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**Investigation of the effects of various cure parameters on
the release of 4-phenylcyclohexene from carpet backed with
styrene-butadiene rubber latex**

LeForce, Mary Hall, M.S.

The University of Arizona, 1991

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INVESTIGATION OF THE EFFECTS OF VARIOUS CURE PARAMETERS
ON THE RELEASE OF 4-PHENYLCYCLOHEXENE FROM CARPET
BACKED WITH STYRENE-BUTADIENE RUBBER LATEX

by

MARY HALL LEFORCE

A Thesis Submitted to the Faculty of the
DEPARTMENT OF PHARMACOLOGY AND TOXICOLOGY
In Partial Fulfillment of the Requirements
for the Degree of

MASTER OF SCIENCE
WITH A MAJOR IN TOXICOLOGY

In the Graduate College
THE UNIVERSITY OF ARIZONA

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ABSTRACT

The effects of cure time, cure temperature, and air circulation on the release of 4-phenylcyclohexene (4-PCH) in tufted carpet backed with carboxylated styrene-butadiene rubber (SBR) latex were examined in this research project. 4-PCH is a suspect cause of certain "building-related" illnesses such as headache, upper respiratory and eye irritation, and fatigue.

A standardized method for preparing carpet backed with SBR latex and analyzing its 4-PCH content was developed. The cure parameters investigated included three cure temperatures (225, 250 and 275 °F), six cure times (0, 5, 10, 20, 40 and 60 minutes), and three air circulation rates (zero, three air exchanges per minute, and ten air exchanges per minute with 1500 linear feet per minute airflow). Increased airflow (1500 linear feet per minute) over the carpet surface produced the greatest impact on 4-PCH release from carpet. Increasing the cure temperature and cure time also significantly increased the release of 4-PCH.

INTRODUCTION

Indoor Air Pollution

Indoor air quality has recently been targeted as a major health concern in the United States. Many indoor air quality problems have resulted from reduced ventilation rates and maximal use of recirculated air in an effort to save energy costs. These energy conservation programs were greatly encouraged in an effort to conserve fossil fuels. As a result, ventilation rates were reduced and buildings were sealed to conserve energy, thereby reducing operating costs. Of the 1,200 Health Hazard Evaluations the National Institute of Occupational Safety and Health (NIOSH) conducted between 1971 and 1978, six involved indoor air quality (0.5%) (Millar 1989). From 1978 to 1980, the percentage of Health Hazard Evaluations involving indoor air quality rose to 12%, and more recently has reached 20%.

Indoor air quality problems may arise from a variety of sources such as human metabolic activity, smoking, structural components of a building, biological contamination, office and mechanical equipment, and outside air pollutants (Millar 1989). Common symptoms reported during indoor air pollution episodes include eye irritation, dry throat, headache, fatigue, sinus congestion, dizziness, nausea, sneezing and shortness of breath. This phenomenon has been referred to as "Sick Building Syndrome" and may

produce effects such as missed work, reassignment, and even termination among workers.

Of the approximately four million non-industrial commercial buildings in the United States, 20 to 30 percent are thought to be "problem" buildings (Morey 1990). A problem building is one in which sick building syndrome or a building-related illness has occurred. Building-related illnesses have been found to be caused by exposure to indoor air contaminants, leading to conditions such as Legionnaires disease, asthma, and dermatitis. Symptoms are often displayed in over 20 percent of the building's occupants and may disappear when the occupant leaves the building.

Over half of the indoor air quality investigations conducted by NIOSH have resulted in a finding of inadequate ventilation (Millar 1989). Heating, ventilation and air conditioning (HVAC) conditions that were found to exacerbate indoor air quality problems included migration of odors or chemicals between areas of a building and entrapment of exhaust emissions due to improper placement of exhaust and intake stacks. In certain instances, HVAC systems had become contaminated with microorganisms and odors. Reduced fresh make-up air from outside resulted in the concentration of indoor air pollutants.

In approximately seventy buildings investigated (Morey 1990), 70 percent were found to have operational problems

with the HVAC system, 75 percent had maintenance problems and 47 percent had design deficiencies, demonstrating that HVAC system deficiencies are often associated with indoor air quality problems. In another study of "sick buildings" conducted by this group, outdoor volatile organic compound (VOC) concentrations averaged 130 ug/m³. Indoor VOC concentrations averaged 660 ug/m³, with some as high as 2,000 ug/m³.

New Carpet Emissions

New carpeting has been suspected as the source of many indoor air health and odor complaints (Pleil 1990). It can emit a variety of organic compounds into the indoor environment because its manufacture generally involves many synthetic chemicals and treatments, such as adhesives, dyes, solvents, bonding materials, fire retardants, and fungicides. Storage in plastic wrapping following manufacture also minimizes any outgassing which could occur between manufacturing and installation. When the carpeting is installed, the volatile and semi-volatile chemicals are released into the indoor environment as the large surface area of the carpet becomes exposed to the indoor environment.

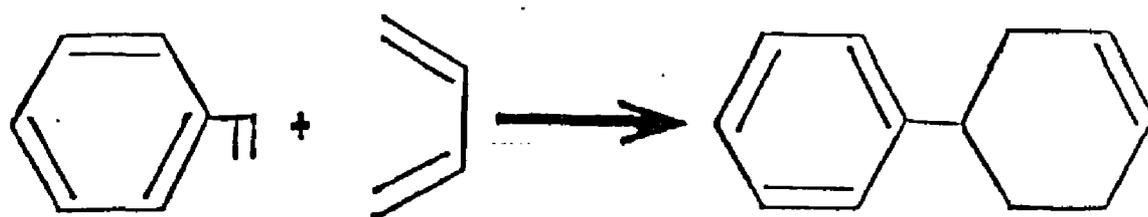
In 1984, Crabb identified a unique contaminant common in three different carpet samples taken from environments in

which people were experiencing symptoms of the "sick-building syndrome". This contaminant was identified as 4-phenylcyclohexene (Crabb, 1984). 4-PCH is a colorless liquid with a vapor pressure of 0.096 mm Hg at 25 °C, and a boiling point of 235 °C. It has a molecular weight of 158.3 and a specific gravity of 0.99 (Chakrabarti 1989). Miksch, Hollowell and Schmidt had previously identified 3-phenylcyclohexene in indoor air samples associated with new carpeting (Miksch 1982). A problem with nomenclature probably led to 4-PCH being originally called 3-PCH (Walsh 1986).

Crabb traced the source of the 4-PCH to the latex which was used as a back-coating adhesive in the carpet manufacturing process. 4-phenylcyclohexene (4-PCH) is produced as a by-product in the production of styrene-butadiene rubber (SBR) latex, when styrene and 1,3 butadiene are polymerized by a Diels-Alder type reaction (Crabb, 1984). (See Figure 1).

Walsh later demonstrated 4-PCH in each environment in which new carpet had been recently installed. Due to its commercial unavailability, the 4-PCH used by Walsh was synthesized by a dehydration reaction using 4-phenylcyclohexanol (4PCOH) as a starting material (Walsh 1986).

4-phenylcyclohexene is a suspect cause of certain



Styrene

1,3-Butadiene

4-PCH

Figure 1. Reaction of styrene and 1,3 butadiene to form 4-phenylcyclohexene.

"tight building syndrome" related illnesses such as headache, fatigue, and upper respiratory and eye irritation. Due to the possibility of health effects being induced by 4-PCH, as well as its odiferous properties, it was of interest to examine the effects of various cure parameters including cure time, cure temperature, and air circulation on its release from freshly prepared tufted carpet backed with carboxylated SBR latex. In the event that 4-PCH could contribute to the onset of health problems, preliminary efforts were undertaken by Demer to investigate the factors which could influence its release from latex samples.

Analysis of 4-Phenylcyclohexene Content in Latex

4-PCH analysis by direct injection on the gas chromatograph (GC) was developed originally by Crabb (1984), and modified by Walsh (1986) and Demer (1989). Demer developed a convenient method for directly extracting 4-PCH from dried and cured latex samples, analyzing the extracted 4-PCH using gas chromatography, and correcting for the extraction efficiency.

The extraction efficiency using carbon disulfide was verified by Soxhlet extraction of dried latex samples followed by direct GC injection. Carbon disulfide was selected as the extraction solvent due to its ability to penetrate cured latex, its minimal flame ionization

detector (FID) response, and 4-PCH's solubility in this solvent (Demer 1989).

Ultimately Demer validated an efficient method requiring a single carbon disulfide extraction of a dried or cured sample of latex, followed by injection of this extract directly onto the GC for quantitation. The measured amount of 4-PCH was then corrected for the extraction efficiency (determined using Soxhlet extraction tests) to give an absolute 4-PCH concentration in the latex. The extraction efficiency was found to be concentration-dependent, with efficiency declining at high 4-PCH-to-solvent ratios. The extraction efficiency was constant only at 4-PCH concentrations in the 7 to 32 nm/ml range, which was the only range at which Demer considered results acceptable. All latex samples were diluted when the measured 4-PCH content exceeded this range.

1-phenylcyclohexene was employed by Demer (1989) as an internal standard in all analyses, providing a constant reference concentration against which all 4-phenylcyclohexene data could be compared to determine the 4-PCH concentration.

Effect of Cure Parameters on Release of 4-PCH from Raw Latex

Demer selected two latexes, one containing a high 4-PCH content, and one a low 4-PCH content. Various cure

parameters of time, temperature, and oven air recirculation were selected for study. Three temperatures (225, 250 and 275 °F) representative of those used in industry, four cure times (5, 10, 15 and 20 minutes), and three air recirculation patterns (0%, 50% and 100% recirculation) were the parameters examined.

The results showed that various combinations of these cure parameters could produce decreases in 4-PCH concentration ranging from 8% to 96%, with temperature being by far the most important factor. Time was of lesser importance, and air recirculation showed the least influence. It was determined that the percent 4-PCH outgassed was independent of the starting concentration. Demer also noted that the latex samples with the strongest odor of "new carpet" contained the highest amounts of 4-PCH.

Demer (1989) concluded that the oven temperature must be high enough to allow the 4-PCH to diffuse through the latex and reach the surface where it can be eliminated into the oven air stream. However, excessive carpet heating can cause scorching and bubbling of the latex. The carpet industry typically employs oven temperatures resulting in an actual carpet temperature of approximately 215 °F. It is possible that industry-accepted cure parameters may be inadequate to remove any substantial amount of 4-PCH from the carpet. Since Demer found the best results at oven

temperatures in the 275 °F range, he recommended that the carpet industry look at the feasibility of curing at higher temperatures, possibly nearing 275 °F.

Demer's studies, however, only examined the effect of cure parameters on 4-PCH release from non-compounded latex samples. Such samples were not representative of carpet backed with carboxylated SBR latex.

Two other methods had been previously employed by the latex and carpet industry to evaluate 4-PCH concentrations in raw SBR latex. Headspace gas chromatography with standard addition was described by Ettre et al (1983). The second method involved methanol coagulation followed by direct gas chromatograph analysis (SBLMC). Both methods were considered to be non-applicable to the measurement of 4-PCH in cured SBR latex. Headspace gas chromatography for example, requires the use of an expensive headspace analyzer; methanol coagulation can only be performed on liquid, not cured latex samples.

RESEARCH OBJECTIVE

Previous research on 4-phenylcyclohexene has focused on determination of 4-PCH concentrations in raw latex samples and the effects of various cure parameters on 4-PCH release from those samples. To date, no research has been conducted on the effect of cure parameters on the release of 4-PCH from actual tufted carpeting backed with carboxylated SBR latex.

The objective of this research was threefold: 1) to develop a method for making tufted carpet samples uniformly coated with carboxylated SBR latex, 2) to develop a method to quantitate 4-PCH levels in cured carpet samples, and 3) to evaluate the effect of specific cure parameters (time, temperature and air circulation) on 4-PCH release. These laboratory studies, although not representative of actual manufacturing conditions, were designed to provide insights on the influence of various cure parameters on typical carpet samples.

METHODS AND MATERIALS

Analytical Equipment

All standards and carpet samples containing 4-PCH were analyzed using a Hewlett-Packard, model 5890A gas chromatograph, with a flame ionization detector (FID). A ten meter, fused silica series 530 micrometer capillary column with an HP-17 (50% phenyl, 50% methyl silicone) liquid phase was used for the separation of 4-PCH. The chromatograph was operated with a nitrogen carrier gas flow of 17 ml/min., and a gas flow through the FID of 200 ml/min. for air and 50 ml/min. for hydrogen. The detector temperature was maintained at 250 °C; the injector temperature was maintained at 190 °C.

