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Removal of Per□uorocarboxylic Acids (PFCAs) from Carpets Treated with Stain-protection Products by Using Carpet Cleaning Machines

SCIENCE

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Removal of Perfluorocarboxylic Acids (PFCAs) from Carpets Treated with Stain-protection Products by Using Carpet Cleaning Machines

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Notice

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Executive Summary

E.1 Background and Objective

Perfluorinated carboxylic acids (PFCAs) are fully fluorinated organofluorine compounds with a carboxylic acid functional group (-COOH). As a member of the PFCA family, perfluorooctanoic acid (PFOA) and its salts were once used as a surfactant in the manufacturing of perfluorinated polymers such as polytetrafluoroethylene (PTFE). Thus, PFOA and its salts may exist as residuals in PFTE or other fluoropolymer products. PFOA and other PFCAs may also exist in fluorotelomer products (such as stain-repellants) as unwanted reaction by-products. The U.S. Environmental Protection Agency (EPA) began investigating PFOA and its related chemicals in the 1990s and found that it is very persistent in the environment, is found at low levels both in the environment and in the blood of the general U.S. population, and causes developmental and other adverse effects in laboratory animals (U.S. EPA, 2012). In 2006, EPA and the eight major companies in the fluoropolymer and fluorotelomer industry launched the PFOA Stewardship Program, in which companies committed to reduce global facility emissions and product content of PFOA and related chemicals by 95 percent by 2010 and to work toward eliminating emissions and product content by 2015 (U.S. EPA, 2012).

Previous studies have shown that consumer articles that are made from or treated with fluoropolymers and fluorotelomers products may contain low levels of PFCAs. PFCAs are found in a variety of consumer products, including, but not limited to, treated clothing and textiles, floor care products, paper containers for food, and carpets. Among the consumer articles examined by Washburn et al. (2005) and Guo et al. (2009), carpet that was pretreated with stain-repellents and carpet that was treated with after-market stain-resistant formulations were the largest PFOA sources in homes. Once PFCAs are brought into the indoor environment, they are expected to stay for a long period of time because PFCAs are persistent in the environment and because most PFCAs are semi-volatile compounds. PFCAs can also be absorbed by household dust, which may serve as a source for inhalation or digestive exposure. Therefore, it is important to understand the feasibility of in-situ removal of PFCAs from treated carpet. To our knowledge, this issue has not been addressed by any publications in the existing literature.

The main goal of this study was to quantify the efficiency of common carpet cleaning methods ― steam cleaning and hot water extraction ― in the removal of PFCAs from residential and commercial carpet that was manually treated with stain-protection solutions. The objective was to determine if these cleaning techniques are viable methods for reducing indoor exposure to PFCAs associated with carpet that was previously treated with PFCA-containing products.

E.2 Test Method

E.2.1 Test Facility

The carpet cleaning experiments were performed under close-to-realistic conditions in the U.S. EPA research house located in Cary, NC. The research house is a three-bedroom, ranch-style house with a crawl space, a central, forced-air heating system that uses natural gas, and an electric air-conditioning system. The total floor area is 126 m^2. The master bedroom (MB) and front-corner bedroom (FCBR) were used for the carpet cleaning tests.

E.2.2 Test Materials

Carpet

By the time this project started, mill-treated carpet containing high levels of PFCAs were no longer available in local stores, an indication that the manufacturers had taken actions to reduce or eliminated PFCAs from their products. In this study, two types of carpet with low-levels of background PFCAs were treated with after-market carpet treatment solutions (see below). The two types of carpet were: 1) residential carpet that was Green-Label Certified by the Carpet and Rug Institute (CRI) with a pile yarn content of 100% polytrimethylene terephthalate (PTT) – a product from recycled plastic bottles – with a textured cut pile and a woven polypropylene backing, and 2) commercial carpet made with Antron® fiber, a nylon 6,6, hollow-filament fiber with a soil-resistant treatment incorporated into the fiber. This type of commercial carpet is commonly used in schools and offices. The PFCA content in these carpets were below 8 ng/g. These background PFCAs may have come from recycled old carpet.

Carpet Treatment Solutions

Two commercial carpet stain-protection treatments (CT-1 and CT-2) were used to treat the carpet. The total PFCA contents in these treatment solutions are presented in Section E.3.1 below.

