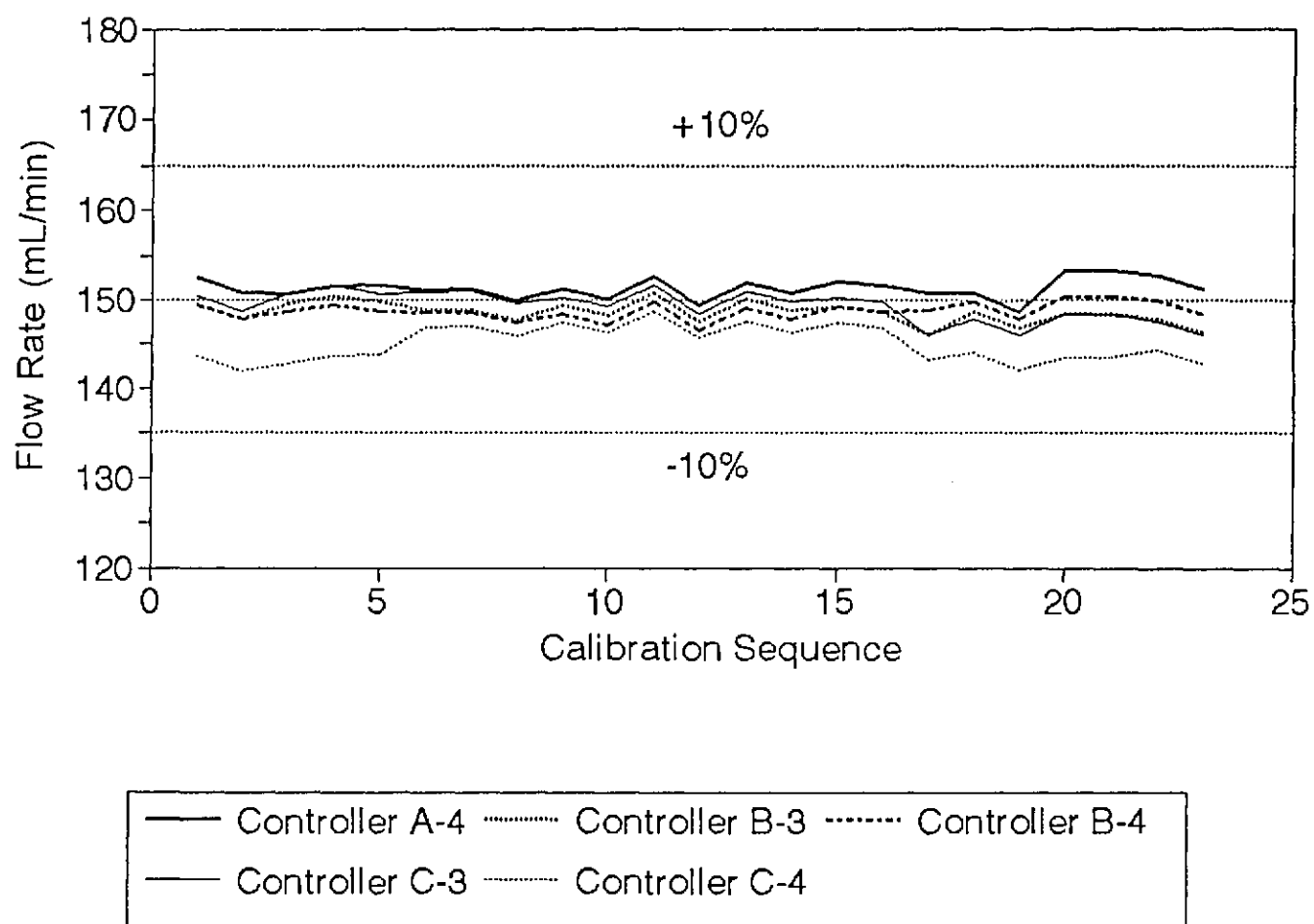


Figure 4-4. Calibration of mass flow controllers (1) Flow rate = 150 mL/min.

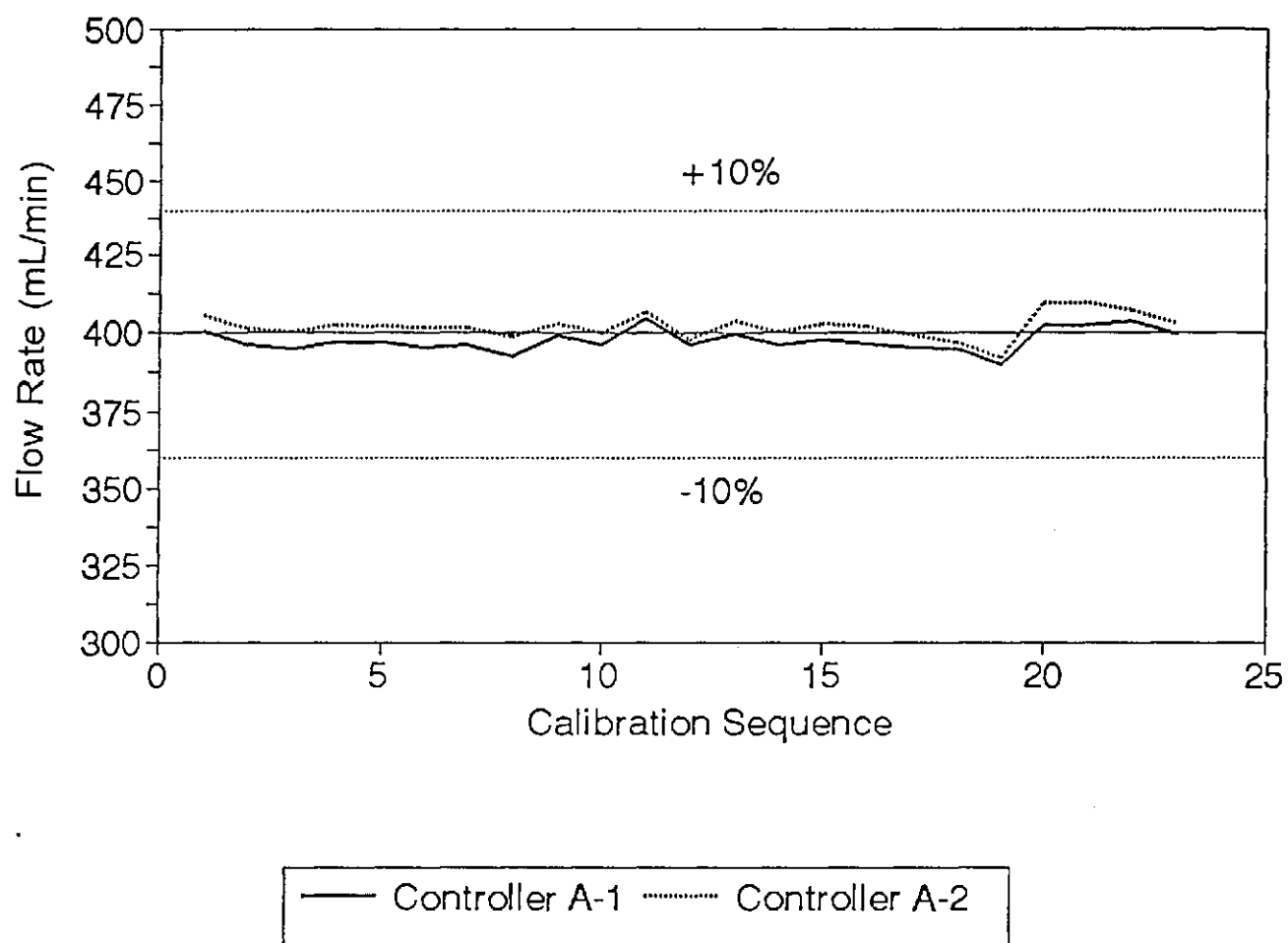


Figure 4-5. Calibration of mass flow controllers (2) Flow rate = 400 mL/min.

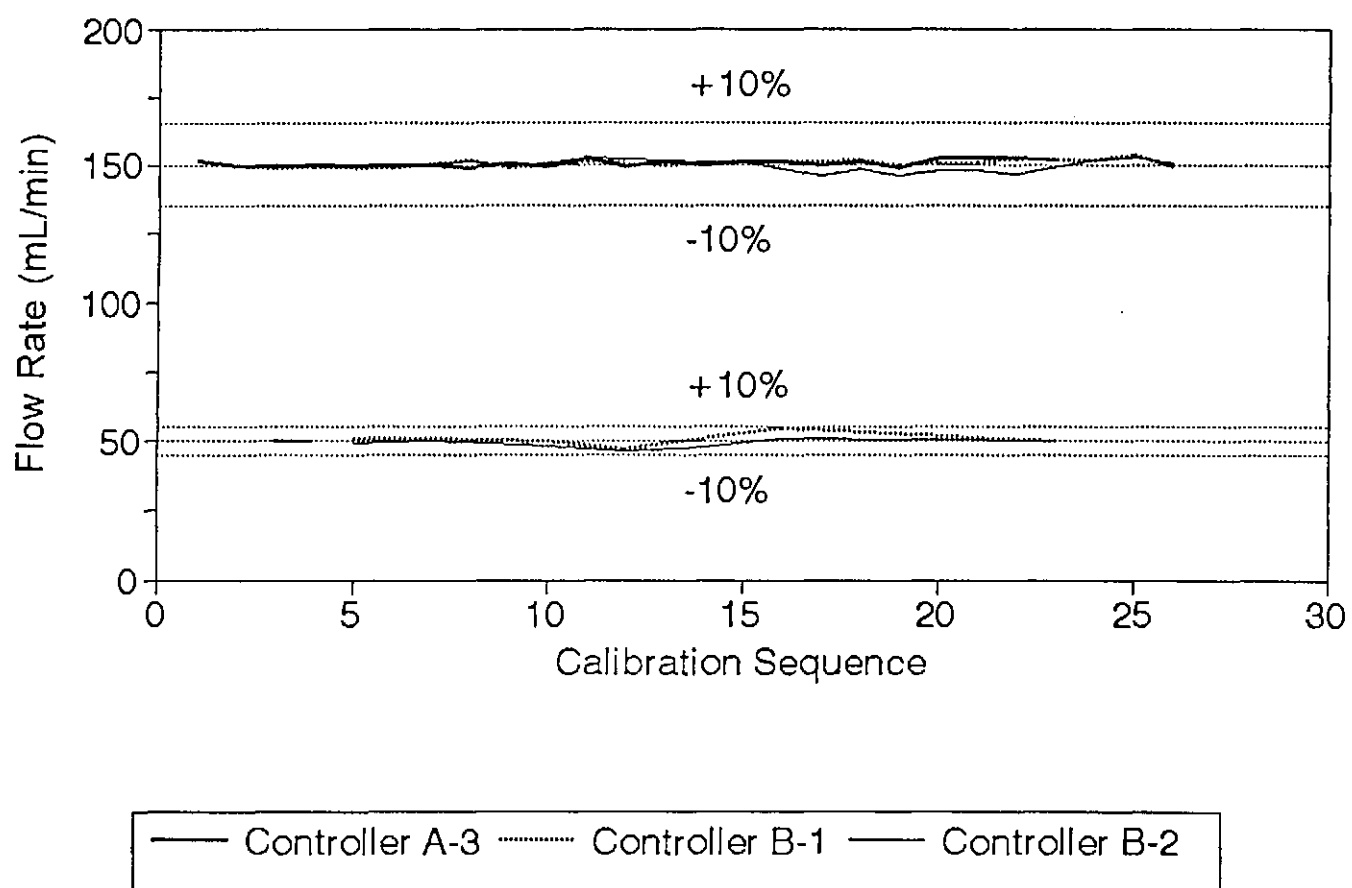


Figure 4-6. Calibration of mass flow controllers (3) Used at two flow rates.

4.5 GC ANALYSIS

4.5.1 Detection Limit and Quantification Limit

The Method Detection Limit (MDL) is the smallest amount qualitatively found in a sample analysis. The Method Quantification Limit (MQL) is defined as the smallest amount that can be accurately quantified in a sample analysis. The detection and quantification limits for each of the instruments used for analysis for this project were determined from the variability in the field blanks collected during the sampling. A field blank is described as a sorbent trap that has been removed from the storage vial, placed on the sampling system, leak-checked, and then returned to the storage vial. The field blank then follows the path of the samples to analysis. Because of a noted system contamination from methylene chloride, all chromatograms would have the area counts for methylene chloride subtracted before any further analysis of the data.

A total of six field blanks were analyzed by EC I and eleven analyzed by EC II. Detection limits for TVOC and specified compounds were determined by the following:

$$\text{MDL} = \text{Mean}_{\text{FB}} + 3 (\text{STD})$$

where Mean = average ng of the background from the field blank minus MeCl_2 .

Quantification limits for TVOC and specified compounds were determined by the following:

$$\text{MQL} = \text{Mean}_{\text{FB}} + 10 (\text{STD})$$

Tables 4-3 and 4-4 give a complete breakdown of detection limits and quantification limits for both analytical systems.

TABLE 4-3. EC I DETECTION AND QUANTIFICATION LIMITS

Compound	Mean (ng)	RSD	MDL (ng)	MQL (ng)
TVOC	40	16	87	197
Toluene	1.4	0.5	2	6
Benzene	13	4	24	48
4PCH	29	3	10	33
<C8	31	13	69	157
C8-C12	5	2	12	27
>C12	2	1	5	13

TABLE 4-4. EC II DETECTION AND QUANTIFICATION LIMITS

Compound	Mean (ng)	RSD	MDL (ng)	MQL (ng)
TVOC	50	21	114	262
Toluene	2	0.5	3	6
Benzene	6	6	25	67
4PCH	2	0.2	0.6	6
<C8	27	11	61	142
C8-C12	18	13	59	153
>C12	5	3	14	37

Because of the lack of any system background in the retention time region of 4PCH, the detection limit for 4PCH was determined by the variability in the lowest concentration standard used for GC response calibration.

$$\text{MDL} = 3 (\text{STD})_{\text{lowest std}}$$

$$\text{MQL} = 10 (\text{STD})_{\text{lowest std}}$$

4.5.2 Daily QC Check

Liquid toluene standards (106 and 280 ng/ μ L) were used for daily QC check. Table 4-5 summarizes the results and Figures 4-7 through 4-8 show the QC chart.