Preparation of Standards

Internal Standard Solution A carbon disulfide/internal standard solution was initially prepared by adding 7.5 ul of 1-phenylcyclohexene (Aldrich Chemical Co., >97% pure, lot #123077) to 500 ml of carbon disulfide (Fisher Chemical, lot #88029). This produced a 1-PCH concentration of 94.13 nm/ml. This solution was used to extract or desorb the samples, and to make up the working standard solutions for the 4-PCH standard curve.

Stock Standard Solution The stock standard solution was prepared by adding 4 ul of 4-PCH (synthesized by the

University of Arizona Chemistry Department, 2-17-86, and found to be >99% pure by GC/MS) to 4 ml of carbon disulfide. This stock solution contained 6351.11 nm/ml of 4-PCH. It was stored in a teflon-capped borosilicate vial and used to prepare the working standard solutions.

Working Standard Solutions Working standard solutions were prepared by adding varying amounts of the stock standard solution (1.00 - 8.00 ul) to vials containing from 2.00 to 10.00 ml of the carbon disulfide/1-PCH internal standard solution. This produced standards ranging from 0.64 to 43.00 nm/ml of 4-PCH. (See Table 1).

Preliminary Studies

Prior to studies designed to evaluate the effect of various cure parameters on the release of 4-PCH from carpet backed with SBR latex, preliminary studies were initiated to:

- * Determine optimum GC operating parameters for the analysis of 4-PCH
- * Determine optimum extraction time and volume
- * Determine moisture content of raw latex
- * Evaluate oven performance (temperature variation) during simulated cure cycles

TABLE I
 REPRESENTATIVE CONCENTRATION OF STANDARD SOLUTIONS

| <u>uliters Stock</u> <u>4-PCH</u> | <u>mls CS₂</u> | <u>nmoles/ml</u> | <u>Peak Area</u> |
|--------------------------------------|---------------------------|------------------|------------------|
| 1.00 | 10.00 | 0.64 | 1197 |
| 1.00 | 8.00 | 0.79 | 1503 |
| 1.00 | 4.00 | 1.59 | 3051 |
| 1.00 | 2.00 | 3.15 | 6233 |
| 2.00 | 2.00 | 6.35 | 11687 |
| 4.00 | 2.00 | 16.80 | 31697 |
| 8.00 | 2.00 | 43.00 | 80456 |

Six latex samples were initially provided by latex manufacturers. Latexes 1 through 4 were supplied in small quantities for the preliminary studies, while two latex samples, designated Latex A and Latex B, were supplied in large quantity for the remainder of the research. Latexes A and B were composite samples of latexes considered representative of those used by the carpet industry. One contained a higher concentration of 4-PCH while the other contained a lower level.

Determination of Optimum GC Operating Parameters

Initial experiments utilizing four different latexes were

designed to determine optimum GC oven temperature profiles for establishing desirable 4-PCH/1-PCH separation and peak characteristics. Oven temperatures of 140 °C and 150 °C, and peak widths of 0.16 and 0.32 minutes were compared.

Determination of Moisture Content in Raw Latex The four latexes used for the preliminary studies were analyzed for moisture and solids content by weighing small volumes of liquid latex before and after drying. A volume of approximately 0.1 ml of raw liquid latex was placed on indented pieces of aluminum foil, weight recorded, and placed in a desiccator. After 24 hours, the latex and foil samples were reweighed and the percent weight loss determined.

4-PCH Desorption Studies All latex and carpet/latex samples were analyzed using Demer's single extraction method. Extraction times and carbon disulfide desorption volumes were optimized with respect to 4-PCH content. Extraction times of 10, 20, 30, 40, 50 and 60 minutes were examined, as well as extraction volumes of two, four, five and ten ml of carbon disulfide/internal standard solution.

Cure Oven Parameters Micronta 22-191 digital thermocouples were employed in duplicate to measure the time required for carpet samples to reach oven temperature. The thermocouples were also employed to examine uniformity of temperature within the oven, as well as temperature changes

during the introduction and removal of samples.

Data Analysis The JCL6000 Chromatography Data System computer software package by Jones Chromatography LTD was interfaced with a Hewlett-Packard gas chromatograph and an Epson FX-850 printer to collect and analyze the data. Only a few of the preliminary experiments utilized a separate Hewlett-Packard Model 3390A integrator-type recorder. The computer software package saved all data and was employed to produce a hard copy of each chromatogram and its integrated area for comparison with standards. The area data from each of the peaks of interest was then analyzed using the LOTUS 1-2-3 program.

Preparation of Carpet Samples

A Standard Operating Procedure for the preparation of carboxylated latex-backed, tufted carpeting, representative of that produced by the carpet industry, was developed for this study following the viewing of a videotape in which tufted carpeting was prepared in a quality control research laboratory in Dalton, Georgia concerned with establishing standards for the carpet industry. (See Appendix A). All materials required to prepare the carpeting were obtained from the carpet industry and are representative of materials used throughout the industry. Latexes A and B represent composite latexes prepared from samples submitted by several

latex manufacturers, and were exclusively used in all curing studies.

Carboxylated SBR latex carpet swatches (10" square) were prepared according to a standard method established by the carpet industry. The carboxylated latex mixture was prepared by adding 20 ml of double distilled water (26 ml if using Latex B) to 187 grams latex (185 grams if using Latex B) and adding 450 grams calcium carbonate filler. 5.71 grams of froth aid and 9.3 grams of thickener (5.8 grams if using Latex B) were then added and mixed as specified. The viscosity was checked; if correct, the mixture was whipped at high speed for the appropriate time (one to two minutes) until frothed. The frothed latex mixture was checked for proper weight (approximately 800 grams per quart), and again for viscosity.

The properly frothed latex was carefully applied across the top of the backing of a 10 inch square piece of tufted carpet sample secured to a wood stretcher frame. A 12 inch by 1/2 inch diameter steel cylinder was carefully rolled to assist in applying the frothed latex evenly across the back of the carpet sample. The secondary carpet backing, fuzzy side down, was placed on top of the frothed latex, and pressed into place by rolling another larger and heavier (14 inch by 2 inch diameter) cylinder across the surface of the backing. The carpet sample was then cut into pieces and

cured.

A representative sample of the carpet produced during the early stages of this research project was sent to the carpet industry in Dalton, Georgia for analysis and found to be similar to carpet samples produced in their own laboratory and therefore, satisfactory for use in curing studies.

Analytical Procedure for Determination of 4-PCH Content in Latex Backed Carpet

Once tufted carpeting could be routinely and reproducibly prepared, a method was developed to extract and analyze the 4-PCH content of tufted SBR latex carpeting. Demer's method of analyzing dried latex buttons for 4-PCH content was adapted to cured carpet samples. The volume of carbon disulfide extractant was increased to approximately 5 ml to compensate for the sample size (1/2" squares) and absorption by the carpet tuft.

Preparation of Carpet Samples for Cure Each experiment to evaluate the impact of various cure parameters required a freshly prepared latex-coated, 10 inch square swatch of carpet. These carpet swatches were then cut into the required five pieces for evaluation of specific cure parameters such as 4-PCH loss at specified cure times. (See Figure 2).

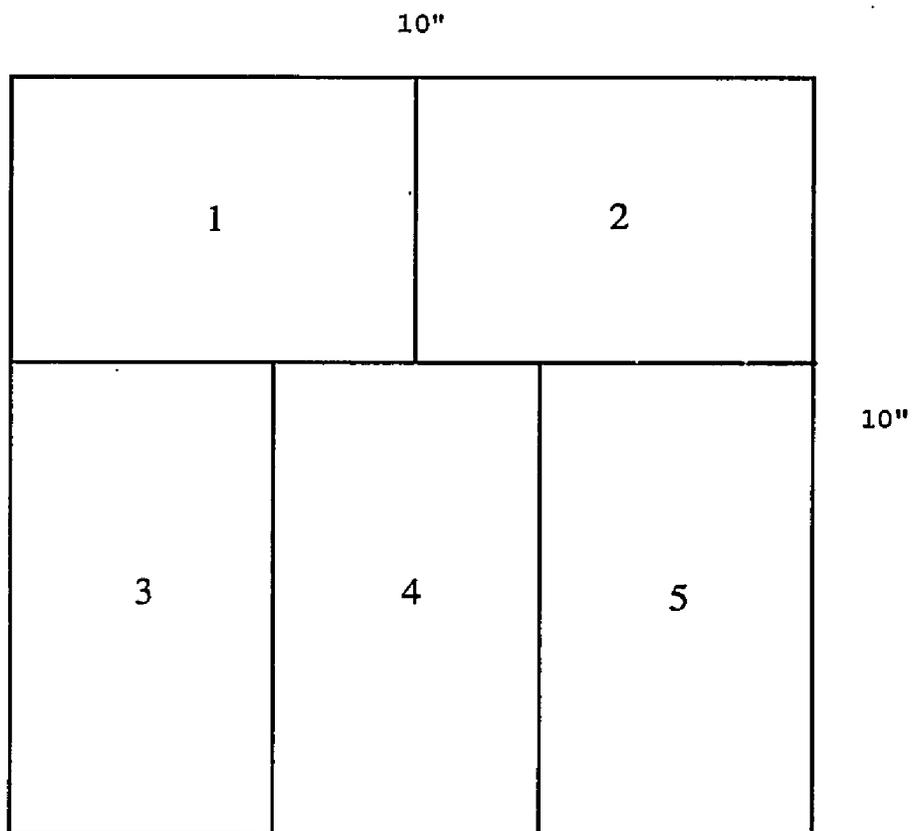


Figure 2. Division of 10" x 10" Carpet Swatch into Cure Samples.

A one-half inch square corner piece was removed from each carpet swatch prior to curing to serve as a reference for "total" 4-PCH content. These uncured sections were individually weighed and placed into separate, sealed vials for drying and subsequent 4-PCH analysis. The remaining carpet pieces were then placed into the oven and subjected to a specific cure parameter. Pieces 1 and 2 were placed on the top rack of the oven, with piece 2 in the back, and pieces 3, 4, and 5 placed on the bottom rack of the oven. Piece 5 was placed in the back, piece 4 in the middle, and piece 3 in front. This sequence was repeated during each cure study to ensure consistency in approach.

After exactly 5 minutes of cure, piece 1 was removed from the oven. Pieces 2, 3, 4 and 5 were removed after 10, 20, 40 and 60 minutes of cure time, respectively. Each piece of carpeting was weighed when cooled to room temperature. Six (6) half-inch squares were cut from each cured piece of carpet, according to the predetermined pattern to ensure consistency in latex application. (See Figure 3). These half-inch squares were weighed and placed into separate, sealed glass reaction vials for desorption and later 4-PCH analysis.

Desorption Procedure The cured and uncured carpet specimens (seven in all) from each piece of carpet were analyzed for 4-PCH content. Five ml of carbon

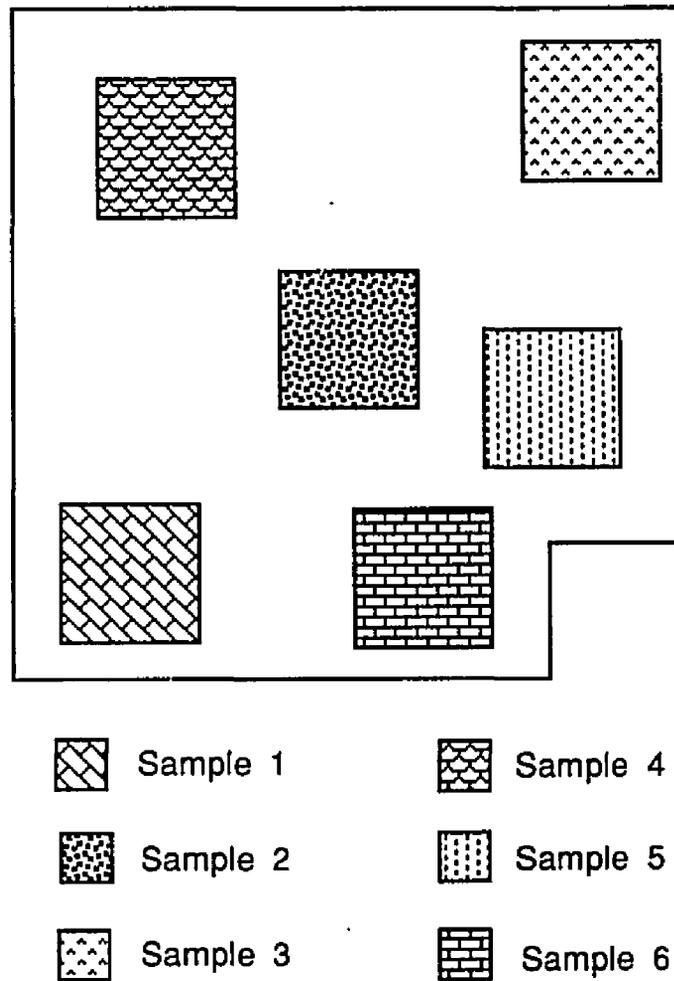


Figure 3. Pattern for the Selection of Individual Cured Carpet Samples for 4-PCH Analysis.

disulfide/internal standard solution was added to each vial. Caps with septa were securely placed onto the reaction vessels, and the vessels agitated vigorously to thoroughly saturate the carpet samples with the solution. The vessels were vigorously agitated for several seconds every five minutes throughout the 25 minute desorption period.

