



## Project Summary

# Characterization of Emissions from Carpet Samples Using a 10-Gallon Aquarium as the Source Chamber

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As part of Phase I of a 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. The full report 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. 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 that 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: two previously used carpet samples plus mock (empty bags) samples.

An 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 chemi-

cal emissions characterization because of poor reproducibility, nonuniform thermal conditions, and emissions from the source chamber itself. The 1-h bake cycle prior to the dynamic mode is not typical of indoor air characterization methods.

*This Project Summary was developed by EPA's Air and Energy Engineering Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).*

### Introduction

In 1992, researchers at Anderson Laboratories, Inc., of Dedham, MA, reported toxic effects in experimental mice exposed to emissions from selected carpet samples. Because of the potential public health significance of their reported findings, the U.S. Environmental Protection Agency (EPA) and the Consumer Product Safety Commission (CPSC) initiated studies in 1993 to evaluate Anderson's experimental method and to replicate the reported findings. In addition to a comprehensive toxicity screen and microbial characterization, the EPA test plan (Phase I) called for a thorough chemical characterization of emissions from carpet samples collected by CPSC. This report summarizes the findings of the carpet emissions characteriza-



tion performed by Acurex Environmental Corporation under EPA Contract 68-DO-0141 at the Indoor Source Characterization Laboratory of EPA's Air and Energy Engineering Research Laboratory (AEERL).

The experimental system used in this work was based on a protocol provided by Anderson Laboratories and was identical to that used by EPA's Health Effects Research Laboratory (HERL) for replicating Anderson Laboratories' tests.

The objectives of the study were two-fold. First, emissions tests were performed to identify potential toxic volatile organic compounds in the emissions from carpet samples and to determine the concentration levels during the exposure. Second, because of the unconventional nature of the method, the experimental system was characterized to determine test conditions and the background emissions from the source chamber.

## Method

### Test System and Protocol

The test system consisted of four functional parts: air supply system, source chamber, exposure chamber, and air flow control. During a test, the carpet sample was placed in the source chamber (a 10-gal — 38 L — glass aquarium) and heated to elevated temperatures. Humidified zero-grade air was then introduced to the chamber to carry the emissions to the exposure chamber, where the experimental animals are tested. Air samples were collected for chemical analysis from the source chamber after the 1-h static period and from the exposure chamber during the dynamic period.

Duplicate tests were conducted on each of the three carpet samples randomly received from CPSC. Samples A and C were previously used carpet samples collected by CPSC, and Sample B was received as an empty bag to indicate an empty chamber test. The samples were received as follows: Sample A for tests 1 and 6; Sample B for tests 2 and 3; and Sample C for tests 4 and 5. Each test consisted of four 1-h exposure periods and took two days to complete.

### Test Parameters

The major test parameters were

Size of carpet sample:	2,900 cm <sup>2</sup>
Observed sample temperature:	~50°C
(at the surface of the carpet backing)	~70°C
(hot spot on the fiber side)	

Target air temperature in source chamber:	37 ± 3°C
Observed average air temperature in source chamber:	~41°C
Volume of source chamber:	38 L
Source chamber air flow rate:	7 L/min
Target relative humidity of inlet air:	45 ± 5%

### Volatile Organic Compound Analysis

The volatile organic compounds (VOCs) were collected on multisorbent traps during chamber testing and analyzed by thermal desorption-capillary gas chromatograph (GC) equipped with a mass selective detector (MSD) for compound identification and a flame ionization detector (FID) for compound quantification. Individually identified compounds were quantified, and the emissions of total volatile organic compounds (TVOCs) were estimated.

The VOCs were measured using sampling and analysis procedures developed and implemented in previous emissions testing at AEERL. These procedures included daily tuning of the MSD for identification, five-point calibration of the GC/FID for quantification, analysis of daily check samples, analysis of field and laboratory blanks, and verification of sorbent trap background concentrations (blanks).