TABLE 4-5. DAILY QC CHECK STATISTICS

	Envirochem I	Envirochem II
Total no. of injections	57	14
No. of injections with error > 15%	6	1

4.5.3 Accuracy

The accuracy of the instruments were estimated by making gas standard injections. The standard used was 165 ppm toluene. Results are summarized in Table 4-6.

TABLE 4-6. DETERMINATION OF ACCURACY FOR TOLUENE

	Envirochem I	Envirochem II
Total no. of injections	32	41
No. of injections with error > 15%	5	3

4.5.4 Precision

The precision of the GC results was measured by comparing duplicate samples. Table 4-7 summarizes the results of the duplicate samples that were collected over the course of this study. Several duplicates were lost because of instrument malfunctions, changes in analysis protocol, and concentrations that were below the quantification limits for the instrument. Tables 4-8 through 4-11 give the precision estimates for TVOCs and individual compounds.

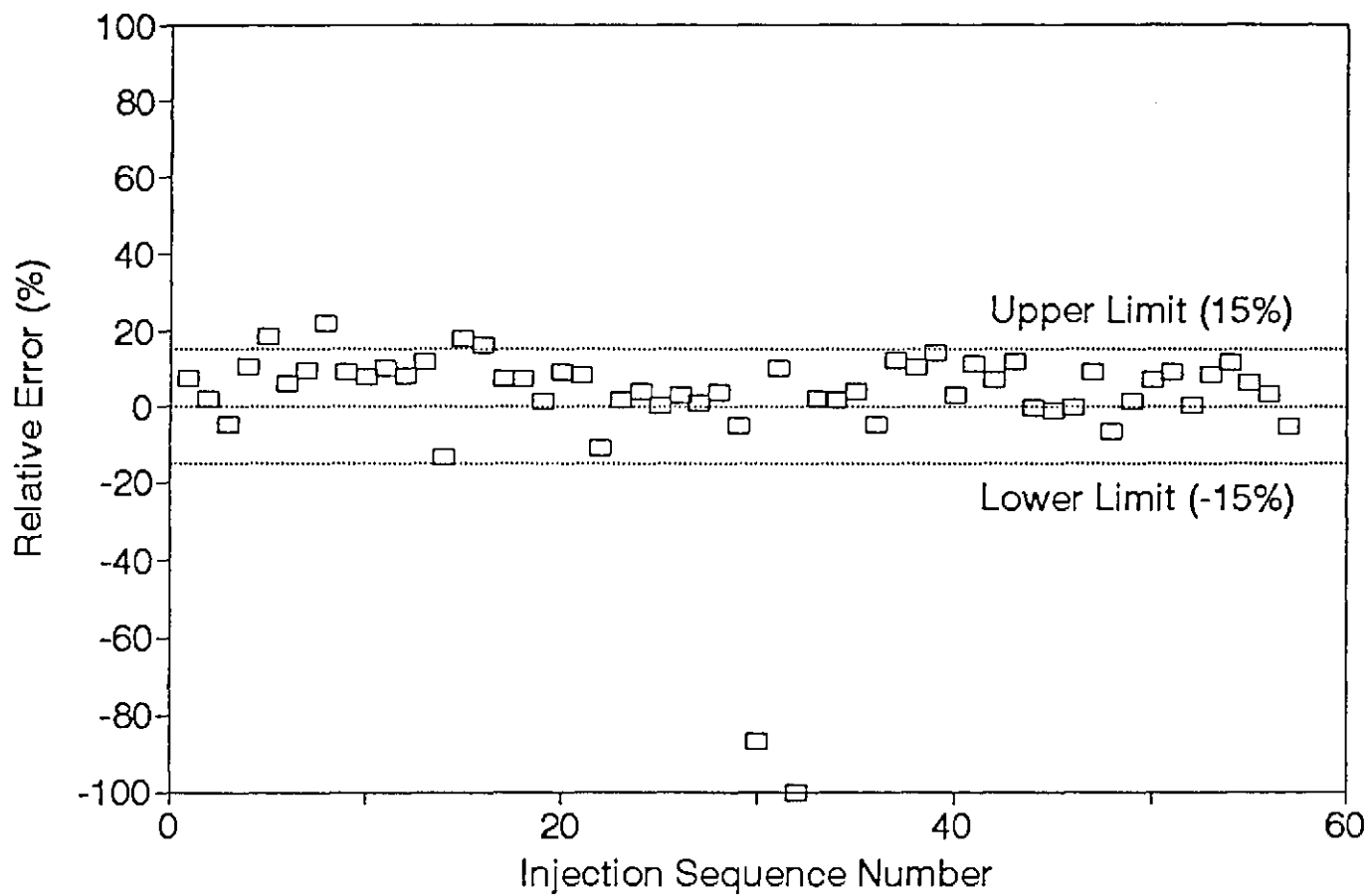


Figure 4-7. GC daily QC check results (1) Envirochem I/Liquid Toluene standard.

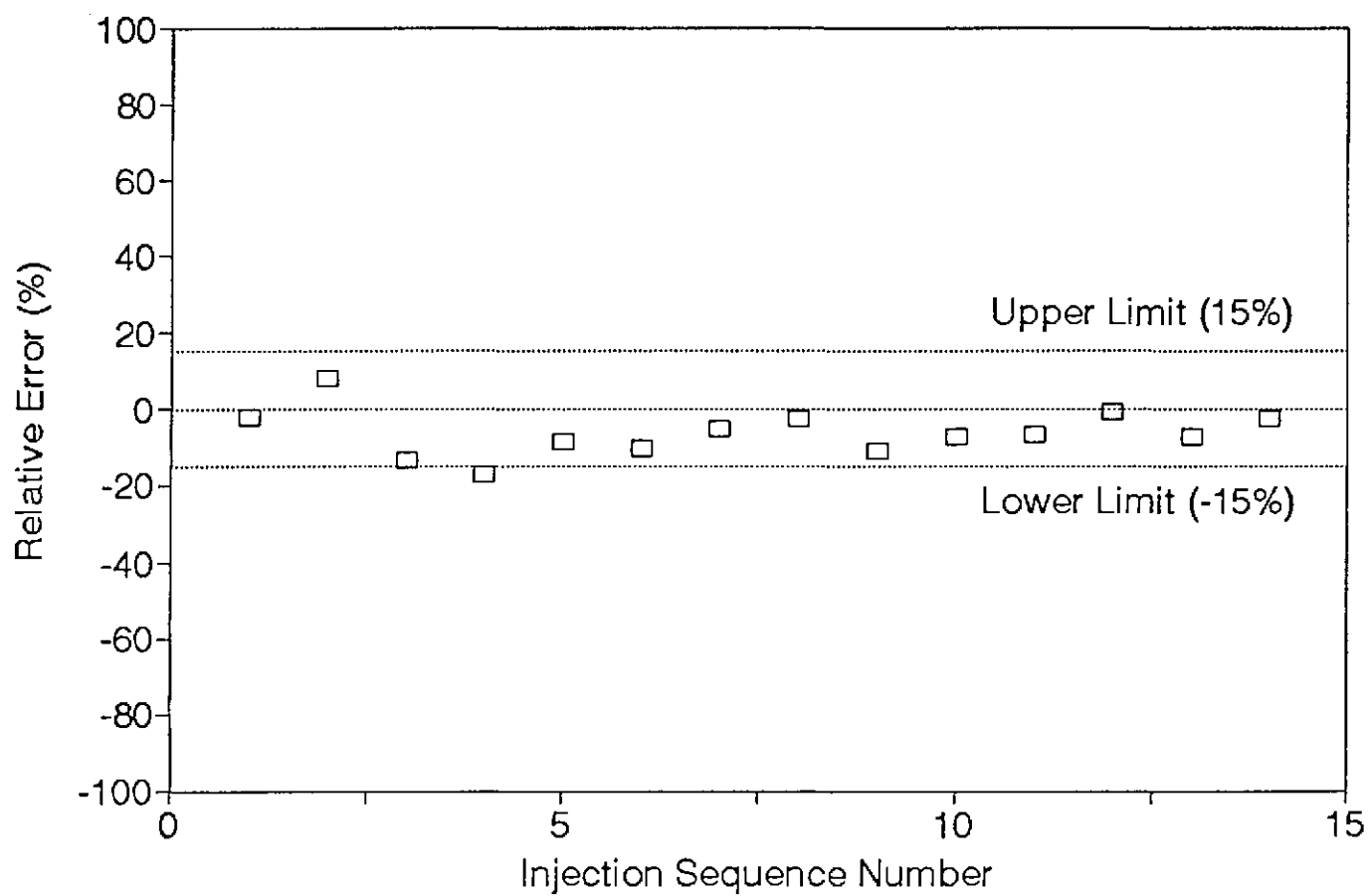


Figure 4-8. GC daily QC check results (2) Envirochem II/Liquid Toluene standard.

TABLE 4-7. DUPLICATE SAMPLES STATISTICS

	Pairs
Total no. of duplicates collected	23
Total no. lost	6
No. with error > 15% for TVOC	2

TABLE 4-8. COMPARISON OF DUPLICATE SAMPLES—TVOC

Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Mean conc.	RSD (%)
2995	1890	2997	1584	1737	12
3055	2300	3058	2041	2170	8.4
3034	1649	3035	1554	1602	4
3105	164	3106	200	182	14
3112	162	3113	129	145	16
3222	112	3225	117	115	4
3240	76	3228	72	74	4
3281	7583	3282	6101	6842	15
3318	7222	3319	7301	7262	1
3315	2468	3316	2751	2610	8
3387	7068	3388	7728	7398	6
3354	3181	3355	1013	2097	73
3510	2016	3358	2033	2024	1
3558	4739	3559	4929	4831	3
3522	2323	3523	2073	2198	8
3587	3457	3586	3350	3404	2
3600	2025	3601	1967	1996	2

RSD = Relative standard deviation

TABLE 4-9. COMPARISON OF DUPLICATE SAMPLES—ACETIC ACID

Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Mean conc.	RSD ¹ (%)
2995	15	2997	13	14	10
3034	11	3065	5	8	55
3318	17	3319	16	16.5	2
3315	4	3316	9	7	57
3558	49	3559	57	53	10
3522	31	3523	24	28	17
3587	28	3586	29	28.6	3
3600	21	3601	20	20.7	1.7

¹ Three out of eight pairs have RSD > 15%.

TABLE 4-10. COMPARISON OF DUPLICATE SAMPLES—NAPHTHALENE

Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Mean conc.	RSD ¹ (%)
2995	20	2997	17	19	10
3034	15.2	3065	15.4	15.3	1
3387	8	3388	10	9	16
3354	5	3355	1.2	3	88
3510	3.7	3358	5.5	5	28
3558	35.1	3559	36.2	35.7	2.3
3522	24	3523	20	22	15
3600	18.2	3601	18.0	18.1	0.6

¹ Three out of eight pairs have RSD > 15%.

TABLE 4-11. COMPARISON OF DUPLICATE SAMPLES—BHT

Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Sample ID	Conc. $\mu\text{g}/\text{m}^3$	Mean conc.	RSD ¹ (%)
2995	413	2997	360	386	10
3055	344	3058	290	317	12
3034	401	3065	411	406	2
3105	27	3106	32	29	12
3112	31	3113	21	26	27
3222	19	3225	17	18	9
3240	14	3228	13	13.5	4
3318	10.4	3319	11.3	10.8	6
3315	16	3316	20	18	15
3387	11	3388	20	16	43
3510	16	3358	12	14	19
3558	515	3559	591	553	10
3522	428	3523	386	407	7
3587	457	3586	460	458	0.4
3600	434	3601	446	440	2

¹ Three out of 11 pairs have RSD > 15%.

4.5.5 Completeness of ST032 Sorbent Samples

The total number of planned observations was 138, and the number of valid observations was 101. This gives the completeness of 73 percent.

4.6 IDENTIFICATION OF INDIVIDUAL COMPOUNDS

The identification of individual compounds required both electronic and manual evaluation of the results as presented in Section 2 of this report. RTI was designated as the primary source for compound identification because of the availability of a high resolution GC/MS system. Acurex Environmental provided backup to RTI with the EC II GC system that split the effluent from the column to an MSD and an FID. This provided both qualitative and quantitative evaluation of the compounds. To ensure comparable responses for all three analytical systems, a standard containing

the even n-alkanes (C_8 - C_{20}), toluene, and 4PCH was utilized for the purpose of establishing a retention time correlation and response factor database between the three analytical systems used in this study. Sorbent traps were spiked with this standard and analyzed on all systems. RTI spiked a series of sorbent traps with their system to verify the comparability of spiking systems for the purpose of daily QC checks of their MS system.

4.7 QUANTIFICATION OF INDIVIDUAL COMPOUNDS

The quantification of individual compounds required correlation of marker retention times from the RTI MS output and Acurex Environmental MSD output to the FID output from EC I. Manual interpretation of the FID chromatogram from EC I and total ion chromatogram (TIC) from EC II MSD and RTI MS to match peak shapes and patterns was also performed. For the prominent emissions/peaks this process proved successful as reported in Table 4-11, results of duplicate analysis for BHT. The evaluation of compounds found in low concentrations or compounds that showed poor chromatography, such as acetic acid (Table 4-9), was more difficult and required extensive manual interpretation of the GC data.

4.8 EFFECTIVENESS OF CHAMBER CLEANING

One question asked in the test plan was, "Is there any memory of carpet emissions in a cleaned, reused aquarium?" Five different aquariums, AQ0-AQ4, were used in this study including those used by HERL. The most repeated use of an aquarium was three times each by AQ3 and AQ4. Figures 4-9 and 4-10 show the TVOC profiles of duplicate 3-L samples collected from heated empty chambers after cleaning. The MSD analysis identifies siloxanes, toluene, and BHT as the major emissions from a cleaned chamber. The differences in the TVOC emissions from each use can be attributed to differences in air supply or poor cleaning. As mentioned previously, the zero-grade air is certified to contain less than 0.1 ppm THC. This can be equated to $\sim 400 \mu\text{g}/\text{m}^3$ toluene. RTI identified 4PCH in the empty chamber experiments (Tests 2 and 3). This was not confirmed by Acurex Environmental's evaluation of the chamber emissions.