GC Analysis and Quantitation When the desorption period was completed, one microliter of the carbon disulfide extraction solution was carefully drawn up into a five microliter Hamilton syringe (Model 87920, #95) and injected onto the gas chromatograph. By comparing 1-PCH/4-PCH count ratios with those of the standard curve (and adjusting for the volume of the carbon disulfide solution added), the amount of 4-PCH (w/w) in the unknown carpet sample was determined.

The amount of 4-PCH present in cured samples was compared to the amount of 4-PCH present in the uncured sample to evaluate the efficacy of a specific cure parameter, since it was not possible to prepare carpet swatches with a uniform thickness of latex each time under laboratory conditions. Therefore, results were reported as percent 4-PCH remaining.

The uncured samples differed from the cured samples in that much more moisture was present in the freshly prepared uncured carpet samples than in the cured samples. The

weight of the uncured samples had to be adjusted accordingly to reflect this difference. (See Primary Experimental Results - Determination of Moisture Content in Freshly Prepared Uncured Carpet Samples).

Standard Curve Data A standard curve of at least three points was run each time an experiment was performed. New standard solutions were prepared approximately every 60 days, when the old standard solutions deteriorated (due to evaporation of the carbon disulfide) as evidenced by variations in the calibration curve.

It was the original intention of this research project to employ an internal standard so that small variations in injection volume would not affect the results. It was soon apparent, however, that when samples of cured carpeting were desorbed and analyzed, other components present in the carpeting possessed retention times similar to those of the 1-PCH internal standard. As a result, the 4-PCH content in the desorbed carpet samples was compared directly with the 4-PCH working standards to determine the quantity of 4-PCH in the unknown (carpet) samples.

Air Change Parameters Unless otherwise specified, all curing studies were performed in an oven with three air changes per minute.

Primary Experimental Protocol

Determination of Moisture Content in Freshly Prepared Uncured Carpet Samples In virtually every experiment utilizing freshly prepared tufted carpeting, carpet samples were weighed both before and after cure cycles to permit the determination of carpet moisture content.

These data were used to correct the weight of the freshly prepared uncured carpet samples so that the amount of 4-PCH found in these samples could be compared to the amount of 4-PCH found in the cured samples. Without correcting for the moisture content of the uncured carpet samples, it would have been impossible to accurately determine the percent reduction in 4-PCH content following cure.

Variability of 4-PCH Content within a Carpet Swatch

The variability of 4-PCH concentration within a swatch of carpeting was examined. Carpeting prepared with Latex A was subjected to cure times of 10, 20, 40 and 60 minutes at 275 °F. The carpet swatches cured for 10 to 60 minute periods were each divided into eight sample pieces and analyzed to determine if the piece varied significantly in 4-PCH content.

Determination of Percent 4-PCH Remaining Following 225, 250 and 275 °F Cure Carpet samples prepared with either Latex A and Latex B were subjected to three cure

temperatures (225, 250 and 275 °F) and analyzed for 4-PCH content. These experiments were repeated except in the case of the 250 °F test run. In each experiment, the carpet swatches were subjected to a specific cure temperature for periods of 5, 10, 20, 40 or 60 minutes. Six samples from each swatch (Figure 3) were analyzed, in addition to the uncured sample, to determine the effect of various cure times and temperatures on the decay of 4-PCH from SBR backed carpet.

Temperature Rise During Curing Process Two carpet samples prepared with either Latex A or B were monitored by thermocouples to determine the length of time required for freshly prepared carpet swatches to reach an oven temperature of 225 °F when placed in a preheated oven. Two Latex B carpet swatches were also evaluated in duplicate at 250 and 275 °F.

Measurement of Moisture Content Over Time Since raw latex samples A and B both contained identical amounts of water, specific experiments examining moisture loss over time were performed only on Latex B. Moisture loss was determined by measuring the weight of carpet swatches before and after cure periods of 1, 2, 4, 6, 10, 15 and 20 minutes at 225, 250 and 275 °F.

Effect of Various Air Circulations on Percent 4-PCH Remaining Experiments varying the air circulation were

conducted at 225 °F, since this temperature is more representative of temperatures used in industry. Duplicate swatches of carpet backed with carboxylated SBR latex A were cured for 5, 10, 20, 40 and 60 minutes at 225 °F, with no air circulation. Duplicate carpet swatches prepared with Latex A were then cured at 225 °F with 1500 linear feet per minute air flow and 10 air changes per minute for 5, 10 and 20 minutes. The experiment was repeated at 225 °F with the fan in place in the oven, but not operating.

RESULTS

Results of Preliminary Studies

Determination of Optimum GC Operating Parameters The best separation of 1-PCH/4-PCH mixtures was achieved using a GC oven temperature of 140 °F and a peak width setting of 0.16 minutes. Representative chromatograms of both the integrator-type recorder and the JCL6000 software package can be seen in Figures 4, 5 and 6.

The data obtained from the chromatograms were analyzed utilizing the Lotus 1-2-3 program. An example of this analysis can be seen in Figure 7.

Estimates of the 4-PCH content of carpet samples were made by reference to 4-PCH/1-PCH standard curves. A typical curve demonstrating the linearity of such curves is presented in Figure 8.

Determination of Moisture Content in Raw Latex The moisture content of four representative latexes made by measuring latex sample weights before and after sample drying is presented in Table II.

Very little variation was observed in the solids content of the four representative latexes, with the average being 53.90% \pm 1.56%, or approximately 45% moisture.

4-PCH Desorption Studies The optimum extraction time required to ensure desorption of all possible 4-PCH from carpet swatches was determined for all four latexes. The

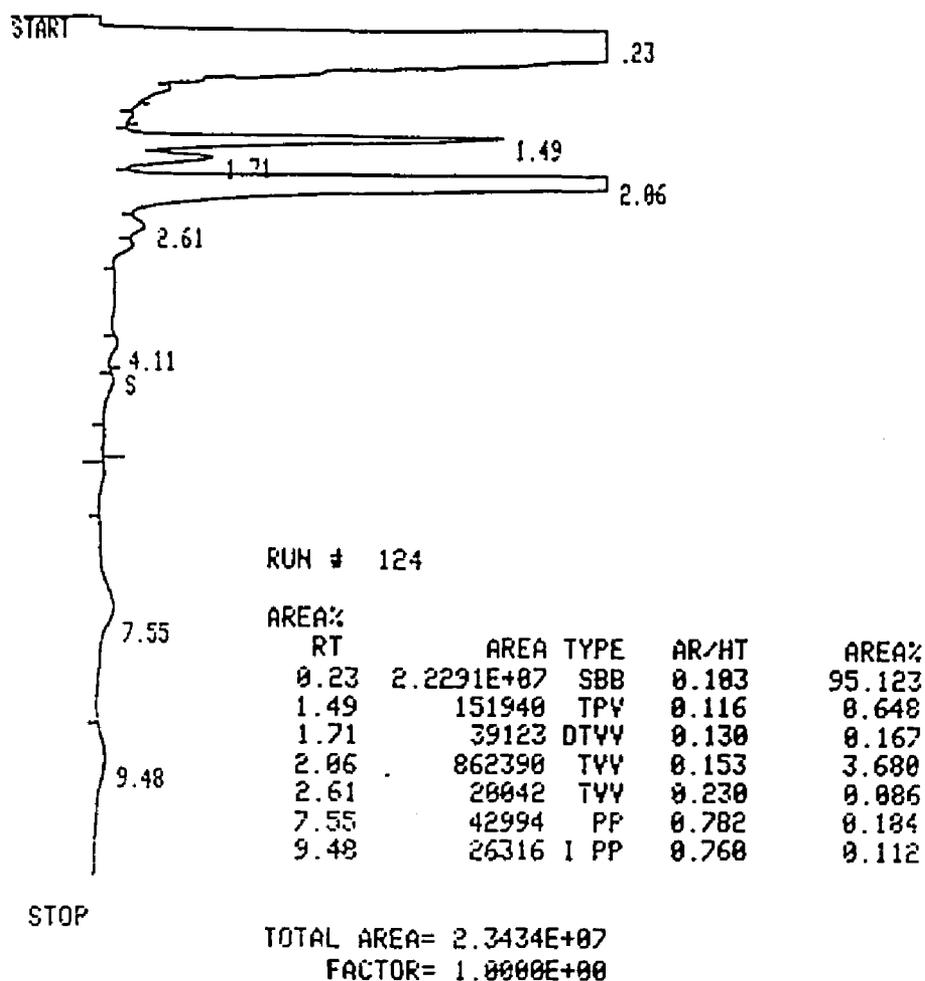


Figure 4. A Representative Chromatogram Utilizing the Hewlett-Packard Model 5890A Gas Chromatograph with Integrator-Type Recorder.

JCL6000 CHROMATOGRAPHY DATA SYSTEM

Date : 7/8/1989 Time : 12: 4:40 Date Acquired: 7/8/1989 Time Acquired: 11: 53:58
Channel : 1 Sample Type : Std Data File : C:\DATA\M4 Method File : C:\METH\usfid
Run Number 4 of 6 Sample Name : 6350 Chan1 Desc. : fid Chan2 Desc. :
Comment :

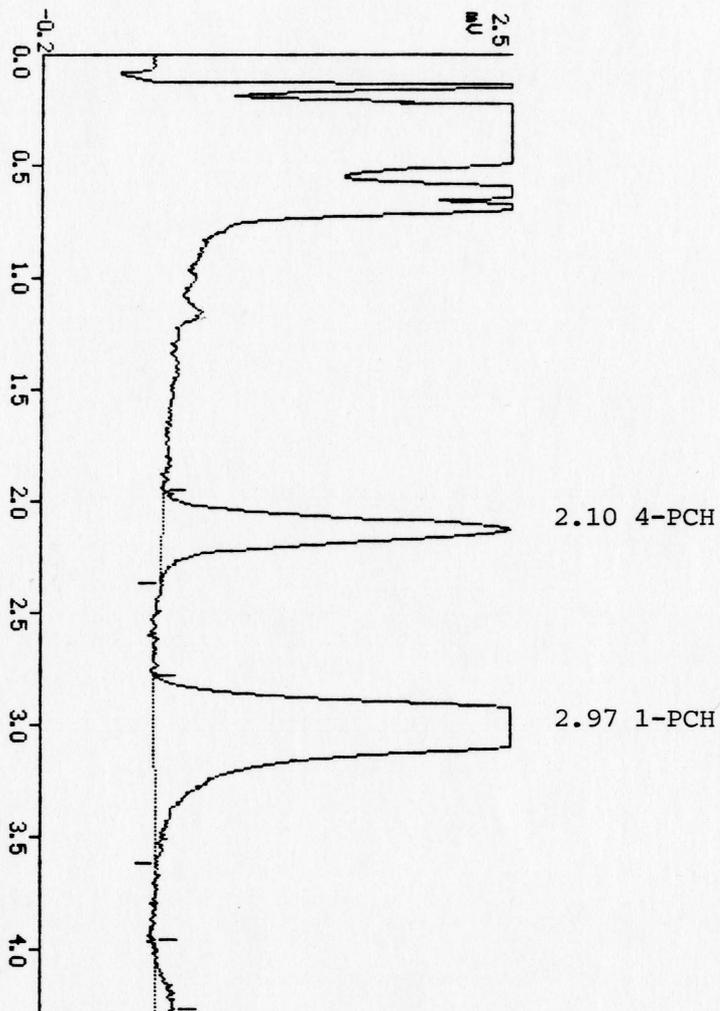


Figure 5. A Representative Chromatogram Utilizing the Hewlett-Packard Model 5890A Gas Chromatograph with the JCL6000 Software Package.