### Measurements of Particle Concentrations

The instrument used for monitoring particle concentration was a model 8010 PortaCount particle counter (TSI). 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<sup>3</sup>). Particle concentrations between 0 and 5 × 10<sup>5</sup> P/cm<sup>3</sup> can be measured with this instrument. For comparison purposes, the particle concentrations in the laboratory air were measured before and after each exposure.

## Results

### Characterization of Physical Parameters

Tracer gas measurements showed that the air in the exposure chamber was well mixed. The inlet and outlet air flow rates were measured with an electronic bubble flowmeter at the start of each test to make sure the difference between the two flow rates was within 10%. Comparison of the outlet air flow prior to each exposure and after the completion of the test indicated

increased leakiness of the system during testing due to heated duct tape and poor seals in the system. The pressure difference between exposure chamber and the laboratory air was negligible.

The source chamber was heated with heating pads from outside, creating a poorly controlled thermal environment. Temperature data from 12 locations were collected at a frequency of one reading every minute and logged by a computer. Figure 1 shows an example of temperature profiles in the source chamber.

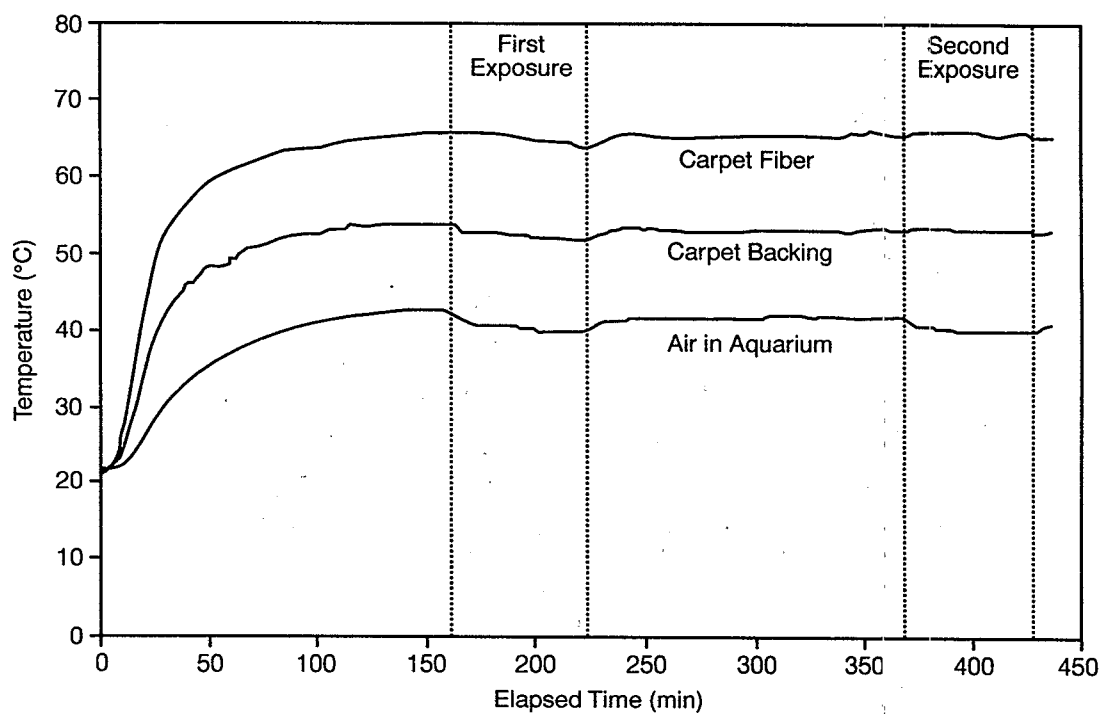
### Total Volatile Organic Compounds

Peak TVOC concentrations found in the source chambers at the end of the initial 1-h static heating period were approximately 10 mg/m<sup>3</sup> for each carpet sample. The background contribution to the TVOC concentration from the source chamber was <1 mg/m<sup>3</sup> and represented <10% of the TVOC during the carpet test. Peak TVOC concentrations averaged 2-8 mg/m<sup>3</sup> in subsequent samples collected from the source chamber after the 1-h static periods with the same carpet sample. Each exposure followed a pattern of an initially high concentration followed by a continuous decay in concentration during the 1-h exposure period because of the dilution by clean air (Figure 2). During subsequent 1-h exposure periods, the source strength was lower and variable (Figure 3).