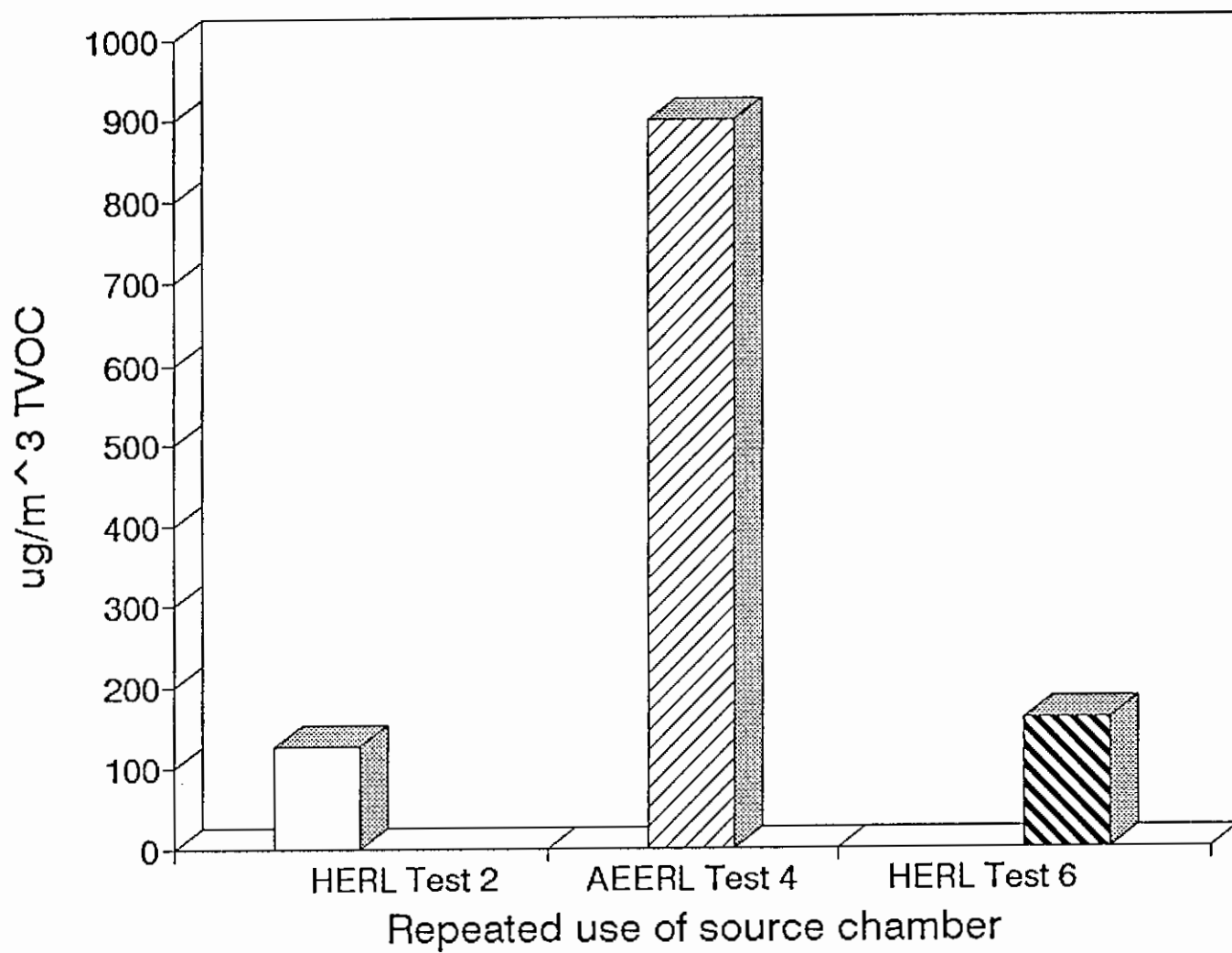


Figure 4-9. Chamber background: Aquarium 3.

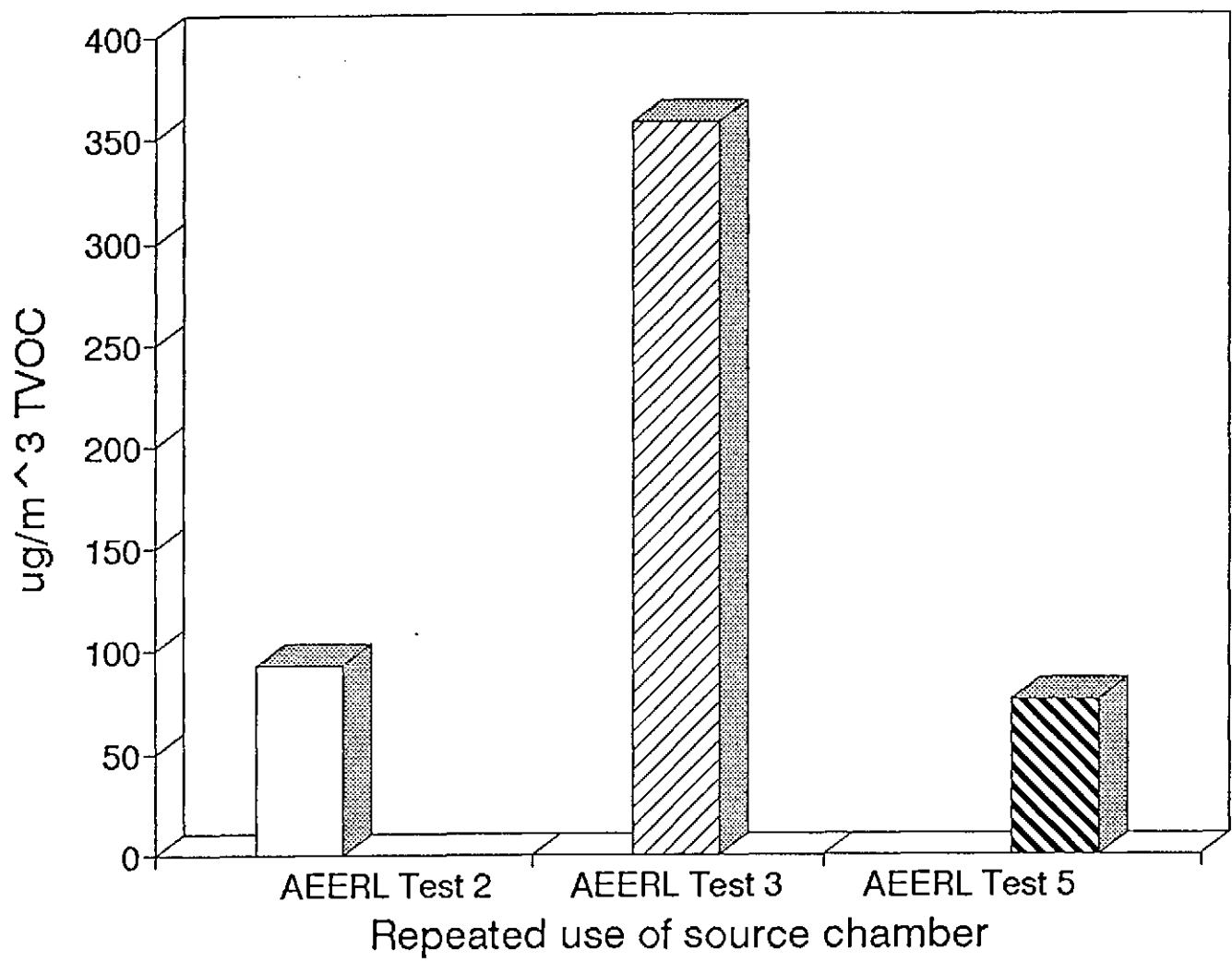


Figure 4-10. Chamber background: Aquarium 4.

4.9 PARTICLE COUNTING

The particle counter used requires factory calibration. The last calibration was made before the tests started (February 8, 1993), and the calibration remains valid for one year.

During the testing period, zero-checking was made each day to ensure that there were no leaks in the instrument or in the sampling line. All the zero-checks passed the 200 P/cm³ objective with typical values below 10 P/cm³ (Table 4-12).

TABLE 4-12. ZERO-CHECKING RESULTS FOR PARTICLE COUNTER¹
(Unit: P/cm³)

Test ID	Exposure 1	Exposure 2	Exposure 3	Exposure 4
1	OK	N/A	OK	N/A
2	N/A	N/A	OK	N/A
3	OK	<20	OK	<10
4	<1	<1	<1	<1
5	<1	<10	<10	N/A
6	<10	<10	<1	<10

¹ In early experiments, the actual zero-check readings were not recorded. OK means the check passed (reading is below 200 P/cm³).

During the testing period, the instrument flooded with newly added 2-propanol several times. (This instrument requires adding alcohol after use for several hours.) This phenomenon may give faulty high concentration readings for a period of time. Serious flooding occurred during Exposure 1 of Test 2 and Exposure 1 of Test 6. Consequently, these two sets of particle data were not used.

After the tests were completed, the problem was discussed with a representative of the manufacturer of the instrument. The manufacturer advised running the instrument for about 10 min while turning it upside down—an action that the operation manual does not recommend. The manufacturer stated that the newer model of this instrument no longer has the problem.

4.10 AUDITS

Three external audits were performed by the AEERL Quality Assurance (QA) program during the course of this project. The first was an audit to evaluate the test plan for the study. The second was a technical systems audit, and the third was audit to examine the analysis and data reduction procedures as compared to those documented in the laboratory QA Project Plan (QAPP). We have responded to all comments and correction measures were made.

The first audit of the test plan resulted in the reconstruction of the study test plan into separate plans for each laboratory. All findings and comments were responded to in an appropriate manner.

The second external audit for the technical systems found only a few problems with the most serious being "operating procedures of the laboratory are scattered throughout several documents and some are not available." At the start of this project, the laboratory had an approved QAPP. However, it did not encompass all the fabricated systems and new equipment that was necessary for this study. Because of the time restraints placed on this project, each issue was addressed as it surfaced. All of the concerns that were noted in the audit were amended.

The analysis and data reduction audit pointed out the need for a more standardized data management process. These issues have been addressed. The data management procedures that proved to be effective during Phase I of this project will be documented in a SOP format and included in the laboratory facilities manual. An internal evaluation of custody and document procedures was also performed by the Acurex Environmental QA staff.

4.11 CONCLUSIONS ON DATA QUALITY REVIEW

All the data quality goals have been achieved except the following.

- (1) Average air temperature in the source chamber: Overall, the actual temperature was 4 °C higher than the 37 °C target.
- (2) Air temperature in the exposure chamber: 9 out of 24 exposure periods had temperatures exceeding the 24 ± 2 °C range.

- (3) Due to sample loss, the completeness of ST032 sorbent samples was 73%, whereas the target was 85%.
- (4) For the analysis of individual compounds, about one-third duplicate samples showed relative standard deviations (RSD) greater than the target 15%. Several pairs of duplicate samples had very large RSD. We recommend that the analytical results for individual compounds be considered semi-quantitative.

SECTION 5

DISCUSSION OF RESULTS

5.1 COMPARISON OF INITIAL TVOC CONCENTRATIONS IN THE SOURCE CHAMBER

After a one-hour heating period without air flow, the peak TVOC concentration in the source chamber was reached. This peak concentration was determined by directly sampling from the source chamber before the dynamic mode started. A comparison of peak TVOC concentrations between the two carpet samples is shown in Figure 5-1. All values in the graph are averages of duplicate tests. We were unable to quantify the initial concentrations for the mock samples (Tests 2 and 3) because the sample volumes were too small.

5.2 COMPARISON OF AVERAGE TVOC CONCENTRATION IN THE SOURCE CHAMBER DURING THE EXPOSURE PERIOD

The calculation of average concentrations found in the source chamber involves the following two steps: (1) calculating the area under the time-concentration curve by means of integration and (2) calculating the average concentration. The curve integration can be approximated by:

$$A_c = \sum_{i=1}^N (C_i \Delta t_i)$$

where A_c is the area under the time-concentration curve, in ($\mu\text{g m}^{-3} \text{ min}$);

C_i is the concentration for sample i , in ($\mu\text{g m}^{-3}$);

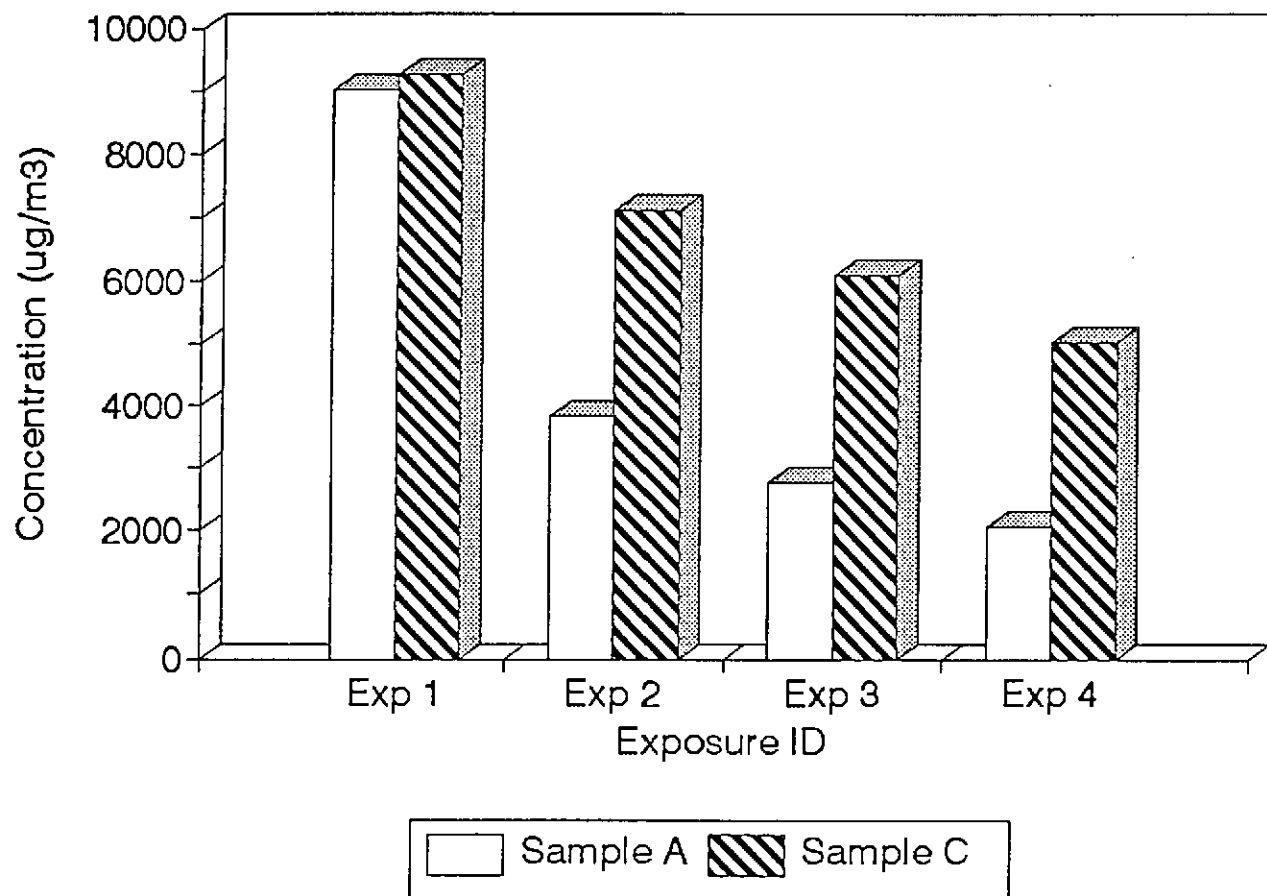


Figure 5-1. Initial TVOC concentrations in the source chamber.