Carpet Cleaning Machines

Two carpet cleaning machines were chosen to clean the carpets after treatment with a carpet stain-protection solution: 1) a residential cleaning machine (CM-1), the Rug Doctor Mighty Pro model MP C-20, and 2) a portable professional steam cleaner (CM-2), the Century 400 Ninja Warrior. The residential unit uses hot tap water for the extraction with no additional heating during the cleaning process; the professional steam cleaner has an 1850-watt, in-line heater that produces steam. Figure E.1 shows the Rug Doctor (front) and the Ninja Warrior (rear).

E.1. Residential and commercial carpet cleaning machines used for the tests

Carpet Cleaning Detergent

Two carpet cleaning agents were selected to test the removal of PFCAs from carpets, i.e., a residential carpet cleaning detergent (CD-2) recommended for use with the residential machine (CM-1) and a commercial carpet cleaning detergent (CD-1) chosen for use with the commercial machine (CM-2). According to the material safety data sheets (MSDSs), CD-1 contains 3 to 6% of dipropylene glycol methyl ether (CAS# 34590-94-8) while CD-2 contains 1 to 5% of sodium 2-ethylhexyl sulfate (CAS# 126-92-1) and <1% of branched tridecylalcohol (CAS# 69011-36-5). Prior to use, each clean agent was screened for PFCAs. The residential detergent measured most PFCAs below the quantification limits of the instrument, with perfluorotridecanoic acid (C_{13}) having the highest concentration at 3 ng/mL. The commercial detergent had only trace levels of C_8 through C_{10}, and all were below the practical quantification limits.

E.2.3 Test Procedure

The test carpet was installed on a solid urethane carpet pad on the bedroom floors. Prior to the application of the carpet treatment solution, carpet samples were taken to determine the PFCA content in untreated carpet. The carpet was then treated with a carpet treatment solution. Following the application of the treatment and a subsequent 48-hour drying period, a series of three carpet cleanings was performed. A drying period of at least 48 hours followed each cleaning before carpet samples were collected. The first set of tests used only hot water or steam for the cleaning process to determine the efficiency of each carpet cleaning machine's method of removing the applied PFCAs. Additionally, four experiments, one of which was a duplicate test, were conducted using cleaning detergents (CD-1 or CD-2) in conjunction with a carpet cleaning machine to assess any additional PFCA removal. The complete experimental test matrix is summarized in Table E.1.

Table E.1. Experimental matrix for testing of carpet care treatments

Experiment	Carpet Type	Carpet Treatment (CT)	Cleaning Machine (CM)	Carpet Detergent (CD)
1	Commercial	CT-1	CM-2	None
2	Commercial	CT-2	CM-2	None
3[a]	Residential	CT-1	CM-1	None
4	Residential	CT-1	CM-2	None
5	Residential	CT-2	CM-2	None
6	Commercial	CT-2	CM-2	CD-1
7	Residential	CT-1	CM-1	CD-2
8[b]	Residential	CT-1	CM-1	CD-2
9	Residential	CT-2	CM-2	CD-1

[a] Initial scouting test; [b] Duplicate test.

E.3 Findings

E.3.1 Extractable PFCAs in Carpet Treatment Solutions

The total extractable PFCAs (i.e., the sum of all PFCAs quantified) in the two carpet treatment solutions were, respectively, 6360 and 7500 ng/g. The distributions of individual PFCAs are shown in Figure E.2. The error bars represent ±1 standard deviation (n = 4 for CT-1 and n = 6 for CT-2).