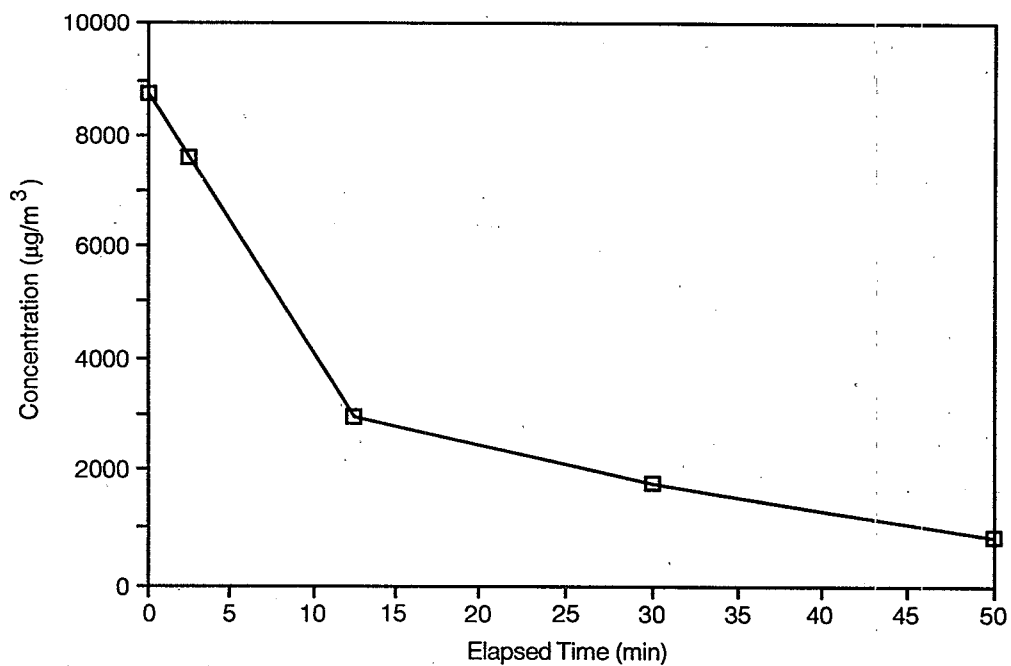
### Individual and Classes of Compounds in Carpet Emissions

More than 200 compounds were observed in the carpet emissions. About 15% were identified and confirmed by interlaboratory comparison of GC/MS analysis, another 70% were tentatively identified, and the remaining 15% were not identified. The identified compounds fell into the following classes: alkanes, alkenes, cycloalkanes, cycloalkenes, oxygenated hydrocarbons, one- or two-ring aromatic hydrocarbons, siloxanes, and phenols. Oxygenated hydrocarbons, aromatic hydrocarbons, and siloxanes were also emitted from an empty source chamber. Table 1 lists compounds identified by Acurex for each test sample. Table 2 shows the classes of compounds found in the emissions from each sample.

Concentrations of the predominant compounds, including those identified by HERL as potentially toxic for each sample, are listed in Table 3. The data are the average concentration from a 60-minute sample during Exposure 2 of each test.