Δt_i is the sampling period for sample i , in (min); and

N is the total sample number, excluding the overlapping samples.

The average concentration can then be determined from:

$$C_x = \frac{A_c}{t_x}$$

where C_x is the average concentration during an exposure, in ($\mu\text{g}/\text{m}^3$); and

t_x is the exposure period, in (min).

The calculated results are given in Table 5-1 and Figure 5-2.

TABLE 5-1. AVERAGE TVOC CONCENTRATIONS IN THE EXPOSURE CHAMBER (in $\mu\text{g}/\text{m}^3$)

Test No.	Exposure 1	Exposure 2	Exposure 3	Exposure 4
1	2740	1740	1280	1600
2	79	182	227	145
3	274	115	63	74
4	4440	1890	2200	2750
5	3480	2100	2500	2020
6	2830	2200	1880	2000

5.3 CALCULATION OF TOTAL AMOUNT OF TVOC ELUTED FROM THE SOURCE CHAMBER DURING AN EXPOSURE

The total amount of a given pollutant eluted from the source chamber can be calculated from:

$$W_E = C_x Q t_x$$

where W_E is the total mass eluted from the source chamber, in (μg); and

Q is the air exchange flow rate through the system, in (m^3/min).

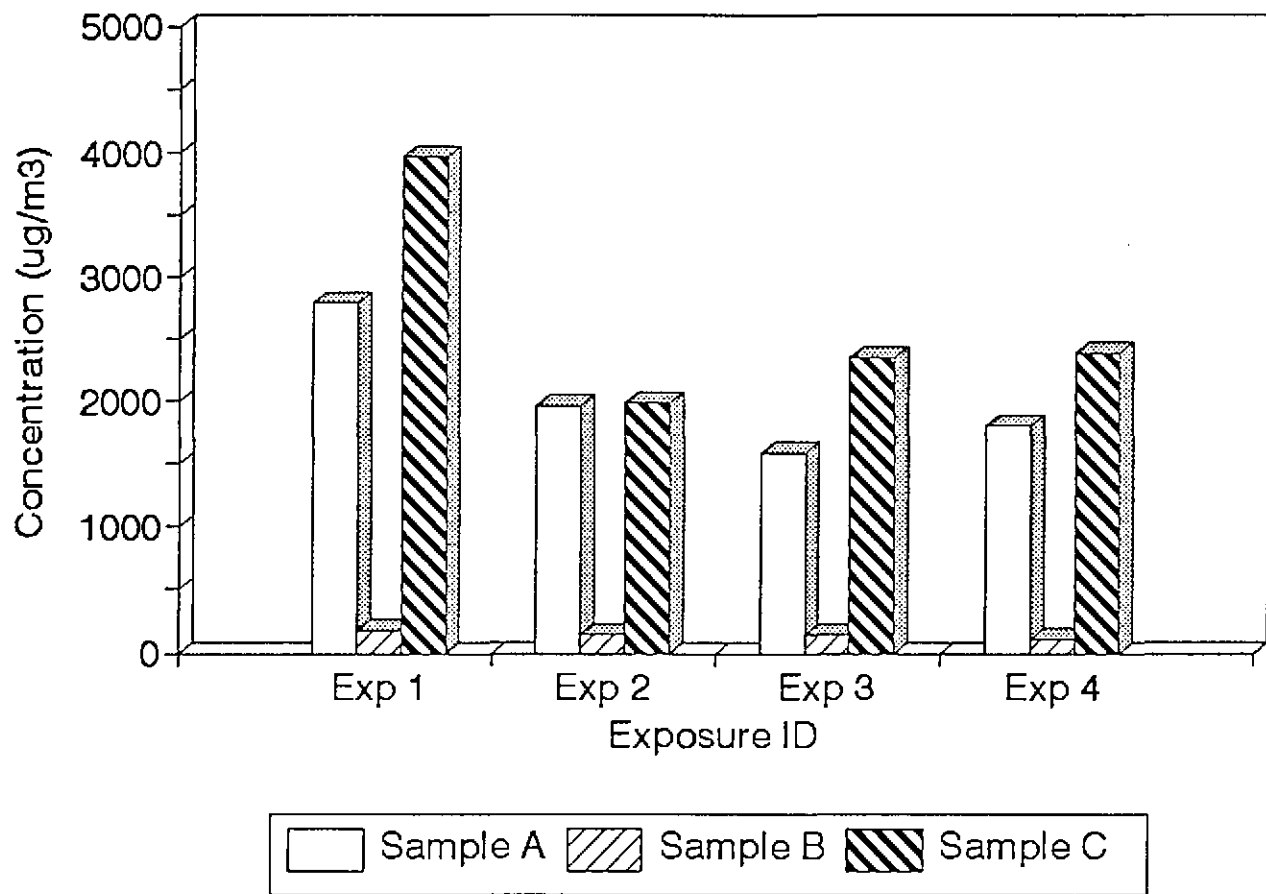


Figure 5-2. Average TVOC concentrations during exposure periods.

In our case, t_x is equal to 60 min and $Q = 8 \times 10^{-3} \text{ m}^3/\text{min}$, which is the sum of the outlet flow rate of the exposure chamber and the total sampling flow. Table 5-2 gives the results for the six tests. As VOC emitters, the source strengths for Sample A and Sample C are of the same order, whereas the source strength of Sample B is about one order of magnitude lower than the other two samples.

TABLE 5-2. THE AMOUNT OF TVOC ELUTED FROM THE SOURCE CHAMBER DURING EACH EXPOSURE (in μg)

Test ID	Sample ID	Exposure 1	Exposure 2	Exposure 3	Exposure 4	Total
1	A	1320	834	616	769	3539
2	B	38	87	109	70	304
3	B	132	55	30	35	252
4	C	2130	908	1060	1320	5418
5	C	1670	1010	1200	972	4852
6	A	1360	1060	904	958	4282

5.4 ESTIMATION OF THE PERCENTAGE OF TVOC EMITTED DURING ONE-HOUR STATIC HEATING PERIOD

The total TVOC emitted in one exposure cycle can be divided into two parts: those from during the one-hour pre-exposure heating period (static mode) and those from the one-hour exposure period (i.e., dynamic mode). The ratio of the two parts can be calculated from:

$$P_s = W_{S0} / (W_E + W_{S1})$$

where P_s is the percentage of TVOC emitted before the dynamic mode started;

W_{S0} is the amount of TVOC in the source chamber before the start of dynamic mode;

W_E is the amount of TVOC eluted during the dynamic mode; and

W_{S1} is the amount of TVOC left in the source chamber after the exposure.

Since the last term is small, a rough estimation can be made by letting $W_{S1}=0$. Data in Table 5-2 can be used as W_E , and W_{S0} is the product of initial concentration (see Table 5-1) and the net volume of the source chamber. The calculated results in Table 5-3 suggest that the majority of the TVOCs were emitted during the exposure period and only less than one quarter were emitted in the static heating period. This can be explained by the "vapor pressure effect"—the elevated TVOC concentration in the air prevented the further emissions from the source. When the air flow started, the chamber air was diluted allowing more VOCs to be emitted from the source.

TABLE 5-3. PERCENTAGE OF TVOCs EMITTED BEFORE DYNAMIC MODE STARTED

Test No.	Exposure 1	Exposure 2	Exposure 3	Exposure 4
1	23%	12%	12%	N/A
2	N/A	N/A	N/A	N/A
3	N/A	N/A	N/A	N/A
4	20%	27%	25%	16%
5	14%	27%	15%	16%
6	23%	16%	13%	15%

5.5 THE CHANGES OF TVOC COMPOSITION DURING THE TEST

Not only did the average TVOC concentration levels change during the test, but the TVOC composition also changed. Figures 5-3 through 5-8 show the different trends for the three samples tested.

For Sample A, the heavier components ($>C_{12}$) were not dominant in the first exposure. In the last exposures, however, they became the most abundant components. In contrast, the lighter components followed a decay trend consistently.

For Sample B, the heavier compounds seemed dominant throughout the test. However, the reasons for differences between the patterns for Test 2 and Test 3 is not known.

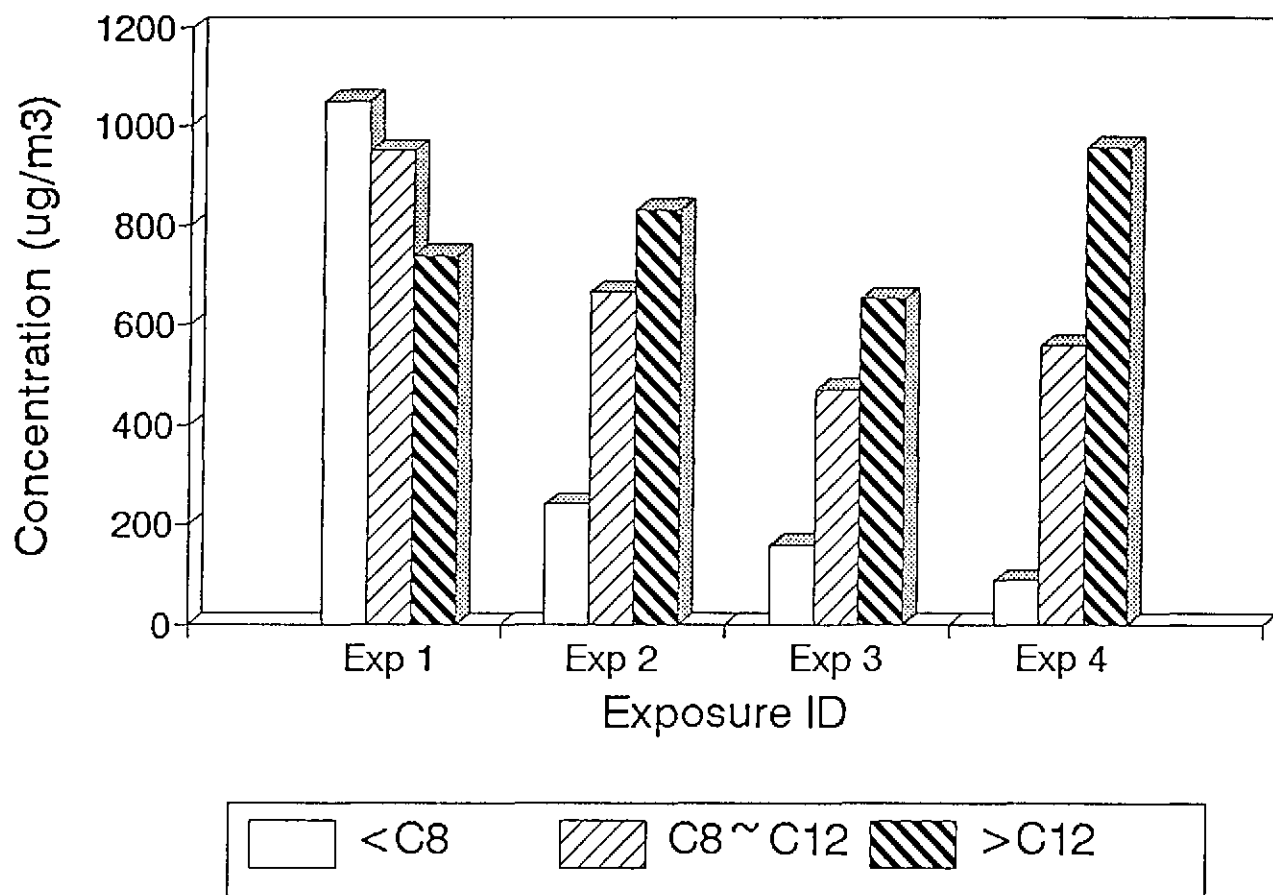


Figure 5-3. The change of TVOC composition during exposure; Sample A, Test 1.