JCL6000 CHROMATOGRAPHY DATA SYSTEM

Date : 7/8/1989 Time : 12: 7:9 Date Acquired: 7/8/1989 Time Acquired: 11: 53:58
 Channel : 1 Sample Type : Std Data File : C:\DATA\NA Method File : C:\METH\msfstd
 Run Number 4 of 6 Sample Name : 6350 Chan1 Desc. : fid Chan2 Desc. :
 Comment :

Integration Results for Channel 1

| Pk # | Ret. Time | Height | Height % | Area | Area % | Modified Response | Response Ratio | Soln. Conc. (ngul/) | Quantify | Derived Result | Component Name |
|------|-----------|--------|----------|--------|--------|-------------------|----------------|---------------------|----------|----------------|----------------|
| 1 | 2.100 | 834 | 27.67 | 13083 | 20.73 | 13083 | 463.10 | 6.059 | Yes | 6.059 | 4-PCH |
| 2 | 2.975 | 2132 | 70.75 | 48802 | 77.33 | 48802 | 463.10 | 22.600 | Int | 22.600 | 1-PCH |
| 3 | 4.225 | 48 | 1.58 | 1220 | 1.93 | 1220 | 0.00 | N/A | | 0.000 | UNKNOWN |
| | | 100.00 | | 100.00 | | | | | | | |

Total Height : 3013
 Total Area : 63105

Area used for calculations.

Figure 6. A Representative Peak Area Data Sheet Produced by the Hewlett-Packard Model 5890A Gas Chromatograph and the JCL6000 Software Package.

| Name: MSF | | | | | | | time Aver. pps STD | | |
|-------------------|-------|-------------|-----------------|------------|---------|----------|--------------------|--|--|
| Latex: B | | | | | | | | | |
| Date: B-B-89 | | | | | | | | | |
| Title: 135 C CURE | | | | | | | | | |
| Expt #: | 1 | GC 140 | INJ 190 | DET 250 | | | | | |
| Standard Curve | UL | | Response factor | | 0 | 24.17 | 1.36 | | |
| | 4-PCH | 1-PCH ratio | INJ | 4-PCH area | 5 | 22.81 | 1.78 | | |
| peoles/ul | | | ul | | 10 | 12.26 | 0.84 | | |
| | | | | | 20 | 3.66 | 0.65 | | |
| | | | | | 40 | 0.00 | 0.00 | | |
| 640 | 1241 | 48742 | 0.025 | 0.95 | 1306.3 | 0.489927 | 0.510068 | | |
| 1590 | 2909 | 48052 | 0.061 | 0.95 | 3062.1 | 0.519250 | | | |
| 3150 | 6256 | 47830 | 0.131 | 1.00 | 6256.0 | 0.503516 | | | |
| 6350 | 12550 | 47259 | 0.266 | 1.00 | 12550.0 | 0.505976 | | | |
| 16800 | 34276 | 51873 | 0.661 | 1.05 | 32643.8 | 0.514645 | | | |
| 43000 | 81579 | 48875 | 1.669 | 1.00 | 81579.0 | 0.527096 | | | |

Response factor
as picomoles/area

| Time | UL | AREA | INJ. | RF | Total | wt of sample | nmols/ppm 4-pch Uncured | | |
|------|----|-------|------|--------|-------------|--------------|-------------------------|-----------|-------|
| | | | | | | | ML5 CS2 | Picomoles | 4-PCH |
| 0 | M1 | 10904 | 1.05 | 0.5101 | 10 52969.43 | 354.9 | 149.25 | 23.58 | 23.58 |
| 5 | M2 | 17719 | 1.00 | 0.5101 | 5 45189.54 | 350.6 | 128.89 | 20.36 | 25.72 |
| 5 | M3 | 22851 | 0.95 | 0.5101 | 5 61345.17 | 404.5 | 151.66 | 23.96 | 23.09 |
| 5 | M4 | 22518 | 1.00 | 0.5101 | 5 57428.65 | 390.7 | 146.99 | 23.22 | 25.87 |
| 5 | M5 | 18148 | 1.00 | 0.5101 | 5 46283.64 | 315.8 | 146.56 | 23.16 | 22.61 |
| 5 | M6 | 26115 | 1.00 | 0.5101 | 5 66602.23 | 413.4 | 161.11 | 25.46 | |
| 5 | M7 | 21731 | 1.00 | 0.5101 | 5 55421.52 | 422.7 | 131.11 | 20.72 | |
| 0 | M1 | 11013 | 1.00 | 0.5101 | 10 56173.88 | 345.1 | 162.78 | 25.72 | |
| 10 | M2 | 12631 | 1.00 | 0.5101 | 5 32213.39 | 409.3 | 78.70 | 12.44 | |
| 10 | M3 | 13440 | 1.00 | 0.5101 | 5 34276.62 | 395.9 | 86.58 | 13.68 | |
| 10 | M4 | 12017 | 1.00 | 0.5101 | 5 30647.48 | 391.9 | 78.20 | 12.36 | |
| 10 | M5 | 11461 | 1.00 | 0.5101 | 5 29229.49 | 404.1 | 72.33 | 11.43 | |
| 10 | M6 | 11025 | 1.00 | 0.5101 | 5 28117.54 | 390.4 | 72.02 | 11.38 | |
| 10 | M7 | 13863 | 1.00 | 0.5101 | 5 35355.42 | 324.6 | 108.92 | 17.21 | |
| 0 | O1 | 11170 | 1.00 | 0.5101 | 10 56974.68 | 389.8 | 146.16 | 23.09 | |
| 20 | O2 | 3239 | 1.00 | 0.5101 | 5 8260.564 | 453.1 | 18.23 | 2.88 | |
| 20 | O3 | 3157 | 1.00 | 0.5101 | 5 8051.436 | 435.5 | 18.49 | 2.92 | |
| 20 | O4 | 4117 | 1.00 | 0.5101 | 5 10499.76 | 424.1 | 24.76 | 3.91 | |
| 20 | O5 | 4910 | 1.00 | 0.5101 | 5 12522.19 | 480.9 | 26.04 | 4.11 | |
| 20 | O6 | 5157 | 1.00 | 0.5101 | 5 13152.12 | 463.1 | 28.40 | 4.49 | |
| 20 | O7 | 6977 | 1.00 | 0.5101 | 5 17793.75 | 485.5 | 36.65 | 5.79 | |

Figure 7. A Representative Analysis of Data Utilizing the Lotus 1-2-3 Program.

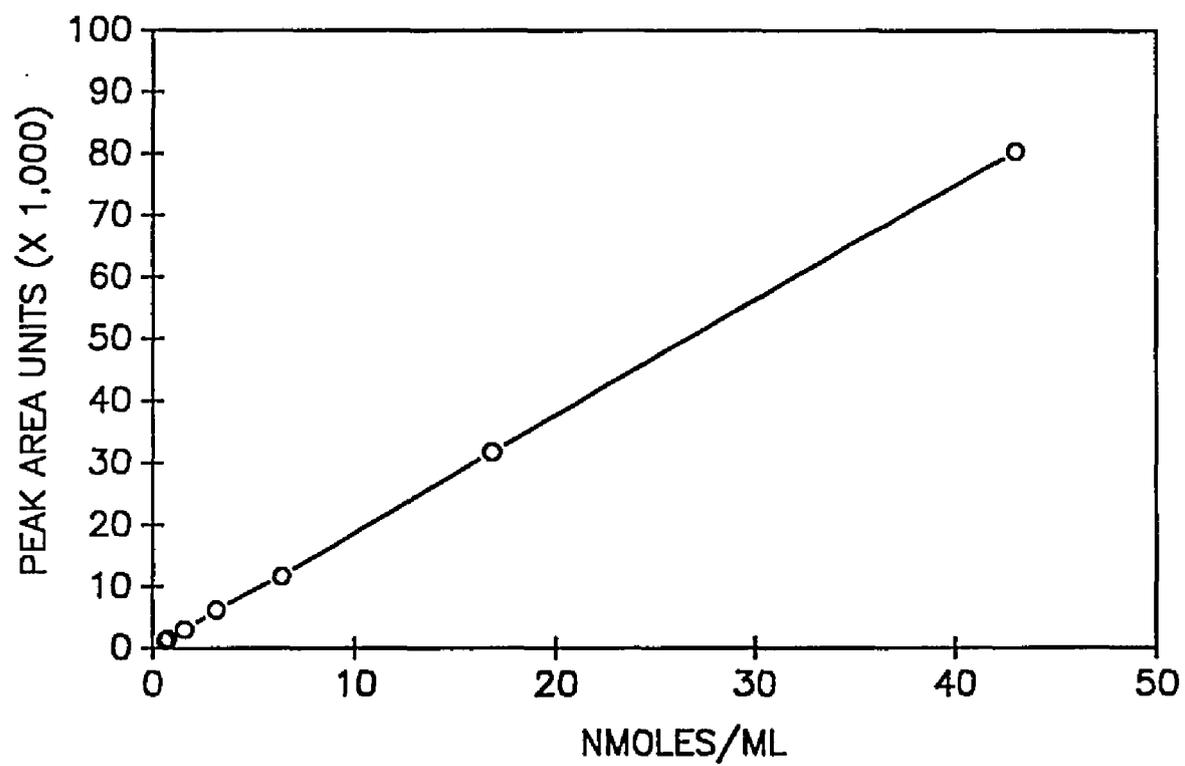


Figure 8. A Representative 4-PCH Standard Curve.

TABLE II.

AVERAGE PERCENT SOLIDS PRESENT IN FOUR LATEXES

| <u>LATEX</u> | <u>PERCENT SOLIDS</u> | <u>PERCENT MOISTURE</u> |
|----------------------------------|-----------------------|-------------------------|
| 1 | 55.12 ± 1.08 | 44.88 |
| 2 | 51.59 ± 0.67 | 48.41 |
| 3 | 53.36 ± 0.51 | 46.64 |
| 4 | 55.53 ± 0.54 | 44.47 |
| <hr/> $\bar{x} = 53.90 \pm 1.56$ | | $n = 4$ samples, 2 runs |

latex samples were analyzed following extraction with the carbon disulfide/internal standard at times of 10, 20, 30, 40, 50 and 60 minutes to determine when the maximum amount of 4-PCH was extracted. The results of Latex 1, which are representative of the rest of the samples, are listed in Appendix D.

An extraction period of 20 minutes was required to achieve 100% extraction of the 4-PCH from the latex samples studied. As a result, all of the studies that followed were desorbed for 25 minutes (20 minutes as determined through experimentation, with a five minute safety factor).

When analyzing 30 mg buttons of latex containing approximately 1200 nm 4-PCH/gram of latex, two ml of carbon disulfide was necessary to extract the 4-PCH. However, when

analyzing 30 mg buttons of latex containing greater than 1500 nm 4-PCH/gram of latex, four ml of carbon disulfide was required to extract the 4-PCH. Therefore, four ml was the minimum volume of desorbent utilized for raw latex samples.

When working with half-inch squares of prepared cured carpeting, five ml of CS₂ was enough to cover and desorb the 4-PCH. However, when working with the uncured samples, other ingredients present in the carpet made the extract too viscous to be drawn through a syringe for injection onto the GC, so 10 ml was required to desorb the uncured carpet samples.

Cure Oven Parameters Thermocouple measurements were taken throughout the oven to validate its temperature profile. When the oven's temperature meter read 225 °F, thermocouples measured 226 °F in the front of the oven, and 225 °F in the back of the oven. When the oven door was momentarily opened and closed, the temperature at various locations within the oven did not change.

Thermocouples were inserted into thoroughly dried carpet samples to determine the time required for the samples to reach oven temperature. These were measured simultaneously utilizing two thermocouples, and the results are shown in Table III. These data demonstrate the ease with which the temperature of dried carpet samples was raised from 89 to 225 and 250 °F, as compared to the time

TABLE III.
 TIME REQUIRED FOR CARPET SAMPLES
 TO REACH SPECIFIED OVEN TEMPERATURE

| <u>Temperature (°F)</u> | | <u>Length of Time</u> |
|-------------------------|-------|-----------------------|
| Initial | Final | Minutes |
| 89 | 225 | 5.5 |
| 89 | 250 | 6.5 |
| 89 | 275 | 16.75 |

n = 2 samples, 1 run

required to raise the temperature to 275 °F.