**Figure 1.** Typical temperature profiles in the source chamber.



**Figure 2.** Observed TVOC emissions from Sample A (Exposure 1, Test 1).

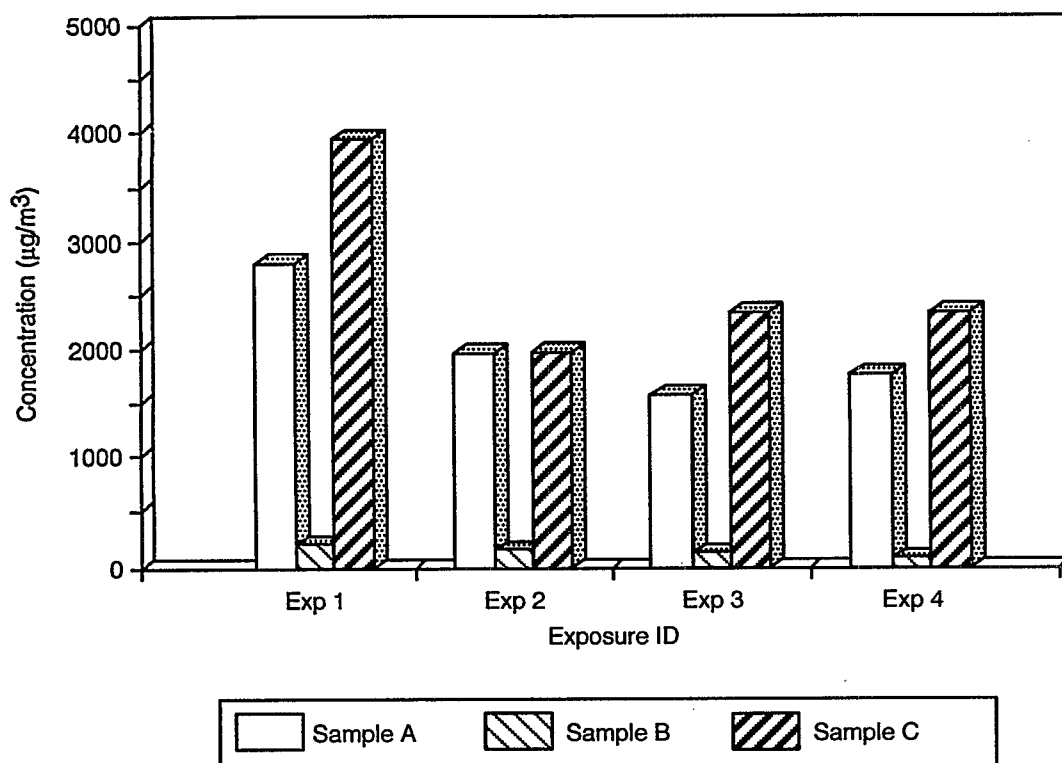


Figure 3. Average TVOC concentrations in the exposure chamber during four 1-h exposure periods of a test.

### Particles

The particle concentrations in the chamber air were one to two orders of magnitude lower than those found in laboratory air.

### 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:

- Nonuniform 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. Of the 200 compounds, 29 (15%) were identified by GC/MSD and confirmed, and another 70% were tentatively identified. Of the 29 compounds that were confirmed, 58% 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). Most 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 tests 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 Exposure 1 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.