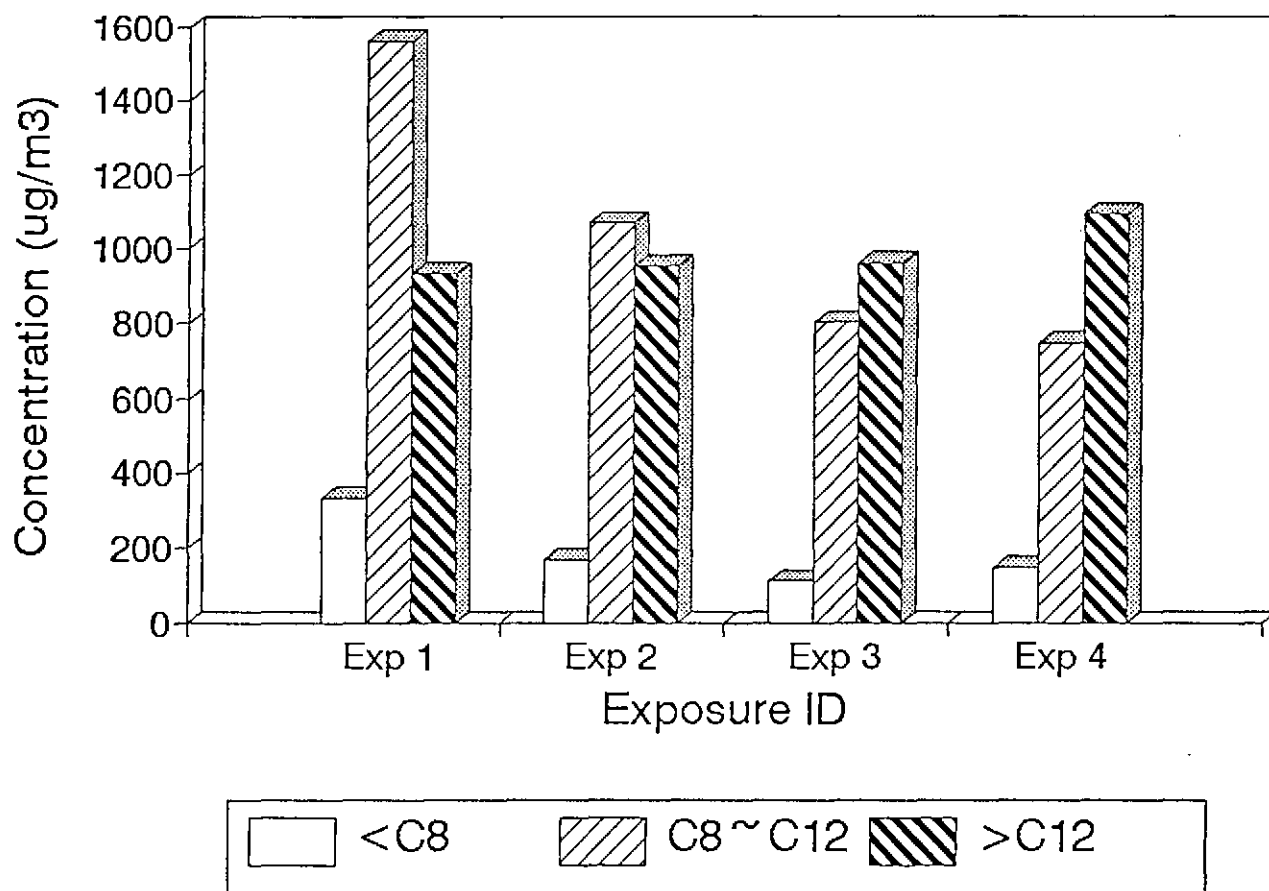


Figure 5-4. The change of TVOC composition during exposures; Sample A, Test 6.

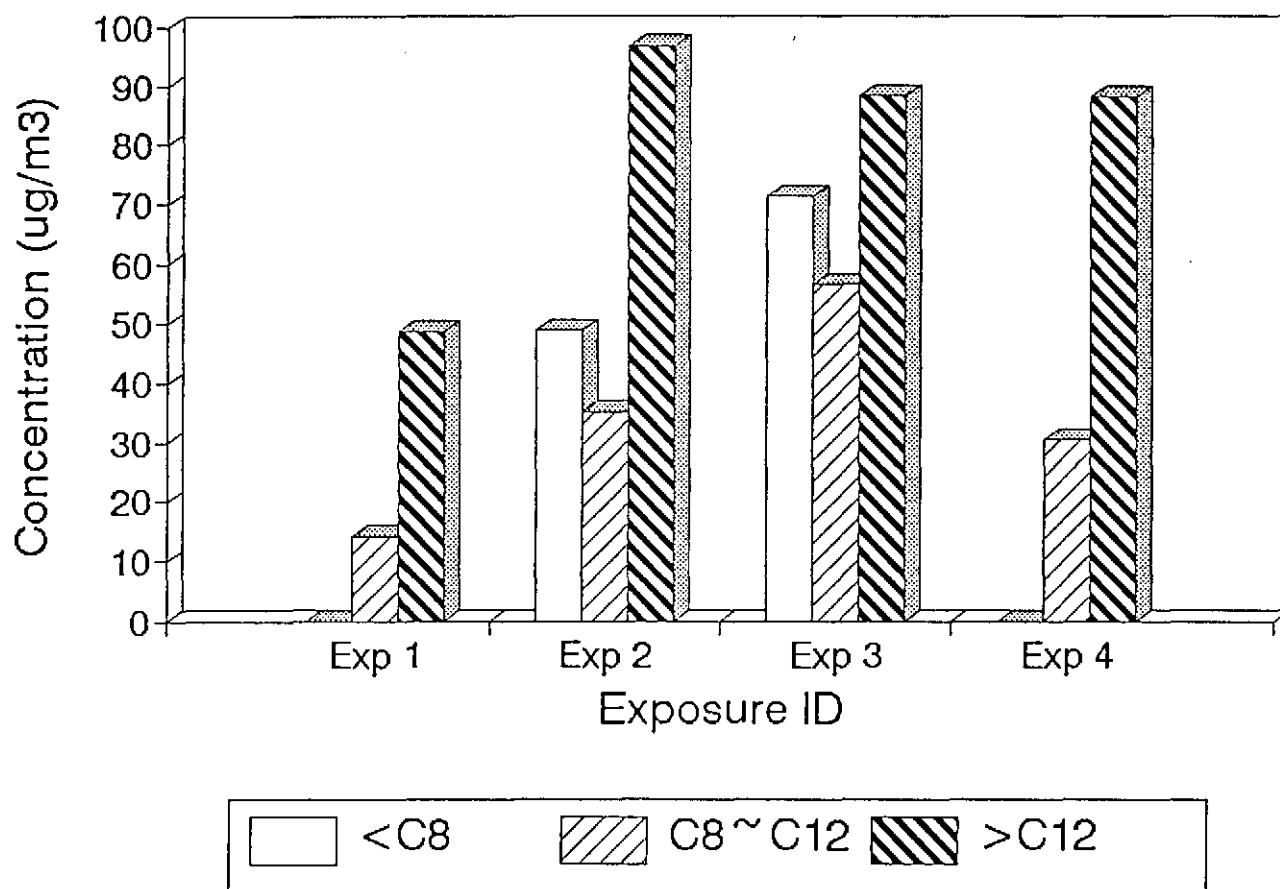


Figure 5-5. The change of TVOC composition during exposures; Sample B, Test 2.

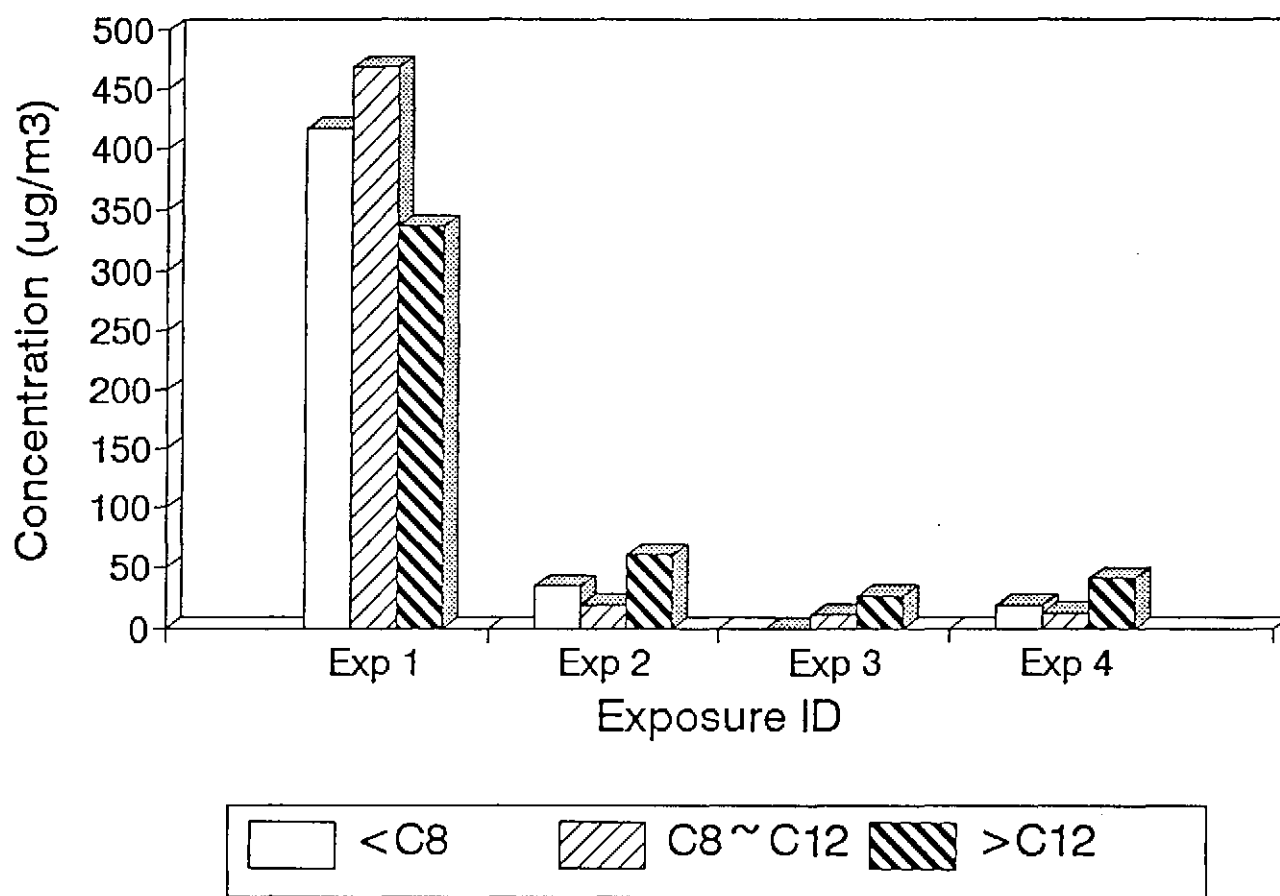


Figure 5-6. The change of TVOC composition during exposures; Sample B, Test 3.

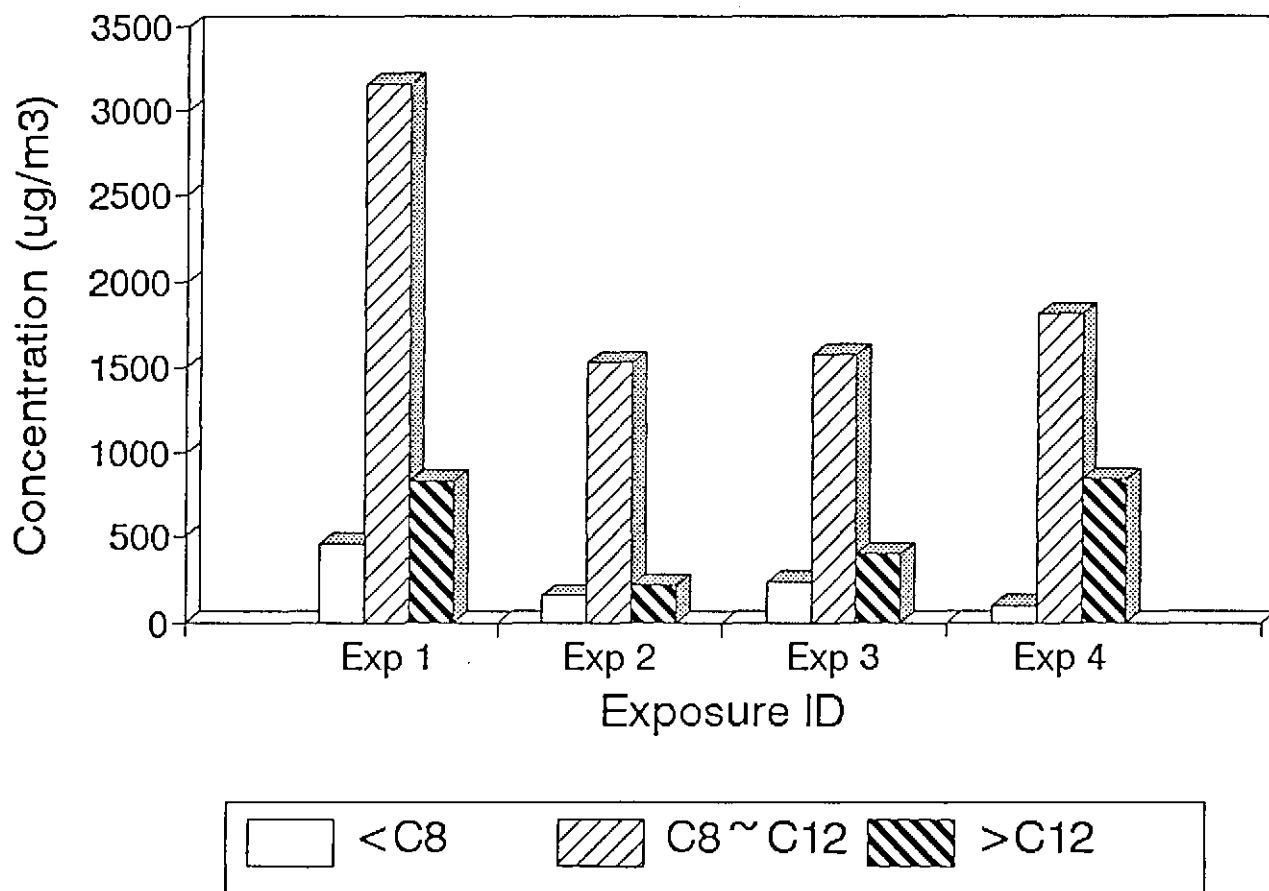


Figure 5-7. The change of TVOC composition during exposures; Sample C, Test 4.