Primary Experimental Results

Determination of Moisture Content in Freshly Prepared
Uncured Carpet Samples

Since the percent reduction in 4-PCH content following specified cure parameters was based on the amount of 4-PCH per milligram of dry carpet, it was first necessary to correct the uncured reference carpet sample for its moisture content. Therefore, the average amount of moisture in a carpet sample was determined by comparing uncured and cured carpet samples. Essentially all carpet samples used in this research project were evaluated for moisture content. Only weights of carpeting cured for at least 20 minutes were compared to the uncured weights, to ensure that all possible evaporation had taken place. The results of this study are shown in Appendix E.

The average percent determined from an analysis of all

carpet samples was $8.60 \pm 0.41\%$. Thus, carpeting prepared during this research contained 91.40 % solids. The 4-PCH content of uncured carpet samples was adjusted by dividing by 0.914.

Variability of 4-PCH Content within Carpet Samples The variability of 4-PCH within freshly prepared pieces of cured carpet was studied to evaluate the uniformity of latex application. Eight standard carpet samples taken from each of four pieces of carpeting prepared with Latex A and cured at 275 °F were analyzed for 4-PCH content. The four pieces of carpeting were each subjected to different cure times (10, 20, 40 and 60 minutes). The results are presented in Appendix F. The average 4-PCH content (ppm) in each is shown in Table IV.

Less than a 13% average variation was found in the amount of 4-PCH present at different locations on a piece of freshly prepared carpeting after a cure time of 10 minutes, which demonstrated a relatively uniform application of latex. Due to the loss of essentially all 4-PCH at extended cure time, percent variation could not be determined at more than 10 minutes cure.

Determination of Percent 4-PCH Remaining Following 225, 250 and 275 °F Cure Carpet pieces coated with Latex A were tested following cure temperatures of 225, 250 or 275 °F, and cure times of 5, 10, 20, 40 and 60 minutes. Six samples

TABLE IV.

**VARIABILITY OF 4-PCH CONTENT IN CARPETING
CURED FOR VARIOUS CURE TIMES**

| <u>Cure Time</u> Minutes | <u>4-PCH Content, ppm</u> | | <u>% Variation</u> |
|-----------------------------|---------------------------|-----------|--------------------|
| | Average | Std. Dev. | |
| 10 | 29.38 | 3.77 | 13 |
| 20 | 3.38 | 0.70 | -- |
| 40 | 0.38 | 0.48 | -- |
| 60 | 0.00 | 0.00 | -- |

n = 8 samples, 1 run

from each piece of test carpet were analyzed for 4-PCH at each cure temperature over specified cure times. Each test run was repeated unless otherwise noted. One uncured sample was analyzed for each duplicate test sample. The results are detailed in Table V and graphed in Figure 9.

Latex B coated carpet pieces were also evaluated in duplicate tests (except where noted otherwise) following cure temperatures of 225, 250 and 275 °F, and cure times of 5, 10, 20, 40 and 60 minutes. Six samples from each carpet test piece were analyzed for 4-PCH content at specified cure times and temperatures. One reference uncured sample were collected from each carpet swatch at each temperature. The test was repeated. The results of these tests are presented in Table VI, and illustrated in Figure 10.

TABLE V
 PERCENT 4-PCH REMAINING IN CARPET SAMPLES
 CURED AT 225, 250 OR 275 °F (LATEX A)

| <u>Cure Time</u> Minutes | <u>Cure Temperature</u> | | |
|-----------------------------|-------------------------|--------------|-------------|
| | 225 °F | 250 °F | 275 °F |
| 0 | 100.0 ± 4.2 | 100.0 ± 4.1* | 100.0 ± 9.5 |
| 5 | 88.2 ± 2.3 | 90.4 ± 3.2* | 82.6 ± 7.9 |
| 10 | 75.6 ± 9.0 | 68.1 ± 5.5* | 37.8 ± 3.3 |
| 20 | 52.2 ± 5.0 | 20.9 ± 1.4* | 15.6 ± 2.4 |
| 40 | 6.6 ± 1.5 | 1.3 ± 0.6* | 0.3 ± 0.3 |
| 60 | 0.1 ± 0.2 | 0.0 ± 0.0* | 0.0 ± 0.0* |

n = 12 samples, two runs

*test not repeated

*n = 6 samples, one run

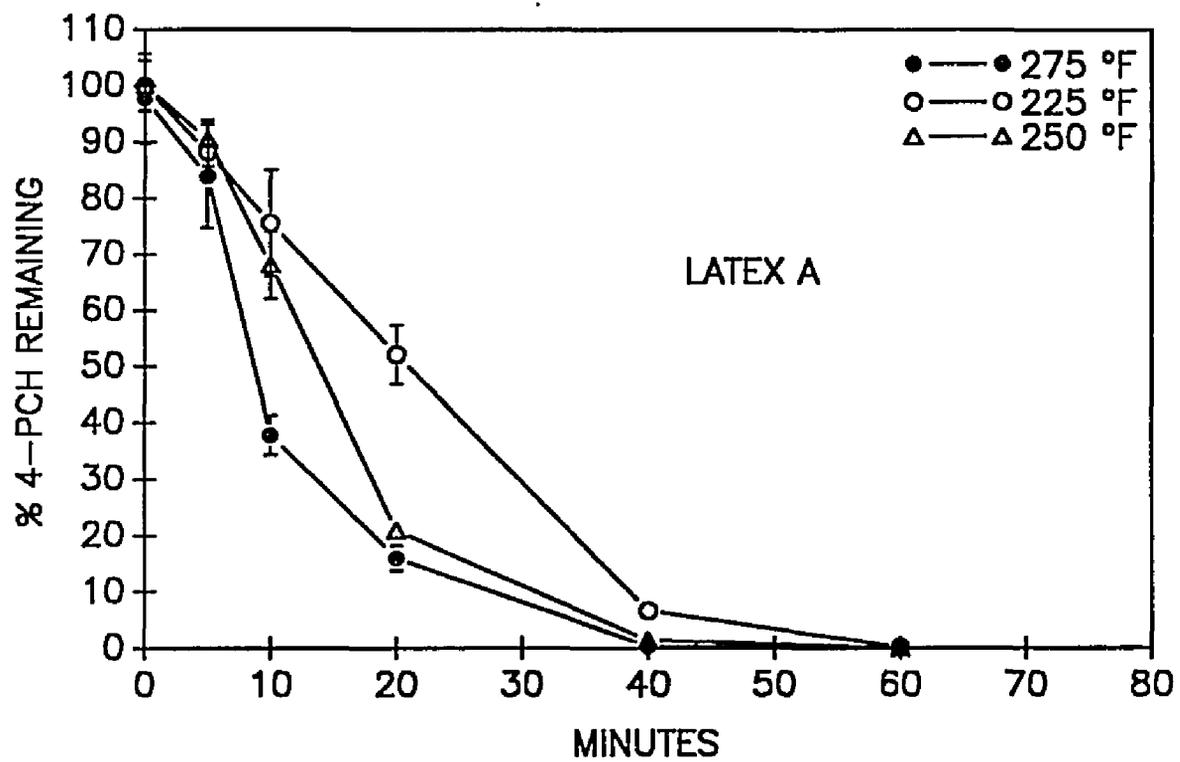


Figure 9. Percent 4-PCH Remaining Following Cure at Temperatures of 225, 250 or 275 °F. (Latex A)

TABLE VI

PERCENT 4-PCH REMAINING IN CARPET SAMPLES
CURED AT 225, 250 OR 275 °F (LATEX B)

| <u>Cure Time</u> Minutes | <u>Cure Temperature</u> | | |
|-----------------------------|-------------------------|--------------|-------------|
| | 225 °F | 250 °F | 275 °F |
| 0 | 100.0 ± 6.4 | 100.0 ± 7.3* | 100.0 ± 5.9 |
| 5 | 87.3 ± 4.5 | 90.5 ± 3.3* | 85.8 ± 6.6 |
| 10 | 70.6 ± 7.9 | 64.3 ± 6.2* | 45.1 ± 4.3 |
| 20 | 53.0 ± 4.4 | 23.2 ± 3.8* | 17.0 ± 2.8 |
| 40 | 12.8 ± 2.7 | 0.9 ± 1.3* | 0.0 ± 0.0 |
| 60 | 0.9 ± 1.0 | 0.0 ± 0.0* | 0.0 ± 0.0 |

*test not repeated

n = 12 samples, two runs
*n = 6 samples, one run

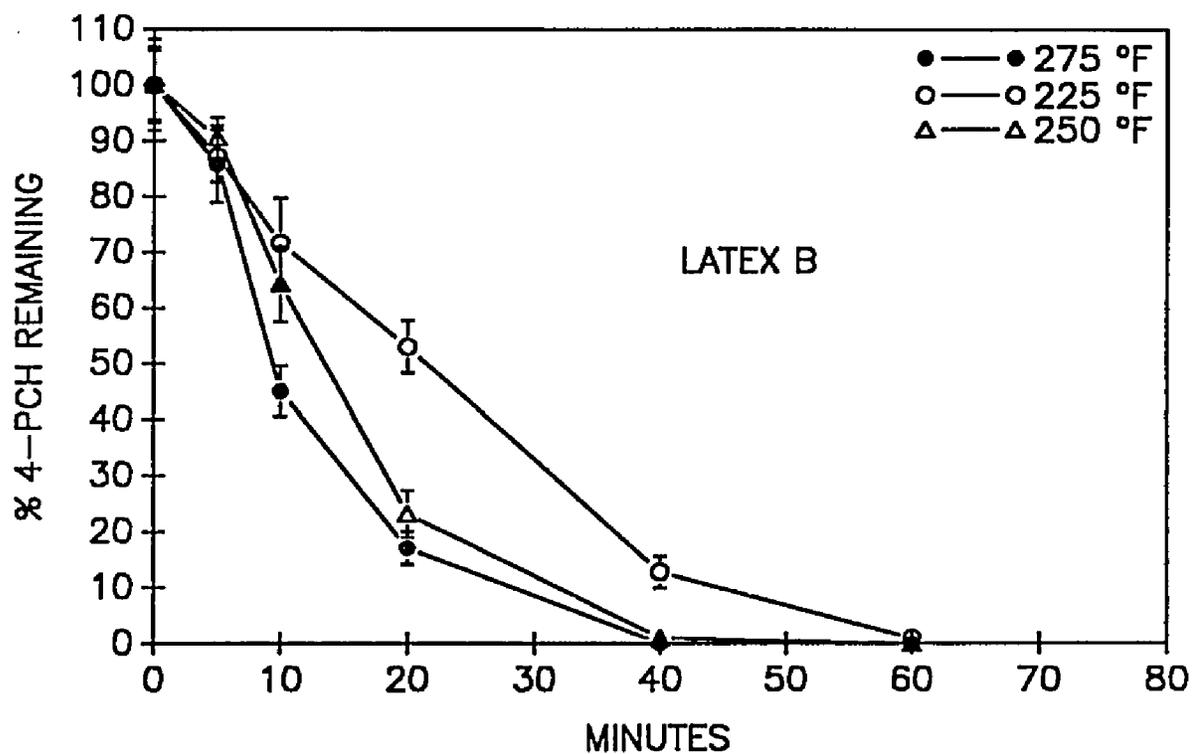


Figure 10. Percent 4-PCH Remaining Following Cure at Temperatures of 225, 250 or 275 °F. (Latex B)

Determination of Time Required for Carpet to Reach Oven Temperature Freshly prepared carpeting coated with carboxylated Latex A or B were monitored with thermocouples to determine the length of time required for carpet to reach oven temperatures of 225, 250 and 275 °F when placed in preheated ovens. Since carpet prepared with either Latex A or Latex B required exactly the same length of time to reach oven temperature at 225 °F, experiments to determine the time required for freshly prepared carpet to reach oven temperatures of 250 or 275 °F were conducted with Latex B only. The experiments were repeated. The results are shown in Table VII.

Figure 11 illustrates the pattern of carpet temperature rise when latex coated samples are placed in ovens preheated to specified temperatures.