**Table 1.** Individual Compounds Identified in the Three Samples

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

**Table 2.** Classes of Compounds Identified in the Three Samples

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

**Table 3. Predominant Emissions by Test**  
(Concentration unit:  $\mu\text{g}/\text{m}^3$ )

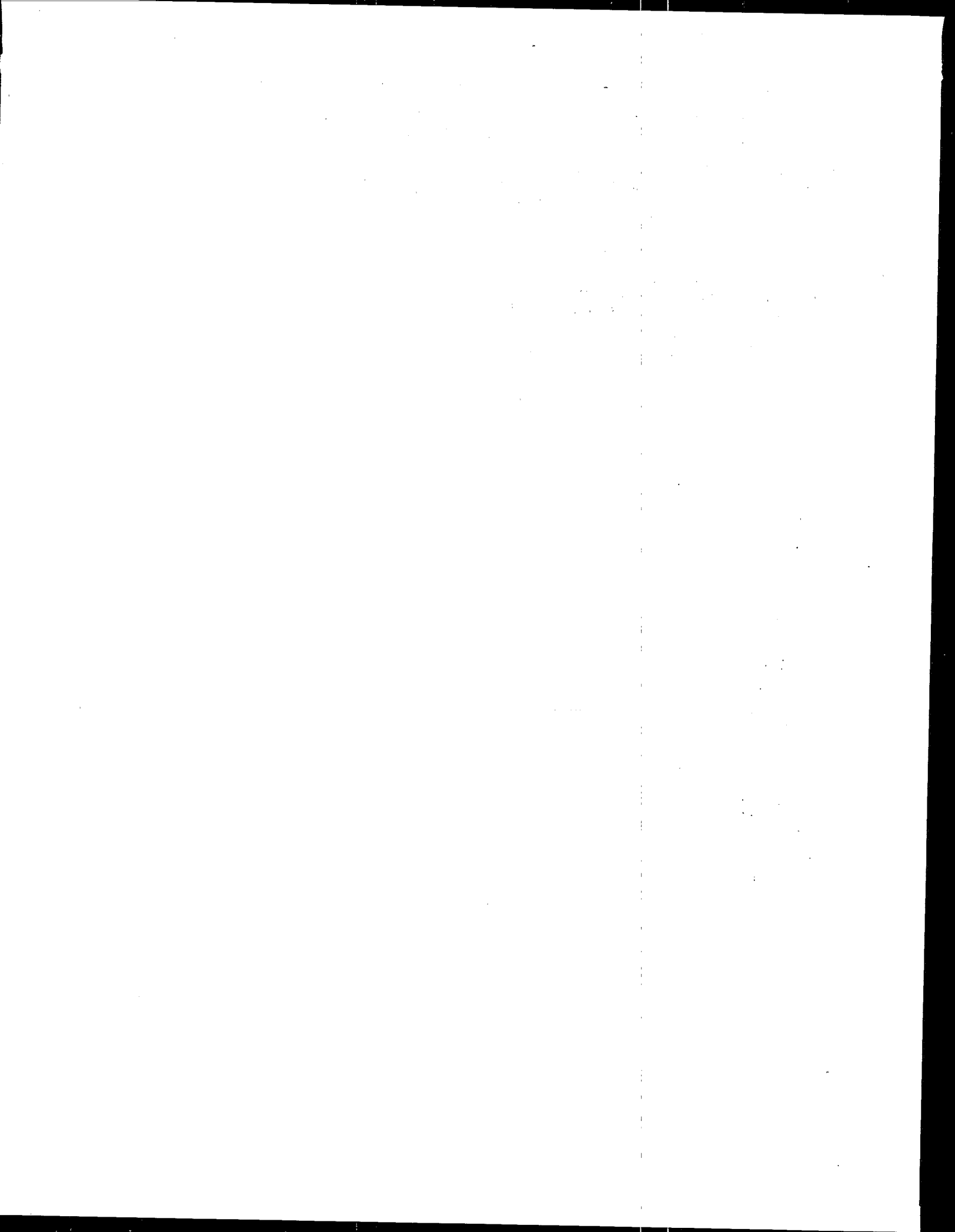
Compound	Sample <sup>1</sup> /Test	A/1	A/6	B/2	B/3	C/4	C/5
Butylatedhydroxytoluene (BHT) <sup>2</sup>		386	407	29	18	2	21
Acetic Acid <sup>2</sup>		14	27	ND	ND	ND	2
Naphthalene <sup>2</sup>		19	22	ND	ND	ND	3
Toluene		134	10	44	21	4	7
Nonanal		49	108	ND	ND	53	169 <sup>3</sup>
Tri(t-butyl) phenol		48	55	ND	ND	ND	ND
Phenol		ND	ND	ND	ND	15	73
Siloxane isomer (retention time 59.9 min)		25	32	6	4	73	39
TVOCs		1737	2198	182	115	1890	3181

<sup>1</sup>Samples: A and C = carpet; B = empty chamber used as a control.

<sup>2</sup>Identified as potentially toxic by HERL.

<sup>3</sup>Coelution of nonanal and siloxane isomer.

ND = not detectable by analytical system.



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Mark A. Mason is the EPA Project Officer (see below).

The complete report, entitled "Characterization of Emissions from Carpet Samples Using a 10-Gallon Aquarium as the Source Chamber," (Order No. PB94-210002;

Cost: \$27.00, subject to change) will be available only from:

National Technical Information Service

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