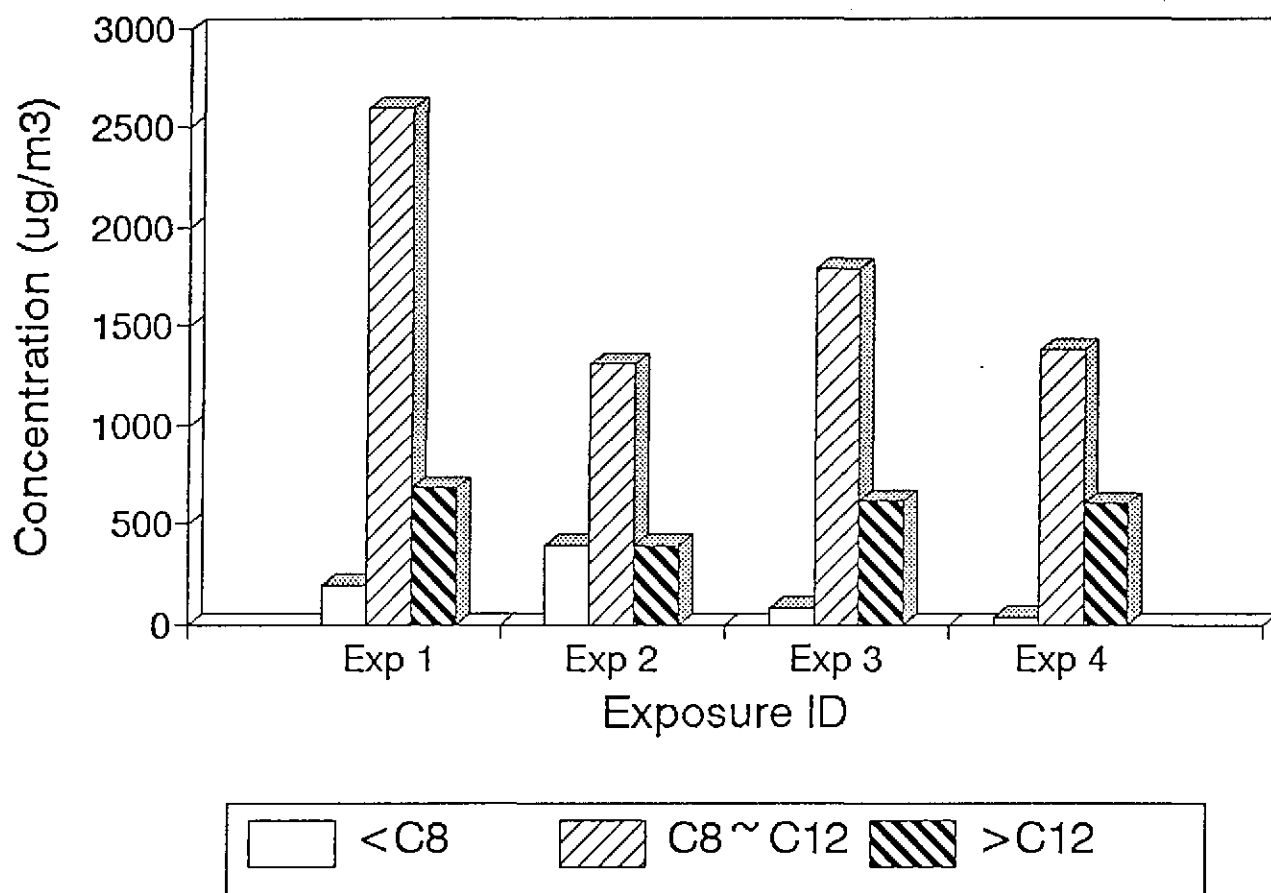


Figure 5-8. The change of TVOC composition during exposures; Sample C, Test 5.

For Sample C, the components in the intermediate range (C_8 ~ C_{12}) were most dominant throughout the test. From Exposure 1 to Exposure 4, the emissions of lighter compounds tended to decay, but the heavier components remained relatively steady.

5.6 EMISSIONS OF INDIVIDUAL COMPOUNDS

5.6.1 Acetic Acid

Sample A was the strongest emitter of acetic acid among the three samples. The average concentration in the exposure period varied from 7 to 50 $\mu\text{g}/\text{m}^3$. A small amount of acetic acid was emitted from Sample C, and no acetic acid was emitted from Sample B.

5.6.2 Benzene

Sample C was the only sample that emitted benzene, and benzene was only found in the first exposure. The average concentration was 36 $\mu\text{g}/\text{m}^3$ for Test 4 and 27 $\mu\text{g}/\text{m}^3$ for Test 5.

5.6.3 Naphthalene

Sample A was the strongest emitter of naphthalene among the three samples. The average concentration in the exposure period varied from 14 to 24 $\mu\text{g}/\text{m}^3$. Smaller amounts of naphthalene were emitted from Sample C (less than 5 $\mu\text{g}/\text{m}^3$), and no naphthalene was emitted from Sample B.

5.6.4 BHT

Again, Sample A was the strongest emitter of BHT (300~400 $\mu\text{g}/\text{m}^3$) and much stronger than the other two samples. Concentration levels for Samples B and C were comparable (10~20 $\mu\text{g}/\text{m}^3$). No significant decay of BHT was apparent during the testing of all three samples.

5.7 CONCENTRATION CHANGES OF INDIVIDUAL COMPOUNDS DURING THE TEST

The concentration changes of individual compounds followed different patterns. Figure 5-9 compares the average concentrations of three compounds from Sample A. During a four-exposure period, the concentration of acetic acid had significant decay (about 50 percent), naphthalene decayed only slightly, and BHT remained fairly stable.

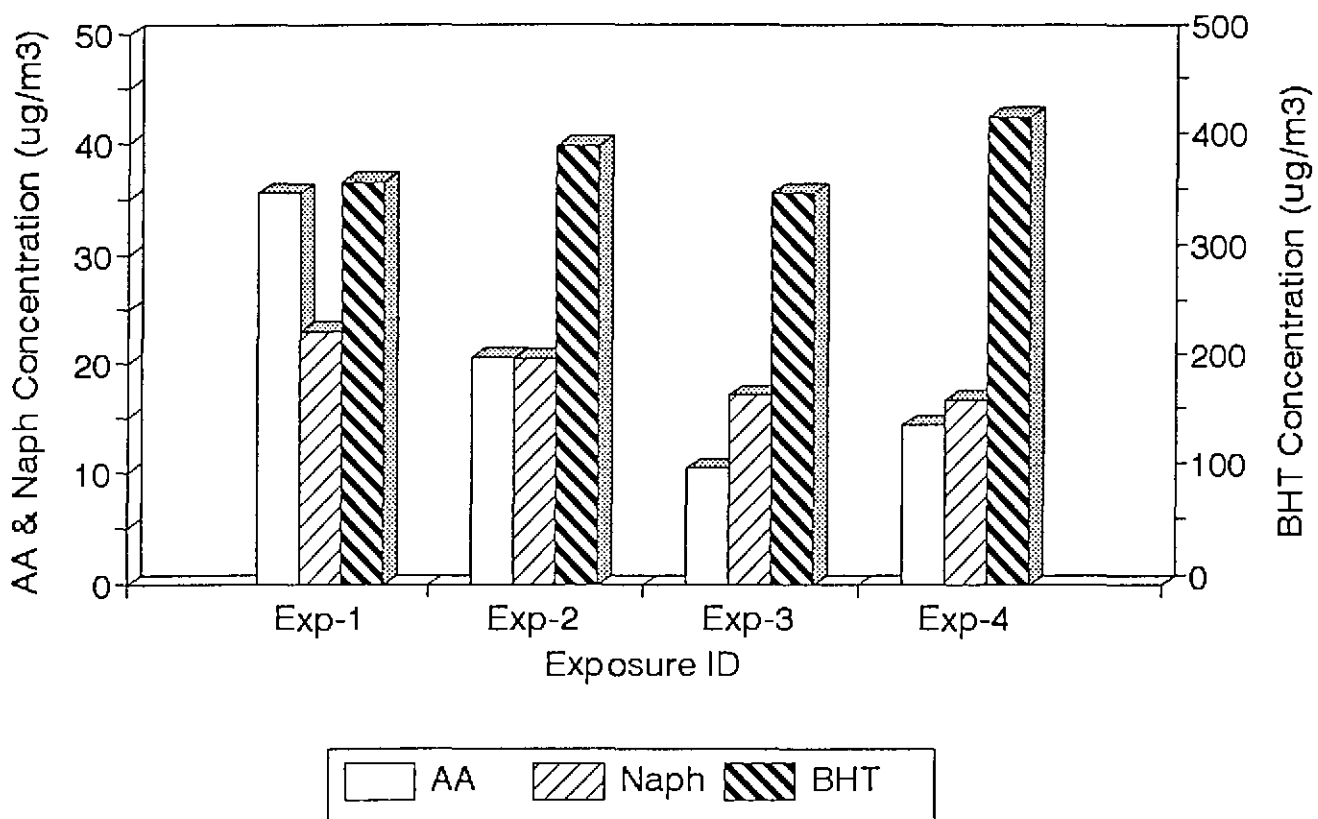


Figure 5-9. Concentration changes for three compounds during exposures the tests of Sample A.

5.8 MOST ABUNDANT VOLATILE ORGANIC COMPOUNDS IN THE CARPET EMISSIONS AS SAMPLED ON MULTI-SORBENT TRAPS

Tables 5-4 through 5-6 outline the 10 most abundant compounds found in the emissions from each of the study sources. The compounds were identifications made from the 60 minute samples taken in the second exposure.

TABLE 5-4. TEN MOST ABUNDANT COMPOUNDS IN THE EMISSIONS FROM SAMPLE A

Experiment 1		Experiment 6	
Compound	Conc. ($\mu\text{g}/\text{m}^3$)	Compound	Conc. ($\mu\text{g}/\text{m}^3$)
Butylatedhydroxytoluene	386	Butylatedhydroxytoluene	407
Toluene	134	Nonanal	108
Nonanal	49	C ₁₂ Alkene	70
Tri(t-butyl) phenol	48	Siloxane Isomer	65
C ₁₂ Alkene	40	Siloxane Isomer	63
C ₁₂ Alkene	34	C ₁₂ Alkene	55
C ₁₂ Alkene	26	Tri(t-butyl) phenol	55
Siloxane Isomer	24	C ₁₂ Alkene	40
n-Hexadecane	27	Siloxane Isomer	35
Isopropanol	26	Unknown	35

TABLE 5-5. TEN MOST ABUNDANT COMPOUNDS IN THE EMISSIONS FROM SAMPLE B

Experiment 2		Experiment 3	
Compound	Conc. $\mu\text{g}/\text{m}^3$	Compound	Conc. $\mu\text{g}/\text{m}^3$
Toluene	44	Toluene	21
Butylatedhydroxytoluene	29	Butylatedhydroxytoluene	18
Siloxane Isomer	7	Acetone	5
Siloxane Isomer	5	Siloxane Isomer	5
Siloxane Isomer	5	Siloxane Isomer	4
Siloxane Isomer	4	Siloxane Isomer	4
C13 Hydrocarbon coelution	3	Siloxane Isomer	3
Siloxane Isomer	3	Siloxane Isomer	3
Siloxane Isomer	3	C13 Hydrocarbon coelution	3
Siloxane Isomer	3	Isopropanol	3

TABLE 5-6. TEN MOST ABUNDANT COMPOUNDS IN THE EMISSIONS FROM SAMPLE C

Experiment 4		Experiment 5	
Compound	Conc. ($\mu\text{g}/\text{m}^3$)	Compound	Conc. ($\mu\text{g}/\text{m}^3$)
Siloxane Isomer	89	Cyclic Alkane	178
C ₁₂ Alkene	64	Nonanal	169
Siloxane Isomer	59	C ₁₂ Alkane	100
Unknown coelution	57	C ₁₂ Alkene	95
Nonanal	53	Alkene	83
Siloxane Isomer	49	Phenol	73
C ₁₂ Alkene	42	C ₁₂ Alkene	69
C ₁₂ Alkene	42	C ₁₂ Alkene	68
Unknown	40	Unknown	63
Unknown coelution	39	Unknown	62

It should be pointed out, however, that the top ten lists were variable depending on when the air samples were taken. Tables 5-7 and 5-8 illustrate such changes by comparing the top 10 compounds in the air sample taken from static chamber prior to exposure 1 and those in the 60-minute sample taken during exposure 2.

TABLE 5-7. THE CHANGE OF TOP TEN LIST DURING TEST 1 (SAMPLE A)

Exposure 1 (Static Chamber)		Exposure 2 (60-min Sample)	
Compound	Conc. ($\mu\text{g}/\text{m}^3$)	Compound	Conc. ($\mu\text{g}/\text{m}^3$)
Toluene	3036	Butylatedhydroxytoluene	386
Isopropanol	1980	Toluene	134
Acetone	1241	Nonanal	49
Sulfur Dioxide	527	Tri(t-butyl) phenol	48
C ₄ Alkene(?)	299	C ₁₂ Alkene	40
Butylatedhydroxytoluene	242	C ₁₂ Alkene	34
Nonanal	146	C ₁₂ Alkene	26
Benzene	139	Siloxane Isomer	24
Siloxane Isomer	99	n-Hexadecane	27
Siloxane Isomer	98	Isopropanol	26

TABLE 5-8. THE CHANGE OF TOP TEN LIST DURING TEST 6 (SAMPLE A)

Exposure 1 (Static Chamber)		Exposure 2 (60-min Sample)	
Compound	Conc. ($\mu\text{g}/\text{m}^3$)	Compound	Conc. ($\mu\text{g}/\text{m}^3$)
Butylatedhydroxytoluene	597	Butlyatedhydroxytoluene	407
Nonanal	357	Nonanal	108
Acetone	343	C ₁₂ Alkene	70
C ₁₂ Alkene	322	Siloxane Isomer	65
Isopropanol	296	Siloxane Isomer	63
Siloxane Isomer	294	C ₁₂ Alkene	55
C ₄ Alkene(?)	289	Tri(t-butyl) phenol	55
Ethanol	251	C ₁₂ Alkene	40
Toluene	232	Siloxane Isomer	35
Siloxane Isomer	232	Unknown	35

SECTION 6

CONCLUSIONS

The objective of this study was to characterize the physical parameters of the test system and the chemical emissions from two specific carpet samples and the empty source chamber under test protocol conditions. The experimental system used for the physical and chemical characterization was identical to the system used by HERL in their bioresponse testing. Although the experimental systems were identical in design and materials, the emissions generated during testing with individual systems could be different based on the following observations:

- Non-uniform heating of chamber surfaces, chamber air, and carpet samples
- Development of air leakage in chambers during testing
- Emissions of pollutants from the source chamber
- Inadequate temperature control because of low precision manual temperature controls

The study results indicate that environmental conditions could not be precisely controlled or reproduced. Therefore, there is no assurance that identical systems would produce identical emissions. More than 200 compounds were emitted by the two carpet samples that were tested. Twenty nine of the 200 compounds (15 percent) were identified by GC/MSD and confirmed, and another 70 percent were tentatively identified. Of the 29 compounds that were confirmed, 58 percent were found in both carpet samples tested and five of the confirmed compounds were observed in all three of the test samples (two carpets and empty chamber). The majority of the emissions from the empty source

chamber were siloxane isomers with most of the emissions being less than the quantification limits of the analytical instruments.