TABLE VII

TIME REQUIRED FOR CARPET TO REACH AN OVEN
TEMPERATURE OF 225, 250 OR 275 °F

| Latex | <u>Time (Minutes)</u> | | |
|-------|-----------------------|--------|--------|
| | 225 °F | 250 °F | 275 °F |
| A | 17 | ND | ND |
| B | 17 | 25 | > 60 |

ND = Not Done

n = 2 samples, 1 run

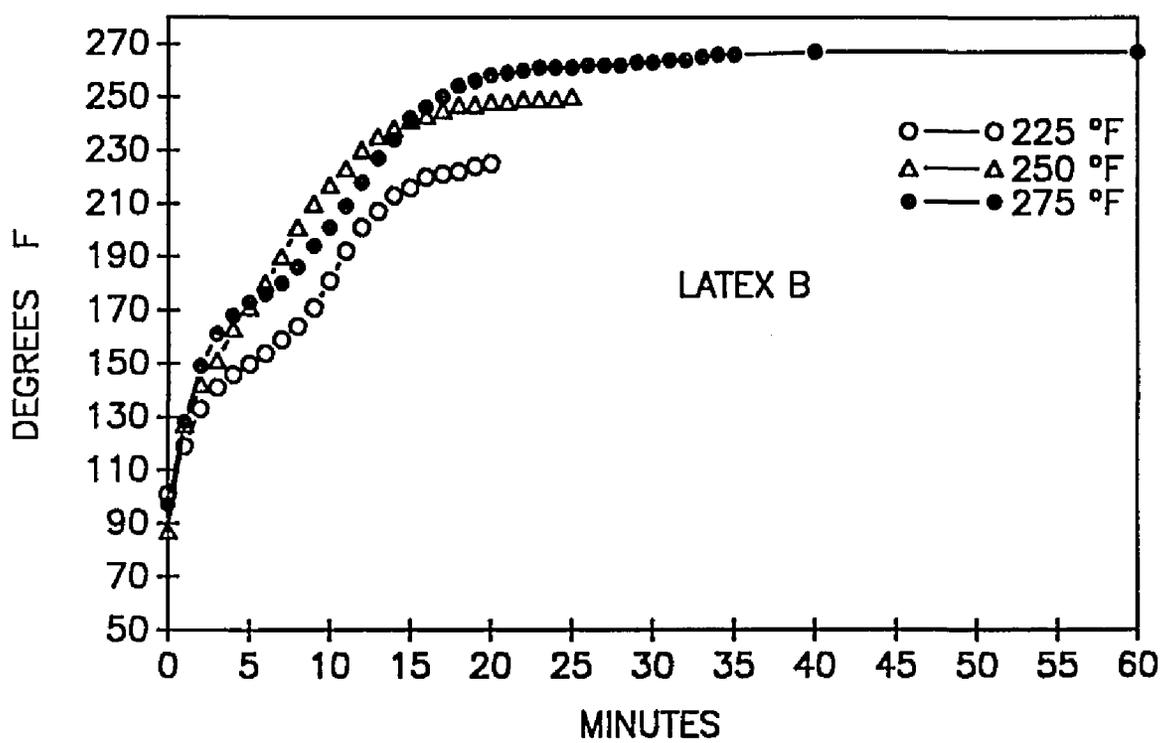


Figure 11. Temperature Rise of Carpet Samples Subjected to Preheated Oven Environments. (Latex B)

Determination of Carpet Moisture Content Following Cure at 225, 250 and 275 °F Freshly prepared carpeting made with Latex B was analyzed for moisture content following cures at 225, 250 and 275 °F for 1, 2, 4, 6, 10, 15 and 20 minutes. Since raw Latex A and B contained exactly the same moisture content (%), experiments with latex coated carpet samples were conducted with Latex B only. Moisture loss following exposure to studied cure parameters are detailed in Table VIII.

TABLE VIII
MOISTURE LOSS FOLLOWING 225, 250 OR
275 °F CURE

| <u>Cure Time</u> | <u>Percent Moisture Lost (%) at Specified Cure Temperatures</u> | | |
|------------------|---|--------|--------|
| Minutes | 225 °F | 250 °F | 275 °F |
| 1 | 20.4 | 17.4 | 24.0 |
| 2 | 36.7 | 34.5 | 36.5 |
| 4 | 61.2 | 67.4 | 71.9 |
| 6 | 57.1 | 66.3 | 71.9 |
| 10 | 87.8 | 93.5 | 95.8 |
| 15 | 96.9 | 94.6 | 96.9 |
| 20 | 100.0 | 100.0 | 100.0 |

n = 2 samples, 2 runs

Figure 12 depicts the moisture loss from carpet coated with Latex B at all three cure temperatures.

Effect of Various Air Circulations on Percent 4-PCH Remaining at a Cure Temperature of 225 °F Freshly prepared carpet made with Latex A was cured at 225 °F for 0, 5, 10, 20, 40 and 60 minutes with no air circulation, with 1500 linear feet per minute (LFM) air flow and 10 air changes per minute, and with no air circulation but with the fan in place and not operating. The carpet swatches were analyzed for 4-PCH content, and the test was repeated. Table IX depicts the results of this experiment.

TABLE IX

EFFECT OF AIR VELOCITY ON PERCENT 4-PCH REMAINING
AT A CURE TEMPERATURE OF 225 °F (LATEX A)

| <u>Cure Time</u> Minutes | <u>Percent 4-PCH Remaining</u> | | |
|-----------------------------|--------------------------------|-------------|--------------|
| | Static | 1500 LFM | Fan in Place |
| 0 | 100.0 ± 3.0 | 100.0 ± 4.7 | 100.0 ± 4.9 |
| 5 | 91.4 ± 2.5 | 42.6 ± 3.7 | 87.7 ± 4.5 |
| 10 | 81.1 ± 3.2 | 9.3 ± 2.2 | 62.1 ± 2.3 |
| 20 | 52.8 ± 5.2 | 0.3 ± 0.5 | 23.2 ± 6.0 |
| 40 | 8.5 ± 1.2 | ND | ND |
| 60 | 1.1 ± 0.6 | ND | ND |

n = 6 samples, 2 runs

The effects of all three air circulations on carpet freshly prepared with Latex A can be seen in Figure 13.

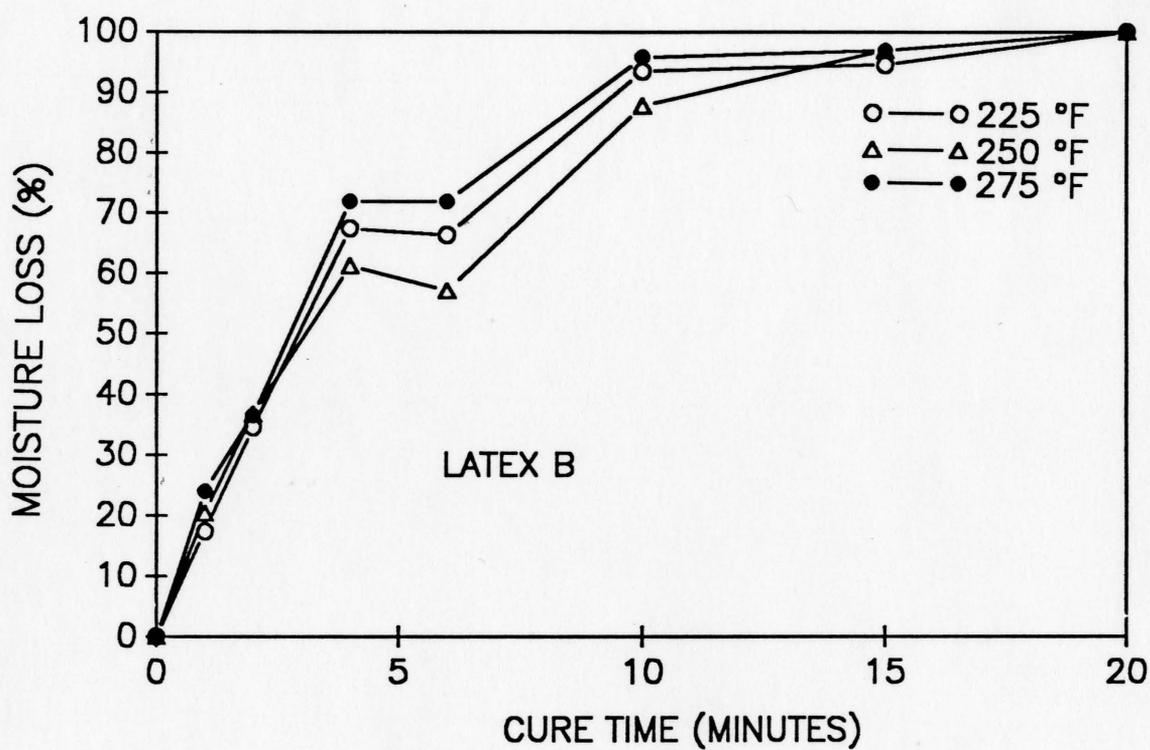


Figure 12. Percent Moisture Loss Over Time at Cure Temperatures of 225, 250 or 275 °F. (Latex B)

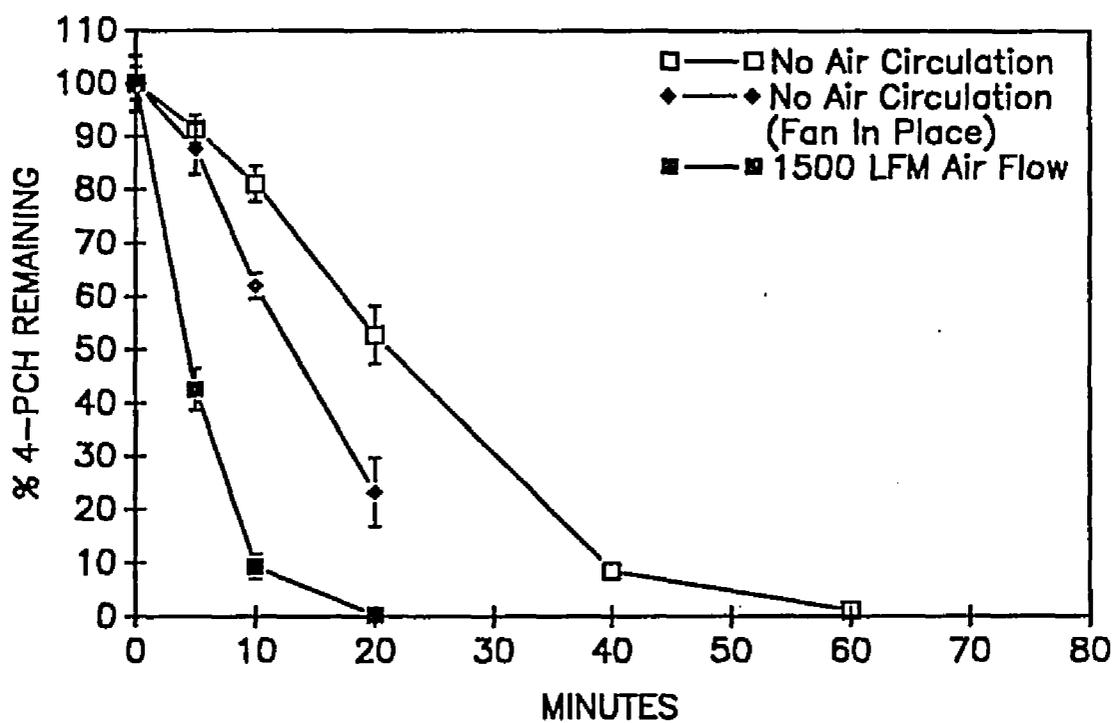


Figure 13. 4-PCH Content of Carpet Following Cure at 225 °F and No Air Velocity, at 1500 Linear Feet per Minute Air Flow, or No Air Circulation but with the Fan in Place.

DISCUSSION

The purpose of this research project was to investigate the effect of specific cure parameters (time, temperature and air flow) on the removal of 4-PCH from freshly prepared carpet backed with carboxylated styrene-butadiene rubber latex.

Release of 4-PCH from Two Latex Composites Containing Different 4-PCH Concentrations

The 4-PCH concentration of the two composite latexes employed in this study had essentially no effect on the decay rate of 4-PCH under a given set of cure parameters. Latex A contained a relatively high concentration (48.2 ± 4.3 ppm) of 4-PCH, whereas Latex B contained a relatively low concentration (18.91 ± 1.8 ppm) of 4-PCH. As shown in Figures 9 and 10, there was no significant difference in rate of 4-PCH release from the high and low 4-PCH composites. Over the range of 4-PCH concentrations tested, the rate of 4-PCH release from carpet backed with carboxylated styrene-butadiene rubber latex is essentially the same, and appears independent of starting concentration.

Effect of Temperature on Reduction of 4-PCH

Increased cure time and cure temperature were found to be effective in reducing 4-PCH levels during the curing

process as shown in Table X. The time required to remove 50% of the 4-PCH from a freshly prepared latex carpet sample was 9, 14 and 21 minutes at cure temperatures of 225, 250 and 275 °F respectively. To eliminate 80% of the 4-PCH required cure times of 18, 20 and 35 minutes at the specified test temperatures, respectively. The rate of loss was 9, 5 and 2% per minute respectively.