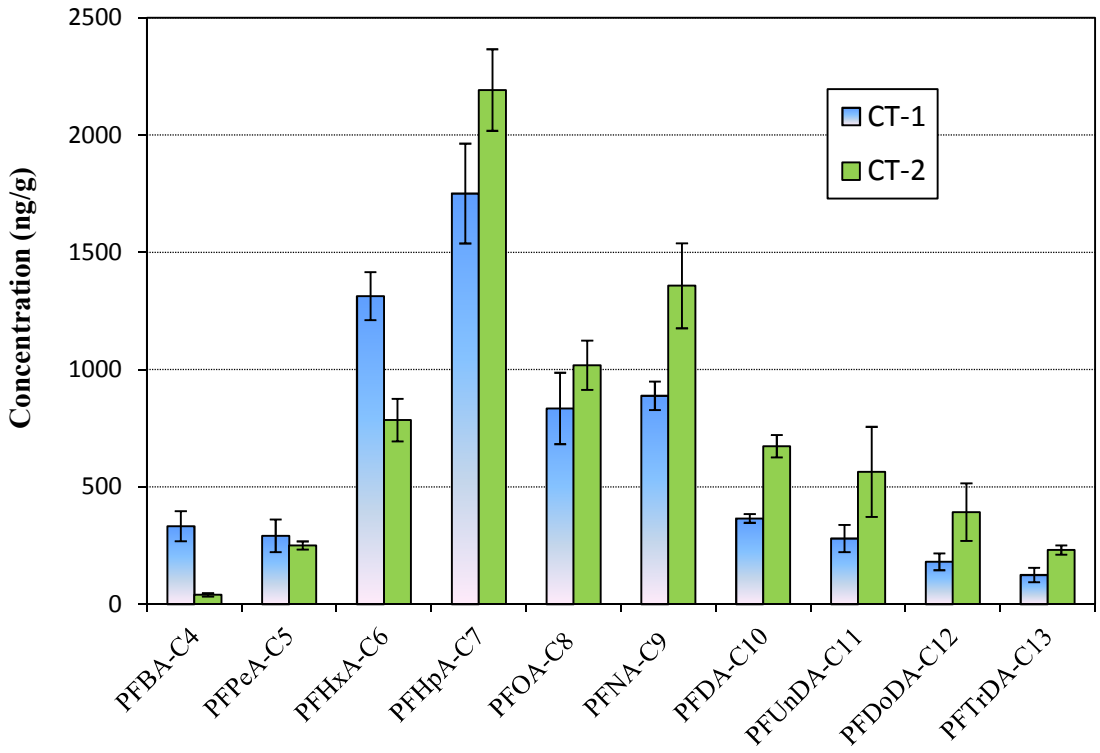

Figure E.2 PFCA contents in the two carpet treatment solutions (CT-1 and CT-2)

E.3.2 PFCA Content in Carpet Before and After Cleaning

As an example, Figure E.3 shows the general patterns of the carpet cleaning tests. The application of the carpet treatment solution increased the PFCA content in the carpet and, following each carpet cleaning event, there was reduction of PFCA content in the carpet. In this experiment, the total reduction after three rounds of cleaning was approximately 50%.