Quantitative differences of some of the individual compounds were observed during an exposure, between the four successive exposure cycles of a single test and between replicate test using different subsets of the same carpet sample. Although the same flow rate and temperature protocols were followed throughout this study and replicate subsets of the same carpet samples were tested, no two exposures produced the same emission profile. During the exposure period, the TVOC concentration and concentrations of some individual compounds decreased with time but did not exhibit an exponential decay. Some of the predominant highly volatile compounds observed in the first exposure were below the detectable limits of the analytical systems in subsequent exposures. The emissions from these tests were a function of the exposure protocol and the time during the exposure at which the samples were collected.

No evidence was found to support the hypothesis that the carpet samples could generate a significant amount of particles under the experimental conditions. The data reported in this document are representative only of the two carpet samples tested during this study. The carpet samples evaluated were not new; some of the emissions may have been of chemicals adsorbed onto the samples during previous use.

SECTION 7

REFERENCES

1. Mason, M., Krebs, K., Roache, N., and Dorsey, J. "Practical Limitations of Multisorbent Traps and Concentrators for Characterization of Organic Contaminants of Indoor Air," Measurement of Toxic and Related Air Pollutants, VIP 25, A&WMA, 1992.
2. TSI Inc., "Porta Count Operation and Service Manual," March 1991.
3. Tichenor, B.A. and Guo, Z. "The Effect of Ventilation on Emission Rates of Wood Finishing Materials, Environmental International, Vol. 17, pp 317-323, 1991.

APPENDIX A
SAMPLING SUMMARY

TABLE A-1. SAMPLING SUMMARY FOR EXPERIMENT 1

Day 1 Experiment 1 March 9, 1993 HERL 93-17 CPSC 1292/60,47,21 Anderson 921 -924 930309.AQ2							
Envirochem 1			Envirochem 2			RTI	
Exposure 1		temp. 21.8°C	RH 33.9%	BP 30.08 in Hg	Dynamic 11:03:50.....12:05:29		T & P
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)	
1.25 SC	2977 3/8	.100	2976 no FID	.100	2974	.100	
5	2979 3/8	.7545					
15	2980 3/8	2.2					
20	2982 3/8	2.98	2981 no FID	2.9			
40	2985 3/15	5.8					
	2993 3/15	LB	2991 3/9	LB	2989	LB	
	2988 3/15	FB	2987 3/9	FB	2986	FB	
Exposure 2		temp. 22.7°C	RH 32.8%	BP 30.18 in Hg	Dynamic 14:16:00.....15:20:40		Temp
1.25 SC	2999 3/15	.100					
1.25 SC	3001 3/15	.100					
60	2995 3/8	9.05					
60	2997 3/15	8.76					
	2994 3/15	LB					
	2998 3/15	FB					
Day 2 Experiment 1 March 10, 1993 930313.AQ2							
Exposure 3		temp. 21.7°C	RH 34.1%	BP 30.04 in Hg	Dynamic 10:41:30.....11:41:48		T & P
1.25 SC	3055 3/8	.100	3064 3/15	.100			
1.25 SC	3058 3/8	.100					
5	3059 3/8	0.8					
15	3047 3/8	2.2					
20	3048 3/8	3.0					
40	3049 3/8	6.0					
	3053 3/8	LB	3060 3/15	LB			
	3051 3/8	LB					
	3054 3/8	FB	3061 3/15	FB			
Exposure 4		temp. 21.7°C	RH 34.1%	BP 30.04 in Hg	Dynamic 14:06:20.....15:06:40		Temp.
1.25 SC	3033 3/8	.100			3066	.100	
60	3034 3/8	9.2					
60	3035 3/8	8.9					
	3031 3/8	LB			3065	LB	

TABLE A-2. SAMPLING SUMMARY FOR EXPERIMENT 2

Day 1 Experiment 2 March 11, 1993 HERL 93-17 CPSC 1768 Anderson 925 - 928 930311.AQ4							
Envirochem 1			Envirochem 2			RTI	
Exposure 1		temp. 20.7°C		RH 29.2%		BP 30.02 in Hg	
						Dynamic 11:03:23.....12:03:51	
						T & P	
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)	
1.25 SC	3013 lost	.100	3030 3/16	.100	3025	.100	
5	3015 lost	.800					
15	3016 lost	202					
20	3006 3/15	3.0	3005 3/16	3.0			
40	3007 3/15	5.9					
	3010 lost	LB	3027 3/16	LB	3024	LB	
	3012 lost	FB	3029 3/16	FB			
Exposure 2		temp. 22.3°C		RH 29.2%		BP 30.07 in Hg	
						Dynamic 14:29:00.....15:34:05	
						Temp.	
1.25 SC	3101 3/8	.100					
1.25 SC	3103 3/8	.100					
60	3105 3/8	9.0					
60	3106 3/8	8.8					
	3107 3/8	LB					
Day 2 Experiment 2 March 12, 1993 930312.AQ4							
Exposure 3		temp. 20.2°C		RH 52.4%		BP 30.23 in Hg	
						Dynamic 9:27:00.....10:36:25	
						T & P	
1.25 SC	3125 lost	.100	3147 3/16	.100			
1.25 SC	3126 lost	.100					
5	3129 3/15	.7595					
15	3118 3/15	2.195					
20	3117 3/15	2.96	3114(Lab Air)a	3.1			
40	3115 3/15	5.996					
	3123 lost	LB	3144 3/16	LB			
	3124 lost	LB	3145 3/16	FB			
Exposure 4		temp. 22.2°C		RH 27.5%		BP 30.22 in Hg	
						Dynamic 13:20:25.....14:32:20	
						Temp.	
1.25 SC	3109 3/15	.100			3149	.100	
60	3112 3/15	3.3					
60	3113 3/15	3.4					
	3108 lost	LB			3148	LB	

TABLE A-3. SAMPLING SUMMARY FOR EXPERIMENT 3

Day 1 Experiment 3 March 23, 1993 HERL 93-19 CPSC 1399 Anderson 940 - 943 930323.AQ4						
Envirochem 1			Envirochem 2		RTI	
Exposure 1	temp. 21.6°C		RH 41.3%		BP 30.36 in Hg	
					Dynamic 10:38:30.....11:52:30 T & P	
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)
1.25 SC			3217 (HERL)	.100 3/24		
1.25 SC	3233 3/22	.100	3214 3/24	.100	3205	.100
5	3229 3/22	.757				
15	3231 3/22	2.27				
20	3209 3/22	2.99	3216 3/24	2.99		
40	3210 3/22	6.02				
60			3208(Lab Air)*	9.09 3/24		
	3226 3/22	LB	3211 3/24	LB	3204	LB
	3227 3/22	FB	3213 3/24	FB		FB
Exposure 2	temp. 23.3°C		RH 59.3%		BP 30.28 in Hg	
					Dynamic 14:17:30.....15:21:20 T & P	
1.25 SC	3237 3/22	.100				
1.25 SC	3221 3/22	.100				
60	3222 3/22	9.05				
60	3225 3/22	8.76				
	3219 3/22	LB				
Day 2 Experiment 3 March 24, 1993 930324.AQ4						
Exposure 3	temp. 22.1°C		RH 58.4%		BP 30.08 in Hg	
					Dynamic 10:38:45.....11:39:35 T & P	
1.25 SC	3256 3/22	.100	3244 3/25	.100		
1.25 SC	3257 3/22	.100				
5	3261 3/22	0.744				
15	3249 3/22	2.25				
20	3250 3/22	2.99				
40	3252 3/22	5.95				
	3255 3/22	LB	3241 3/25	LB		
	3258 3/22	FB	3245 3/25	FB		
251			3246(Lab Air)*	12.6 3/25		
Exposure 4	temp. 22.2°C		RH 61.2%		BP 30.06 in Hg	
					Dynamic 13:49:00.....14:53:10 T & P	
1.25 SC	3253 3/22	.100			3239	.100
60	3240 3/22	8.9				
60	3228 3/22	8.9				
	3247 3/22	LB			3238	LB

TABLE A-4. SAMPLING SUMMARY FOR EXPERIMENT 4

Day 1 Experiment 4 March 25, 1993 HERL 93-20 CPSC 1925/27,56,65 Anderson 944 - 947 930325.AQ3							
Envirochem 1			Envirochem 2			RTI	
Exposure 1	temp. 21.7°C		RH 49.6%		BP 30.18 in Hg		Dynamic 10:54:35.....11:55:50 T & P
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)	
1.25 SC			3298 (HERL)	.100 3/26			
6.67 SC	3287 3/29	0.997	3299 3/25	1.0	3304	.997	
5	3289 4/9	0.748					
15	3291 4/9	2.26					
20	3305 4/9	2.97	3301 3/26	2.99			
40	3306 4/9	6.02					
	3286 lost	LB	3295 3/26	LB	3303	LB	
	3309 3/22	FB	3300 3/26	FB			
Exposure 2	temp. 21.8°C		RH 46.6%		BP 30.23 in Hg		Dynamic 14:20:50.....15:22:10 T & P
6.67 SC			3281 4/8	0.998			
6.67 SC			3282 4/12	0.992			
60					3283 *	9.04	
60			3285 4/8	8.6			
	3280 3/22	LB					
Day 2 Experiment 4 March 26, 1993 930326.AQ3							
Exposure 3	temp. 21.8°C		RH 46.6%		BP 30.23 in Hg		Dynamic 10:49:00.....11:51:10 T & P
6.67 SC	3318 4/12	0.999	3311 3/33	0.993			
6.67 SC	3319 4/12	0.993					
5	3323 lost	0.745					
15	3325 lost	2.25					
20	3329 lost	2.97					
40	3327 4/12	5.96					
	3317 lost	LB	3310 3/30	LB			
	3322 3/29	FB	3313 3/30	FB			
Exposure 4	temp. 21.8°C		RH 46.6%		BP 30.23 in Hg		Dynamic 14:14:45.....15:17:30 T & P
6.67 SC	3330 4/12	0.999			3333	0.993	
60	3315 4/12	3.09					
60	3316 4/12	3.03					
	3324 3/29	LB			3331	LB	

TABLE A-5. SAMPLING SUMMARY FOR EXPERIMENT 5

Day 1 Experiment 5 March 30, 1993 HERL 93-21 CPSC 1508/5,42,21 Anderson 958-961 930330.AQ4						
Envirochem 2			Envirochem 2		RTI	
Exposure 1 temp. 22.4°C RH 42% BP 29.88 in Hg Dynamic 11:43:35.....12:49:15 T & P						
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)
1.25 SC						
6.67 SC	3369 4/13	1.00	3365 4/1	1.00	3353 *	0.99
5	3372 4/13	0.74				
15	3373 4/13	2.24				
20	3383 4/13	2.97	3364 4/1	2.99		
40	3384 4/13	6.02				
60			3361 (LA) 4/5	9.08		
	3367 4/13	LB	3360 4/1	LB	3357 *	LB
	3371 4/13	FB	3363 4/1	FB		
Exposure 2 temp. 23.6°C RH 47.1% BP 29.83 in Hg Dynamic 15:05:15.....16:06:45 T & P						
6.67 SC	3387 4/14	0.99				
6.67 SC	3388 4/14	0.98				
60	3354 4/14	3.02	3347 (LA) 4/5	9.01		
60	3355 4/14	2.89				
	3382 4/13	LB				
Day 2 Experiment 5 March 31, 1993 930401.AQ4						
Exposure 3 temp.20.8 °C RH 49.6% BP29.91 in Hg Dynamic 10:52:15.....11:53:40 T & P						
6.67 SC	3501 4/16	1.02	3390 4/3	1.01		
6.67 SC	3502 4/14	1.01				
5	3505 4/16	0.75				
15	3507 4/16	2.29				
20	3508 4/16	3.02				
40	3509 4/15	5.99				
	3500 4/13	LB	3389 4/1	LB		
	3503 4/13	FB	3391 4/1	FB		
60			3393 (LA) 4/3	9.162		
Exposure 4 temp. 22.7 °C RH 68.3% BP 29.56 in Hg Dynamic 14:11:25.....15:15:00 T & P						
6.67 SC	3349 4/15	0.98			3352 *	0.98
60	3510 4/15	2.83	3395 (LA) 4/3	8.97		
60	3358 4/15	2.78				
	3506 4/13	LB			3348 *	LB