In the first 5 minutes of cure, no significant amount of 4-PCH is lost regardless of the cure temperature and, after 40 minutes of cure time, most of the 4-PCH has been outgassed regardless of the test cure temperature (see Figures 9 and 10). Therefore, it is this period between 5 minutes and 40 minutes of cure that temperature appears to be critical to the removal of 4-PCH. Significant reductions are apparent at 250 and 275 °F after approximately 20 minutes. At 275 °F, 60% of the 4-PCH was lost after only 10 minutes compared to 35% and 25% at 250 and 225 °F, respectively.

Carpet cured at 275 °F had reached a temperature of 194 °F by the time 50% of its 4-PCH content had been released, while samples cured at 250 and 225 °F reached temperatures of 238 and 221 °F, respectively, before a comparable loss of 4-PCH content was achieved. Similarly, carpet cured at 275 °F reached a temperature of 255 °F by the time 80% of its 4-PCH content had been released, while samples cured at 250

TABLE X
EFFECT OF TEMPERATURE ON 4-PCH LOSS (%)

| <u>Cure Time</u> | <u>275 °F</u> | <u>250 °F</u> | <u>225 °F</u> |
|------------------|---------------|---------------|---------------|
| 5 minutes | 15 % | 10 % | 10 % |
| 10 minutes | 60 % | 35 % | 25 % |
| 20 minutes | 85 % | 80 % | 50 % |
| 40 minutes | 100 % | 99 % | 90 % |
| 60 minutes | 100 % | 100 % | 100 % |

and 225 °F reached temperatures of 248 and 221 °F, respectively, before a comparable loss of 4-PCH content was attained. Intuitively one would expect carpet cured at 275 °F to have reached a higher temperature at a given time than carpet cured at 250 °F, but this was not the case for measurements made between 5 and 15 minutes at these temperatures.

One explanation for this phenomenon is that the temperature measurements may not always reflect actual carpet temperature. The thermocouples were inserted into the carpet tufts as close as possible to the base, but the latex was applied to the opposite side of the backing. Air trapped within the tufts (or another unknown phenomena) may have produced some variation in temperature readings. The variation in individual temperature readings (2 runs) averaged 6, 5 and 8 degrees at 275, 250 and 225 °F, respectively.

Effect of Temperature on Moisture Loss

All three cure temperatures required approximately the same length of time, ten minutes, to eliminate carpet moisture with approximately 91.4% of the carpet's original weight remaining. At 225 °F and 10 minutes cure time, 91.36% of the solids remained. At 250 °F and 10 minutes cure time, 91.43 % of the solids remained; and at 275 °F

and 10 minutes cure time, 90.84% of the solids remained. Since all three latexes contained the same percentage of moisture, and water boils at 212 °F, all three cure temperatures were sufficient to evaporate the moisture at about the same rate.

Effect of Moisture Loss on Rate of Temperature Rise

A review of the graphs depicting temperature rise as a function of time demonstrates that at all three cure temperatures tested, the rise in carpet temperatures is similar over the first 15 minutes, reaching maximum at approximately 20 minutes. At 250 and 275 °F, the increase in the temperature rise profile is different than that at 225 °F, the rate of increase being less for the latter (225 °F), especially at 5 to 10 minutes. This demonstrates the fact that after the ten minutes required for evaporation of the moisture has elapsed, a greater increase in the rate of temperature rise occurs. (Due to the fact that the energy produced by the oven heat which had previously been used to evaporate the moisture can now be used to raise the carpet temperature.)

Effect of Air Exchange and Air Flow on Percent 4-PCH

Remaining

The percent 4-PCH remaining in carpet samples cured at

225 °F and 3 air changes per minute was compared to that remaining in carpet samples cured at 225 °F with no air exchanges. Only a 2% average reduction in percent 4-PCH remaining was obtained using 3 air changes per minute over no air changes.

A noticeable difference in percent 4-PCH loss was obtained between samples cured at 225 °F and 3 air changes per minute and samples cured at 225 °F and 1500 linear feet per minute air flow with 10 air changes per minute. The 3 air changes per minute samples lost approximately 25% of their 4-PCH after ten minutes, whereas the 1500 linear feet per minute flow samples lost nearly 90% of their 4-PCH over the same time period.

A noticeable difference was also observed between the samples cured with no air circulation, and the samples cured with no air circulation but with the fan in place and not operating. After 10 minutes of cure, the samples with no air circulation had lost 19% of their 4-PCH content, while those cured with the non-operational fan in place had lost 38%. The same phenomenon was observed after 20 minutes of cure, when 47% and 77% were lost, respectively. One possible explanation may be that the fan absorbed heat and then served as a radiant heat source. Therefore, samples cured in the oven with the fan in place were exposed not only to the heat from the oven, but also the heat radiated

by the fan. This phenomenon may have also affected 4-PCH loss at 1500 linear feet per minute and ten air exchanges/min., although it was not within the scope of this research project to explore this possibility.

CONCLUSION

The purpose of this research was three-fold: 1) to develop a method for making tufted carboxylated SBR latex backed carpet uniformly and reproducibly; 2) to develop a method to quantitate the 4-PCH levels in tufted carpet; and 3) to determine the effect that the cure parameters of temperature, time and air circulation have on the release of 4-PCH from latex backed carpeting.

The release of 4-PCH from freshly prepared carpet samples was found to be most prominent once moisture has been released from the carpet; this initial moisture release phase lasts for approximately 5 minutes. This phenomenon of an enhanced 4-PCH release following moisture loss was demonstrated at all three temperatures tested. Increased cure temperature and time also enhanced the loss of 4-PCH from standardized carpet samples. Since increased cure temperature has a profound effect on 4-PCH release in carpet, increasing the oven temperature and cure time to the highest feasible level would seem advisable to reduce 4-PCH odor in carpets supplied to the public.

The effect of air circulation on 4-PCH release was also examined in a limited fashion. It appears that a cure temperature of 275 °F, in combination with 1500 linear feet per minute air flow (10 air changes per minute) impacting the carpet surface, may produce the greatest reduction in 4-

PH content of all parameters studied. Further research in this area appears to be warranted.

These curing experiments were conducted on small samples of carpeting under laboratory conditions and do not necessarily represent values which can be extrapolated to manufacturing conditions. However, the findings here may be used to predict general findings and trends which one may expect to see in the manufacturing process.

Since the odor and potential health effects associated with new carpet have been linked to 4-PH, efforts should be made to reduce its content. This research indicates that cure temperature, time and air velocity are critical to the reduction of 4-PH in SBR latex backed carpeting and, if optimized, could result in significant reductions of 4-PH content.

APPENDIX A

PREPARATION OF CARPET BACKED WITH
STYRENE-BUTADIENE RUBBER LATEX

1.0 INTRODUCTION

1.1 Purpose of the Standard Operating Procedure

The purpose of this SOP is to define the methodology for the preparation of styrene-butadiene rubber latex backed carpet.

1.2 Sources of the Method

1.2.1 VCR Tape from carpet industry

1.2.2 Research Proposal for Investigating the Impact of Various Cure Parameters on the Release of 4-Phenylcyclohexene from Styrene-Butadiene Rubber Latex by Frank R. Demer.

1.3 References

1.3.1 VCR Tape from carpet industry

1.3.2 Research Proposal for Investigating the Impact of Various Cure Parameters on the Release of 4-Phenylcyclohexene from Styrene-Butadiene Rubber Latex by Frank R. Demer.

1.4 Principles of the Method

1.4.1 Carpet made by standard procedures-applicable to industry.

1.4.2 Samples prepared for gas chromatography, analyzed for 4-PCH content.

2.0 APPLICABILITY

This method shall be used for the preparation of standardized carpet samples for use in further research.

3.0 DEFINITIONS

- (a) 4-PCH = 4-phenylcyclohexene
- (b) 1-PCH = 1-phenylcyclohexene
- (c) SBR = styrene-butadiene rubber
- (d) SBLMC = Styrene Butadiene Latex Manufacturer's Council
- (e) Filler = calcium carbonate (Georgia Marble)
- (f) Frothing agent = Southern Chemical 391
- (g) Thickener = Paragum 165
- (h) Carpet = 26 oz. nylon 6:6 pile Saxony with a 3.2 oz. polypropylene backing
- (i) Action backing = secondary backing
- (j) GC = gas chromatography

4.0 RESPONSIBILITIES

- 4.1 The Project Director will be responsible for the determination and implementation of appropriate laboratory procedures.
- 4.2 The Internal Quality Assurance Officer will be responsible for insuring the proper conduct of these procedures, and for periodically reviewing their performance.
- 4.3 The Laboratory Technician will be responsible for the preparation of carpet samples and the analyses of those samples according to the procedures specified in this SOP.

5.0 EQUIPMENT

5.1 Materials

- (a) 26 oz. nylon 6:6 pile Saxony with a 3.2 oz. polypropylene backing
- (b) Action (secondary) backing
- (c) 10" x 10" carpet frames
- (d) 3/4" metal latex roller
- (e) 1 1/2" metal carpet roller
- (f) Pyrex Desiccator
- (g) Desiccant
- (h) 16 ml. reaction vessels
- (i) Reaction vessel septa, caps
- (j) Hamilton Series 700 10ul syringes, Model 87920
- (k) 32 oz. plastic cups with lids
- (l) 20 oz. plastic cups
- (m) 10 oz. plastic cups

- (n) 3.2 oz. plastic cups
- (o) Oxford repipettor
- (p) rubber spatula
- (q) 10" by 10" plexiglass square pattern

5.2 Instrumentation

- (a) Brookfield Portable Viscometer; Model RVT
- (b) Sartorius Series U Balance; Model U6100D+
- (c) Fisher Isotemp Controlled Atmosphere Moisture Oven; Model 496
- (d) Lightnin Labmaster; Model DS3004
- (e) Kitchen Aid 10 Speed Commercial Mixer
- (f) Computing Integrator (HP 3350A or equivalent)
- (g) Hewlett Packard Gas Chromatograph 5890A
- (h) Hewlett Packard Integrator 3390A
- (i) Micronta Thermocouples, Model 22-191
- (j) Millivolt Voltmeter (digital)

5.3 Reagents

- (a) Carbon Disulfide, reagent grade
- (b) Gas Cylinders for GC
- (c) Latexes A & B
- (d) DD Water
- (e) Georgia Marble filler
- (f) Froth Aid
- (g) Paragum thickener
- (h) 4-PCH standard
- (i) 1-PCH standard

6.0 STANDARD OPERATING PROCEDURE

6.1 Prior preparations

6.1.1 Glassware

- (a) Prior to analysis, clean all glassware used to prepare standards, and all reaction vessels, caps, and septa with an approved laboratory cleaning solution.
- (b) Rinse all cleaned glassware 3 times with double-distilled water.

6.1.2 1-PCH Internal Standard

- (a) Low Sensitivity Standard - add 7.5 ul

of 1-PCH standard to 500 ml of carbon disulfide, to give a 1-PCH concentration of 94.13 nmol/ml. Store refrigerated in brown bottle.

- (b) High Sensitivity Standard - add 1.8 ul of 1-PCH standard to 500 ml of carbon disulfide, to give a 1-PCH concentration of 22.6 nmol/ml. Store refrigerated in brown bottle.

6.1.3 4-PCH Standard Solutions

6.1.3.1 Prepare stock 4-PCH standard solution by adding 4 ul of pure 4-PCH to 4 ml carbon disulfide to make a stock standard solution of 6351 nmols/ml.

6.1.3.2 Use the 1-PCH internal standard solution and the 4-PCH stock standard solution to prepare working 4-PCH standard solutions.

(a) 1.57 nmoles 4-PCH/ml working standard solution prepared by mixing 0.50 ul of stock standard 4-PCH to 2 ml internal standard solution.

(b) 3.15 nmoles 4-PCH/ml standard solution prepared by mixing 1.00 ul of 4-PCH to 2 ml internal standard solution.

(c) 9.45 nmoles 4-PCH/ml standard solution prepared by mixing 3.00 ul of 4-PCH to 2 ml internal standard solution.

(d) 15.75 nmoles 4-PCH/ml standard solution prepared by mixing 5.00 ul of 4-PCH to 2 ml internal standard solution.

- (e) 22.05 nmoles 4-PCH/ml standard solution prepared by mixing 7.00 ul of 4-PCH to 2 ml internal standard solution.
- (f) 31.50 nmoles 4-PCH/ml standard solution prepared by mixing 10.00 ul of 4-PCH to 2 ml internal standard solution.