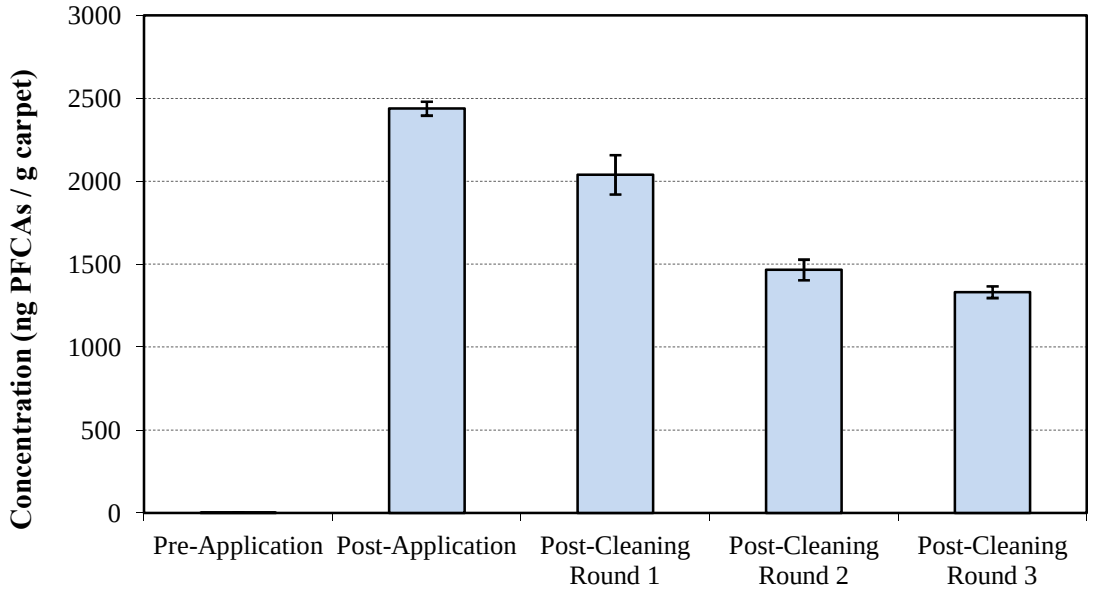

Figure E.3 Average total PFCAs in composite carpet fiber samples for Experiment 4

[Residential carpet treated with carpet treatment solution CT-1; hot-water extraction with the residential cleaning machine (CM-1); no detergent]

E.3.3 Percent Removal of PFCAs by Carpet Cleaning

For each experiment, the percent removal of total PFCAs after the final cleaning was calculated by comparison of the total PFCA concentration after application of the carpet treatment to the total PFCA concentration after the third round of cleaning. As shown in Figure E.4, the percent removal of the total PFCA ranged from 26% to 76% with an average of 55%.

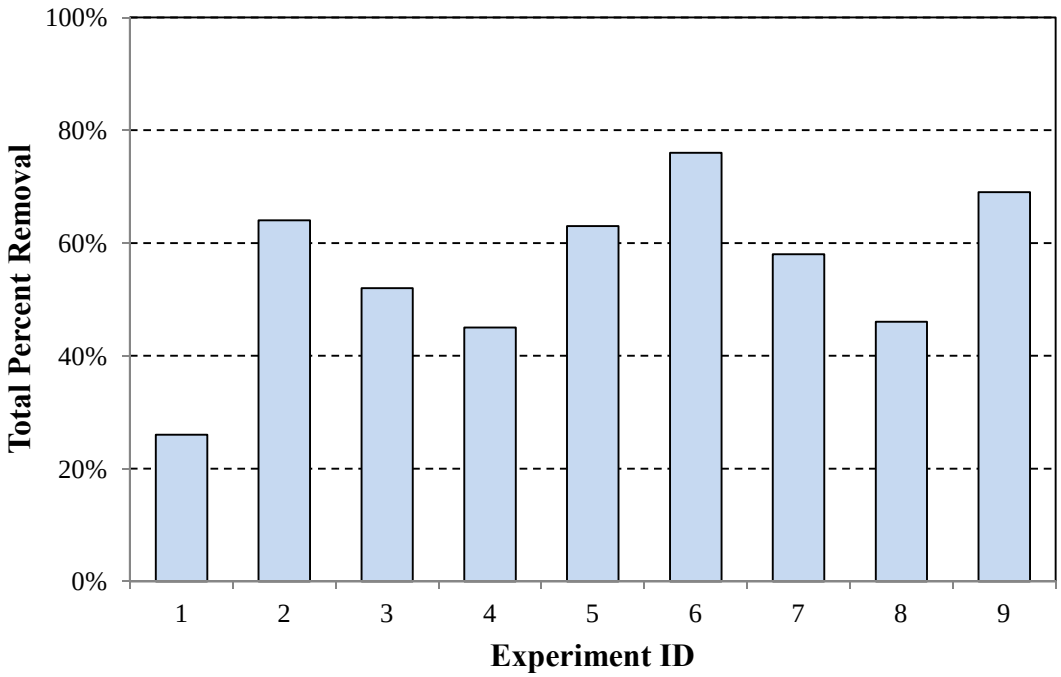

Figure E.4. Percent removal of total PFCAs following three rounds of cleaning for each experiment

E.3.4 Effect of Cleaning Machine

The two types of cleaning machines yielded similar removal efficiency for total PFCAs. The difference shown in Table E.2 is not statistically significant.

Table E.2 Comparison of the removal efficiency of the two cleaning machines for total PFCAs following three rounds of cleaning

ID	Machine Type	Cleaning Method	Mean	SD	n
CM-1	Residential	Hot-water extraction	52.2%	6.2%	3
CM-2	Commercial	Steam cleaning	57.4%	18.5%	4

E.3.5 Effect of Detergent

Using detergent during carpet cleaning showed modest increase in removal efficiency (Figure E.5). However, comparison of the test with detergent CD-1 with the test without using any detergent yielded the two-tailed P value of 0.1063. By conventional criteria, this difference is considered to be not statistically significant.