TABLE A-6. SAMPLING SUMMARY FOR EXPERIMENT 6

Day 1 Experiment 6 HERL 93-22 CPSC 1378/45,12,50 Anderson 962-965 930401.AQ1						
Envirochem 1			Envirochem 2		RTI	
Exposure 1	temp. 22.3°C		RH 65.8%		BP 29.62 in Hg	
	Dynamic 10:12:00.....11:14:00		T & P			
Time (min)	Tube ID	Volume (L)	Tube ID	Volume (L)	Tube ID	Volume (L)
1.25 SC			3543 (HERL)*	0.100 4/2		
6.67 SC	3549 4/12	1.00	3545 4/2	1.01	3520 *	1.00
5	3552 4/12	0.80				
15	3553 4/12	2.27				
20	3555 4/12	2.98	3547 4/2	3.00		
40	3556 4/12	6.04				
60						
			3542 4/2	LB	3519 *	LB
			3546 4/2	FB		
Exposure 2	temp. 23.7°C		RH 40.8%		BP 29.6 in Hg	
	Dynamic 13:27:00.....14:29:25		T & P			
6.67 SC	3558 4/12	0.99				
6.67 SC	3559 4/12	0.99				
60	3522 4/12	3.23			3521 *	8.93
60	3523 4/12	2.89				
	3554 4/12	LB				
Day 2 Experiment 6 April 2, 1993 930402.AQ1						
Exposure 3	temp. 21.5°C		RH 41.7%		BP 29.72 in Hg	
	Dynamic 09:12:30.....10:14:10					
6.67 SC	3587 4/12	1.00	3591 4/12	1.01		
6.67 SC	3586 4/12	1.00				
5	3584 4/12	0.76				
15	3593 4/12	2.25				
20	3594 4/12	2.99				
40	3595 4/12	5.97				
	3588 4/12	LB	3597 4/12	LB		
	3585 4/12	FB	3598 4/12	FB		
Exposure 4	temp. 22.2°C		RH 42.2%		BP 29.73 in Hg	
	Dynamic 12:27:10.....13:32:10					
6.67 SC	3569 4/12	0.99			3570	0.99
60	3600 4/12	3.27				
60	3601 4/12	3.06				
	3592 4/12	LB				LB

TABLE A-7. SUMMARY OF DNPH SAMPLE IDS SENT TO RTI

Test ID	Exposure 1		Exposure 2		Exposure 3		Exposure 4	
1	3067		3070	LB	3159	LB	3161	LB
1	3069		3071		3160		3162	
2	2775	LB	3165		2779	LB	3166	LB
2	2776				2781		3167	
2	2777							
3	3186	LB	3185		3184		3183	
3	3187							
3	3189							
4	3180	LB	3181		3171	LB	3173	
4	3178				3172			
4	3179							
5	3175	LB	3177		3638		3639	
5	3188							
5	3169							
6	3633	LB	3636		3632	LB	3631	
6	3634				3630			
6	3635							

LB = Laboratory blank

APPENDIX B
TEMPERATURE DATA

TABLE B-1. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 1 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	36.4	41.9	36.3	42.0
Laboratory Air	22.4	22.7	22.0	22.3
Air in Exposure chamber (Port 3) ¹	23.9	26.0	23.9	27.0
Air in Exposure chamber (Port 5) ²	23.2	24.2	23.0	24.6
Left Panel of Source Chamber	39.1	44.4	39.1	44.5
Right Panel of Source Chamber	38.5	43.8	38.3	44.0
Top Panel of Source Chamber	44.4	50.9	44.0	50.8
Bottom Panel of Source Chamber	65.9	75.0	70.8	74.8
Back Panel of Source Chamber	37.7	44.3	37.4	44.7
Front Panel of Source Chamber	29.1	32.2	28.6	31.8
Sample Backing (inward)	41.5	46.3	40.3	46.9
Sample Fiber (outward) ³	59.0	67.2	62.9	67.1

¹ Marked "P3" in Figure 2-2.² Marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

TABLE B-2. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 2 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	43.4	45.6	42.7	43.6
Laboratory Air	22.2	22.4	21.3	22.1
Air in Exposure chamber (Port 3) ¹	23.3	28.0	27.4	27.9
Air in Exposure chamber (Port 5) ²	22.7	24.3	23.8	24.0
Left Panel of Source Chamber	45.4	50.3	45.9	46.8
Right Panel of Source Chamber	44.2	47.2	44.0	44.8
Top Panel of Source Chamber	47.6	50.8	47.6	48.4
Bottom Panel of Source Chamber	68.2	69.3	68.4	69.0
Back Panel of Source Chamber	44.6	47.7	44.5	45.4
Front Panel of Source Chamber	34.0	36.1	34.4	34.9
Sample Backing (inward)	41.4	44.3	41.6	42.5
Sample Fiber (outward) ³	38.4	42.6	39.6	40.8

¹ Marked "P3" in Figure 2-2.² marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

TABLE B-3. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 3 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	44.4	41.1	39.8	42.2
Laboratory Air	22.7	23.2	22.5	22.6
Air in Exposure chamber (Port 3) ¹	28.1	27.5	24.9	27.2
Air in Exposure chamber (Port 5) ²	25.7	25.8	23.7	25.3
Left Panel of Source Chamber	44.5	42.7	39.5	42.6
Right Panel of Source Chamber	44.1	42.2	38.7	41.4
Top Panel of Source Chamber	49.9	46.1	45.6	48.8
Bottom Panel of Source Chamber	68.9	67.7	67.7	68.8
Back Panel of Source Chamber	47.0	43.4	40.3	43.3
Front Panel of Source Chamber	34.1	31.9	31.3	32.6
Sample Backing (inward)	43.1	39.6	37.5	40.7
Sample Fiber (outward) ³	42.9	39.1	37.5	40.5

¹ Marked "P3" in Figure 2-2.² Marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

TABLE B-4. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 4 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	36.4	41.9	36.3	42.0
Laboratory Air	44.4	41.1	39.8	42.2
Air in Exposure chamber (Port 3) ¹	22.7	23.2	22.5	22.6
Air in Exposure chamber (Port 5) ²	28.1	27.5	24.9	27.2
Left Panel of Source Chamber	25.7	25.8	23.7	25.3
Right Panel of Source Chamber	44.5	42.7	39.5	42.6
Top Panel of Source Chamber	44.1	42.2	38.7	41.4
Bottom Panel of Source Chamber	49.9	46.1	45.6	48.8
Back Panel of Source Chamber	68.9	67.7	67.7	68.8
Front Panel of Source Chamber	47.0	43.4	40.3	43.3
Sample Backing (inward)	34.1	31.9	31.3	32.6
Sample Fiber (outward) ³	43.1	39.6	37.5	40.7

¹ Marked "P3" in Figure 2-2.² Marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

TABLE B-5. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 5 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	43.2	44.3	42.7	41.6
Laboratory Air	23.0	23.9	22.4	22.4
Air in Exposure chamber (Port 3) ¹	27.9	28.7	27.3	27.1
Air in Exposure chamber (Port 5) ²	25.7	26.5	25.0	24.7
Left Panel of Source Chamber	45.6	47.0	45.0	43.3
Right Panel of Source Chamber	46.0	47.5	45.4	43.6
Top Panel of Source Chamber	48.3	50.0	47.8	45.8
Bottom Panel of Source Chamber	71.4	71.7	71.1	71.6
Back Panel of Source Chamber	49.3	51.4	49.3	47.4
Front Panel of Source Chamber	34.5	36.0	34.2	33.4
Sample Backing (inward)	69.4	69.4	68.7	68.9
Sample Fiber (outward) ³	56.3	57.2	56.0	55.7

¹ Marked "P3" in Figure 2-2.² Marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

TABLE B-6. AVERAGE TEMPERATURE AT 12 LOCATIONS FOR TEST 6 (IN °C)

Thermocouple Location	Exposure 1	Exposure 2	Exposure 3	Exposure 4
Air in Source Chamber	39.6	39.9	40.1	41.6
Laboratory Air	22.8	23.7	22.3	22.4
Air in Exposure chamber (Port 3) ¹	26.9	27.8	26.6	26.9
Air in Exposure chamber (Port 5) ²	25.5	26.3	24.8	24.9
Left Panel of Source Chamber	44.6	44.3	45.8	47.4
Right Panel of Source Chamber	41.6	41.8	42.7	43.9
Top Panel of Source Chamber	50.9	50.0	51.7	53.9
Bottom Panel of Source Chamber	75.0	75.3	75.9	76.3
Back Panel of Source Chamber	49.2	48.6	49.9	52.3
Front Panel of Source Chamber	30.0	31.0	30.1	30.7
Sample Backing (inward)	71.4	71.7	72.1	72.4
Sample Fiber (outward) ³	46.4	46.8	46.7	47.8

¹ Marked "P3" in Figure 2-2.² Marked "P5" in Figure 2-2.³ Sample surface in contact with heated chamber bottom.

APPENDIX C

CALIBRATION DATA FOR TEMPERATURE AND HUMIDITY PROBES

TABLE C-1. CALIBRATION OF THERMOCOUPLE 1 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.4	-0.6	0.04
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.00
03/29/93	Warm Water	46.3	13	45.7	-0.6	0.05
04/08/93	Ice/Water	0.0	16	-0.1	-0.1	0.04
04/08/93	Warm Water	47.8	16	47.4	-0.4	0.06

TABLE C-2. CALIBRATION OF THERMOCOUPLE 2 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.2	-0.8	0.00
03/29/93	Ice/Water	0.0	13	0.2	0.2	0.00
03/29/93	Warm Water	46.3	13	45.8	-0.5	0.08
04/08/93	Ice/Water	0.0	16	0.4	0.4	0.00
04/08/93	Warm Water	47.8	16	47.4	-0.4	0.08

TABLE C-3. CALIBRATION OF THERMOCOUPLE 3 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0
03/04/93	Boiling Water	100	16	99	-1.0	0.09
03/29/93	Ice/Water	0.0	13	0.2	0.2	0.03
03/29/93	Warm Water	46.3	13	46	-0.3	0.1
04/08/93	Ice/Water	0.0	16	0.3	0.3	0.04
04/08/93	Warm Water	47.8	16	47.7	-0.1	0.05

TABLE C-4. CALIBRATION OF THERMOCOUPLE 4 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	00	16	0.1	0.1	0.03
03/04/93	Boiling Water	100	16	99.2	-0.8	0.02
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.04
03/29/93	Warm Water	46.3	13	45.7	-0.6	0.00
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.00
04/08/93	Warm Water	47.8	16	47.6	-0.2	0.11

TABLE C-5. CALIBRATION OF THERMOCOUPLE 5 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.3	-0.7	0.03
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.05
03/29/93	Warm Water	46.3	13	45.9	-0.4	0.04
04/08/93	Ice/Water	0.0	16	0.1	0.1	0.05
04/08/93	Warm Water	47.8	16	47.6	-0.2	0.00

TABLE C-6. CALIBRATION OF THERMOCOUPLE 6 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.07
03/04/93	Boiling Water	100	16	99.1	-0.9	0.04
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.05
03/29/93	Warm Water	46.3	13	45.8	-0.5	0.00
04/08/93	Ice/Water	0.0	16	N/A	N/A	N/A
04/08/93	Warm Water	47.8	16	N/A	N/A	N/A

TABLE C-7. CALIBRATION OF THERMOCOUPLE 7 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.2	-0.8	0.05
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.04
03/29/93	Warm Water	46.3	13	46	-0.3	0.05
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.04
04/08/93	Warm Water	47.8	16	47.7	-0.1	0.06

TABLE C-8. CALIBRATION OF THERMOCOUPLE 8 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.1	-0.9	0.05
03/29/93	Ice/Water	0.0	13	0.2	0.2	0.00
03/29/93	Warm Water	46.3	13	46	-0.3	0.05
04/08/93	Ice/Water	0.0	16	N/A	N/A	N/A
04/08/93	Warm Water	47.8	16	N/A	N/A	N/A

TABLE C-9. CALIBRATION OF THERMOCOUPLE 9 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.4	-0.6	0.00
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.00
03/29/93	Warm Water	46.3	13	45.9	-0.4	0.06
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.02
04/08/93	Warm Water	47.8	16	47.7	-0.1	0.00

TABLE C-10. CALIBRATION OF THERMOCOUPLE 10 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.2	-0.8	0.04
03/29/93	Ice/Water	0.0	13	0.2	0.2	0.00
03/29/93	Warm Water	46.3	13	46.2	-0.1	0.05
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.04
04/08/93	Warm Water	47.8	16	47.7	0.06	0.06

TABLE C-11. CALIBRATION OF THERMOCOUPLE 11 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.1	0.1	0.00
03/04/93	Boiling Water	100	16	99.3	-0.7	0.03
03/29/93	Ice/Water	0.0	13	0.1	0.1	0.00
03/29/93	Warm Water	46.3	13	46.3	0.0	0.04
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.00
04/08/93	Warm Water	47.8	16	47.8	0.0	0.06

TABLE C-12. CALIBRATION OF THERMOCOUPLE 12 (UNIT: °C)

Date	Temperature Media	Actual Temp.	No. of Readings	Mean Temp.	Error	STD
03/04/93	Ice/Water	0.0	16	0.2	0.2	0.00
03/04/93	Boiling Water	100	16	100.3	0.3	70.04
03/29/93	Ice/Water	0.0	13	0.2	0.2	0.05
03/29/93	Warm Water	46.3	13	45.9	-0.4	0.05
04/08/93	Ice/Water	0.0	16	0.2	0.2	0.04
04/08/93	Warm Water	47.8	16	47.7	-0.1	0.02

TABLE C-13. CALIBRATION OF HUMIDITY PROBE 1

Solution of Salt	Temp. (°C)	R.H. (%)	Readings ¹ (Volt)	Temp. (°C)	R.H. (%)	Readings ² (Volt)
LiCl	22	11.3	0.62	21	11.6	0.60
LiCl	22	11.3	0.64	21	11.6	0.61
LiCl	23	10.9	0.63	21	11.6	0.62
NaCl	22	75.5	3.46	21	75.6	3.44
NaCl	22	75.5	3.44	21	75.6	3.44
NaCl	23	75.3	3.45	21	75.6	3.45

¹ Calibrated on 03/23/93² Calibrated on 04/03/93

TABLE C-14. CALIBRATION OF HUMIDITY PROBE 2

Solution of Salt	Temp. (°C)	R.H. (%)	Readings ¹ (Volt)	Temp. (°C)	R.H. (%)	Readings ² (Volt)
LiCl	22	11.3	0.73	21	11.6	0.71
LiCl	22	11.3	0.74	21	11.6	0.71
LiCl	23	10.9	0.75	21	11.6	0.72
NaCl	22	75.5	3.52	21	75.6	3.51
NaCl	22	75.5	3.53	21	75.6	3.51
NaCl	23	75.5	3.51	21	75.6	3.52

¹ Calibrated on 03/23/93² Calibrated on 04/03/93