6.2 Preparation of SBR Latex

- 6.2.1 Mix latex thoroughly and weigh out appropriate amount into tared 32 oz. cup (See Appendix B/C).
- 6.2.2 Add appropriate amount of DD water (See Appendix B/C).
- 6.2.3 Mix thoroughly with the Lightning Mixer at 500 rpm for 2 minutes.
- 6.2.4 Add appropriate amount of calcium carbonate filler slowly while mixing at 700 rpm (See Appendix B/C). Continue mixing for 5 more minutes to completely wet filler.
- 6.2.5 Tare 32 oz. cup again and add appropriate amount of froth aid (See Appendix B/C).
- 6.2.6 Mix at 900 rpm for 30 seconds.
- 6.2.7 Tare 32 oz. cup again and add appropriate amount of thickener (See Appendix B/C).
- 6.2.8 Mix at 900 rpm for 5 minutes.
- 6.2.9 Check viscosity with the Brookfield Viscosity Meter.
 - 6.2.9.1 Attach #5 spindle to viscosity meter and set speed on 20 rpm.
 - 6.2.9.2 Fill 10 oz. tared cup about half full with latex mixture.

- 6.2.9.3 Place cup under meter and lower spindle until the top of the latex mixture just reaches the notch around the spindle.
- 6.2.9.4 Turn viscosity meter on and allow to run for 1 minute, or until reading stabilizes. If longer than 2 minutes, check setting.
- 6.2.9.5 Record number on viscosity dial and multiply by factor from conversion table supplied by Brookfield (200) to get viscosity reading in centipoises.
- 6.2.10 Add latex mixture to 10 oz. tared cup until the weight in the cup is 500 grams.
- 6.2.11 Scrape latex mixture out of cup and into stainless steel bowl of Kitchen Aid mixer.
- 6.2.12 Whip mixture with wire whip at highest speed for appropriate time, then at lowest speed for 1 minute.
- 6.2.13 Fill 3.2 oz tared cup with frothed latex mixture, tapping cup gently to remove air bubbles.
 - 6.2.13.1 Weigh the latex mixture in the tared cup.
 - 6.2.13.2 Multiply amount by 9.7 (in the case of this 3.2 oz. cup) to get weight of frothed latex mixture per quart.
- 6.2.14 If the mixture weighs 800 grams/quart (\pm 25 grams), continue to 6.2.15.
 - 6.2.14.1 If the mixture weighs less than 775 grams, continue to 6.2.15, check viscosity. If within range continue.

- 6.2.14.2 If the mixture weighs more than 825 grams, froth for 5 seconds more at high speed, then 1 minute more at lowest speed.
 - 6.2.14.3 Repeat step 6.2.13.
 - 6.2.14.4 If the mixture weighs 800 grams/quart (± 25 grams), continue to 6.2.15.
 - 6.2.14.5 If the mixture still weighs more than 825 grams, keep repeating step 6.2.14.1 and 6.2.14.2 until it reaches 800 grams/quart (± 25 grams).
 - 6.2.14.6 If the mixture now weighs less than 775 grams/quart, continue to 6.2.15.
- 6.2.15 Check the viscosity of the latex mixture.
- 6.2.15.1 Attach the #6 spindle to the viscosity meter.
 - 6.2.15.2 Place the 3.2 oz cup filled with the frothed latex mixture under the meter and lower spindle until the notch just meets the top of the latex mixture.
 - 6.2.15.3 Allow the meter to run at 20 rpm for 30 seconds.
 - 6.2.15.4 Record the number from the meter dial and multiply by number from chart (500) to get viscosity in centipoises.
 - 6.2.15.5 If the viscosity is between 20,000 and 27,000 centipoises, the procedure may be continued and latex used in the preparation of the carpet. If the viscosity is not between these limits, the latex mixture must be discarded and a new batch prepared, starting from

6.2.

- 6.3 Applying the latex mixture to the carpet
- 6.3.1 Mark a 10" by 10" square in black indelible Sharpie marker on the back of a 12" by 12" piece of carpet, using the plexiglass pattern.
 - 6.3.2 Attach carpet to carpet frame by stretching and impaling the marked carpet on top of the nails, tuft side down, aligning the nails along the top and bottom of the square marked on the carpet back, and centering the marked sides so they are equidistant on each side of the nails. (The extra piece of wood that makes up the carpet frame should be in place between the two rows of nails, underneath the carpet sample.)
 - 6.3.3 Place the 1/2" roller on top of the carpet, between the two rows of nails, flush against the top nails.
 - 6.3.4 Weigh the tared 3.2 oz. cup filled with the frothed latex mixture, and remove latex until the cup contains 71 grams of latex mixture. Record exact amount of latex mixture in cup.
 - 6.3.5 Spread the latex mixture evenly along the bar, on the carpet, on the side of the bar opposite to the nails. Record weight of latex mixture left in cup, and determine exact amount applied to carpet.
 - 6.3.6 Grasp bar, placing thumbnails just below bar, thumbs touching carpet, on vertical black lines.
 - 6.3.7 Drag bar toward you, applying no pressure to the bar other than its own weight, keeping thumbnails along black line, so that the latex mixture does not go outside of the black lines, and is applied onto the carpet.

- 6.3.8 When bar is within 1" of nails at bottom, spin bar such that latex mixture is carried upward and around until it is on side of bar opposite to nails, and bar is flush against bottom nails. Spread excess latex mixture evenly along bar.
- 6.3.9 Push bar away from you, toward top nails, using index fingernails to keep latex mixture within black vertical lines, and again applying no pressure to the bar other than its own weight.
- 6.3.10 When bar reaches within 1" of top nails, spin bar again, carrying latex mixture upward and around until it is on side of bar opposite to nails, and bar is flush against top nails. Spread excess latex mixture evenly along bar.
- 6.3.11 Continue until no excess latex mixture remains on bar (1 or 2 times more if necessary).
- 6.3.12 Place 12" by 12" piece of carpet backing, fuzzy side down, so that one side is just below top set of nails, and sides are even with sides of carpet.
- 6.3.13 Gently press down carpet backing and attach evenly to bottom nails.
- 6.3.14 Using heavy, 1 1/2" bar, roll the 10" by 10" square from the bottom nails up to top nails.
- 6.3.14.1 The first roll should have no pressure applied, other than weight of bar.
- 6.3.14.2 The second roll should have slight pressure applied to the bar.
- 6.3.14.3 The third roll should have no pressure applied, other than weight of bar.

- 6.3.15 Carefully remove the completed carpet piece from the frame. Trim along the black lines, producing a 10" by 10" piece.
- 6.3.16 The standardized piece of carpet has been prepared, and may be used as desired in the experiment.

7.0 RECORDS

7.1 Data Collected by this Procedure

7.1.1 Data Sheets

- 7.1.1.1 Data Sheets are prepared for both Latex A and Latex B, with the recipe (appropriate amounts) for each type of latex written on the appropriate sheet. (See Appendix B/C).
- 7.1.1.2 Each sheet also has blank spaces so that the actual amount used can be recorded. There are also spaces to record viscosity data, and weights determined throughout the process.
- 7.1.1.3 All information pertaining to the preparation of this batch of latex should be recorded on this sheet, and the sheet dated.

7.2 Location/Placement of Record Forms

- 7.2.1 Each day's data sheet should be placed in a manila folder, marked with the name of the experiment and the date, and kept in the file cabinet. The gas chromatograph data and all computer data using this batch of carpet will also be kept in this same file.

APPENDIX B

LATEX A COMPOUNDING DATA SHEET

Date _____ Factor _____
 Batch _____

| | <u>DRY</u> grams | <u>WET</u> grams | <u>MEASURED</u> grams |
|---------------------------------|---------------------|---------------------|--------------------------|
| LATEX A | 100.0 | 187.0 | _____ |
| WATER | - | 20.0 | _____ |
| WHITING 200D | 450.0 | 450.0 | _____ |
| SCT 391 FROTHAID | 2.0 | 5.71 | _____ |
| PARAGUM 165 | 1.21 | 9.30 | _____ |
| COMPOUND SOLIDS | 82% | | |
| INITIAL VISCOSITY | _____ | _____ | _____ |
| FROTH | | | |
| FROTH TIME (SEC) | _____ | _____ | _____ |
| FROTH CUP WEIGHT (PER QUART) | _____ | _____ | _____ |
| FROTH VISCOSITY | _____ | _____ | _____ |
| TOTAL WEIGHT | _____ | _____ | _____ |

From Gencorp Polymer Products, Dalton Ga.

APPENDIX C

LATEX B COMPOUNDING DATA SHEET

Date _____ Factor _____
 Batch _____

| | <u>DRY</u> grams | <u>WET</u> grams | <u>MEASURED</u> grams |
|---------------------------------|---------------------|---------------------|--------------------------|
| LATEX A | 100.0 | 185.0 | _____ |
| WATER | - | 26.0 | _____ |
| WHITING 200D | 450.0 | 450.0 | _____ |
| SCT 391 FROTHAID | 2.0 | 5.71 | _____ |
| PARAGUM 165 | 0.75 | 5.80 | _____ |
| COMPOUND SOLIDS | 82% | | |
| INITIAL VISCOSITY | _____ | _____ | _____ |
| FROTH | | | |
| FROTH TIME (SEC) | _____ | _____ | _____ |
| FROTH CUP WEIGHT (PER QUART) | _____ | _____ | _____ |
| FROTH VISCOSITY | _____ | _____ | _____ |
| TOTAL WEIGHT | _____ | _____ | _____ |

From Gencorp Polymer Products, Dalton Ga.

APPENDIX D

Determination of Optimum Extraction Time
and Extraction Volume

| Minutes of Extraction | nm 4-PCH/gram of Latex | ml of CS ₂ |
|-----------------------|------------------------|-----------------------|
| 10 | 832 | 2 |
| | 819 | 2 |
| 20 | 1281 | 2 |
| | 1278 | 2 |
| 30 | 1254 | 2 |
| | 1263 | 2 |
| 40 | 1248 | 2 |
| | 1256 | 2 |
| 50 | 1229 | 3 |
| | 1195 | 3 |
| 60 | 1246 | 4 |
| | 1246 | 4 |

APPENDIX E

Depiction of Moisture Content of Carpet Swatches
After > 20 minutes of Cure at Specified Temperatures

| Minutes of Cure | n | Cure Temperature | Moisture Content (%) (Avg. per run) |
|--------------------|--------------------|---------------------|---|
| 20 | 24 samples, 4 runs | 275 °F | 8.68 |
| | | | 8.30 |
| | | | 9.08 |
| | | | 8.86 |
| 20 | 12 samples, 2 runs | 250 °F | 8.14 |
| | | | 8.51 |
| 20 | 36 samples, 6 runs | 225 °F | 8.11 |
| | | | 8.67 |
| | | | 8.39 |
| | | | 8.73 |
| | | | 8.46 |
| | | | 8.08 |
| 40 | 24 samples, 4 runs | 275 °F | 8.81 |
| | | | 8.63 |
| | | | 8.40 |
| | | | 9.51 |
| 40 | 12 samples, 2 runs | 250 °F | 8.63 |
| | | | 8.97 |
| 40 | 36 samples, 6 runs | 225 °F | 8.24 |
| | | | 8.76 |
| | | | 8.27 |
| | | | 9.79 |
| | | | 8.70 |
| | | | 8.94 |
| 60 | 18 samples, 3 runs | 275 °F | 8.24 |
| | | | 8.88 |
| | | | 8.83 |
| 60 | 12 samples, 2 runs | 250 °F | 8.01 |
| | | | 9.02 |
| 60 | 36 samples, 6 runs | 225 °F | 8.28 |
| | | | 8.49 |
| | | | 7.85 |
| | | | 8.52 |
| | | | 8.25 |
| | | | 9.03 |

APPENDIX F

Variability of 4-PCH Content Within Representative
Sample of Prepared Carpeting Cured at 275 °F

| | Cure Time, Minutes | | | |
|---------------|--------------------|-----------|-----------|-----------|
| | <u>10</u> | <u>20</u> | <u>40</u> | <u>60</u> |
| 4-PCH Content | 24 | 3 | 0 | 0 |
| (ppm) | 33 | 4 | 1 | 0 |
| | 25 | 4 | 0 | 0 |
| | 34 | 4 | 1 | 0 |
| | 27 | 3 | 0 | 0 |
| | 33 | 4 | 0 | 0 |
| | 27 | 2 | 0 | 0 |
| | 32 | 3 | 1 | 0 |

n = 8 samples, 1 run

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