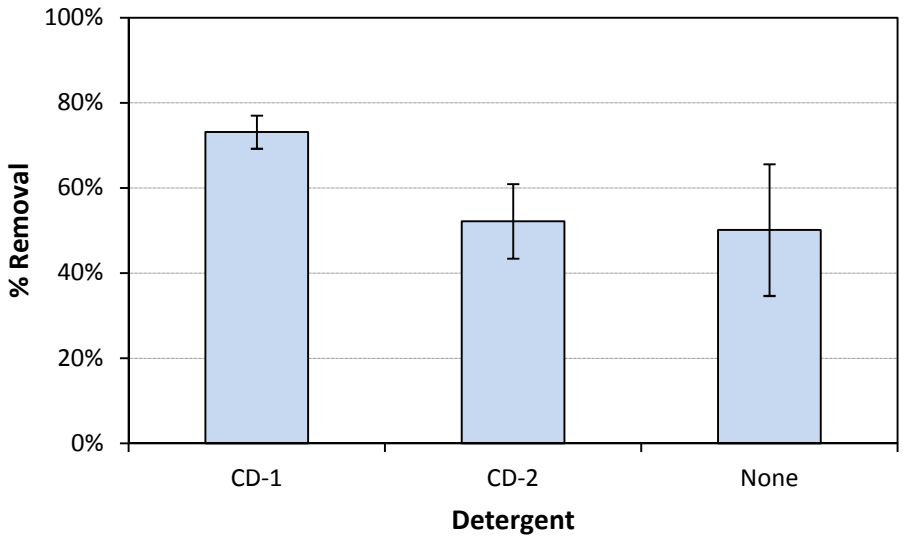

Figure E.5 Effect of using carpet detergent on the average removal efficiency for total PFCAs following three rounds of cleaning

E.4 Research Implications

The average American home has about 1000 square feet (93 m^2) of carpet. Previous research has shown that treated carpets represent one of the largest sources of PFCAs in homes. Additionally, PFCAs are highly stable, with the potential for a long residence time indoors, and can also bind to house dust, making it difficult to remove the PFCAs from the indoor environment. The results of this research indicated that cleaning carpets can reduce the amount of PFCAs they contain, but the chemicals cannot be totally eliminated by the cleaning process. Carpet cleaning by hot-water or steam cleaning is only modestly effective in removing PFCAs. On average, the removal efficiency for each round of cleaning is approximately 20%. At this removal efficiency, it requires three, seven, and ten rounds of cleaning to remove, respectively, 50%, 80%, and 90% of total PFCAs in treated carpet.

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Acronyms and Abbreviations

AOC	Articles of Commerce
APPCD	Air Pollution Prevention and Control Division
BDL	below detection limit
CAS#	Chemical Abstract Service Registry Number
CD	carpet detergent
CFR	Code of Federal Regulations
CM	carpet cleaning machine
CRI	Carpet and Rug Institute
CT	carpet treatment
DCC	daily calibration check
FCBR	front corner bedroom
HPLC	high performance liquid chromatography
IAP	internal audit program
IDL	instrument detection limit
IS	internal standard
LC/MS/MS	liquid chromatography/tandem mass spectrometry
LOQ	limit of quantification
MBR	master bedroom
MDL	method detection limit
MQO	measurement quality objective(s)
MSDS	material safety data sheet
NR	not reported
NRMRL	National Risk Management Research Laboratory
PFAA	perfluoroalkyl acid
PFBA	perfluorobutyric acid
PFC	perfluorochemical
PFCA	perfluorocarboxylic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
PFTeDA	perfluorotetradecanoic acid
PFTrDA	perfluorotridecanoic acid
PQL	practical quantification limit
PTFE	polytetrafluoroethylene
PTT	polytrimethylene terephthalate
QAPP	Quality Assurance Project Plan
QC	quality control
RCS	recovery check standard
RIS	recovery internal standard
RSD	relative standard deviation
TPFCA	total perfluorocarboxylic acids (the sum of all monitored PFCAs)
U.S. EPA	United States Environmental Protection Agency

1. Introduction

1.1 Background

The potential impacts of perfluorocarboxylic acids (PFCAs) on human health and the global environment did not draw much attention until the turn of this century, when evidence of their widespread presence in various environmental compartments appeared (Renner, 2001; Giesy and Kannan, 2002). PFCAs have been detected in air, water, and soil (Boulanger et al., 2004; Stock et al., 2007) and in a variety of wildlife around the world, including the United States (Kannan et al., 2002), Arctic Canada (Martin et al., 2004), Europe, and the Mediterranean Sea (Giesy and Kannan, 2002). More recently, low levels of PFCAs have been found in various consumer products that are either made from or treated with perfluorinated chemicals (Washburn et al., 2005; Guo et al., 2009).

PFCAs persist in the environment due to their high stability, and there is evidence that they bioaccumulate (Moody et al., 2002), a significant factor in their potential toxicity. Perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA), two types of perfluoroalkyl acids (PFAAs) that have been the most extensively studied PFCs to date, are essentially ubiquitous in humans; they have been found in human blood and breast milk in the United States (Tao et al., 2008; Calafat et al., 2006), China (So et al., 2006), and other countries across Europe, South America, and Asia (Kannan et al., 2004). Toxicological studies indicate that PFCAs cause developmental and systemic toxicity in laboratory animals (Kennedy et al., 2004; Lau et al., 2004; U.S. EPA, 2005). In particular, PFOS has been shown to act as an endocrine disruptor (Austin et al., 2003). The potential health risks associated with PFCAs have inspired extensive research on the sources, transport, transformation, and distribution of these chemicals and their precursors in environmental media, along with research into ways to reduce potential health risks.

Despite significant progress thus far, researchers have yet to reach a consensus on the most important routes by which the general population is exposed to these chemicals. In particular, opinions differ on whether consumer products containing PFCAs are significant contributors to overall exposure. For instance, a study in 2005 concluded that exposures to PFOA during consumer use of the articles evaluated were not expected to cause adverse health effects in infants, children, adolescents, or adults, or result in quantifiable levels of PFOA in human serum (Washburn et al., 2005). In a study conducted in 2009 by Fromme et al., data from indoor measurements in Canada and Norway were used to estimate the average daily intake of PFOA for the general population in Western countries, and it was found that the inhalation of house dust contributed only 0.6% of the average daily intake of PFOA and a maximum of only 8.2% of the highest daily intake levels. By contrast, however, Tittlemier et al. (2007) identified treated carpeting as an important source of PFOA exposure, second only to ingestion with food. Trudel et al. (2008) agreed that the consumption of contaminated food is the most significant exposure pathway for PFOA and that the ingestion of dust and inhalation of air containing PFOA is the second most likely route of exposure in low- and intermediate-exposure scenarios. Their study also found that direct, product-related exposure dominates in high-exposure scenarios in which consumers have treated carpets in their homes or regularly use PFCA-containing products, such as stain-protection sprays. It is apparent, then, that the scarcity of data related to indoor PFCA sources

and exposures contributes to significant uncertainty and differences of opinion about the most prevalent exposure routes for these compounds.

Elevated levels of PFCAs have been detected in house dust in Japan (Moriwaki et al., 2003), Canada (Kubwabo et al., 2005), and the United States (Strynar and Lindstrom, 2008), strongly suggesting the presence of significant indoor sources. Kubwabo et al. correlated the percentage of indoor carpet, in particular, to increased PFCA levels in dust. In 2005, Washburn and colleagues reported the PFOA content in 14 groups of articles based on theoretical calculations and analytical measurements. Of these groups, it was found that pre-treated carpet and carpet treated with carpet-care solution had the highest PFOA loadings, i.e., 0.2 to 0.6 mg and 0.2 to 2 mg of PFOA per kg, respectively (Washburn et al., 2005). Previous research by Guo et al. (2009) examined 14 classes of consumer products and also found pre-treated carpet and carpet treated with PFCA-containing carpet-care solutions to have the largest PFCA source strength, i.e., approximately 70 mg of PFCAs (C5 through C12) in a typical home. One study found that PFCA contamination was higher on the inside than the outside of sampling films placed in windows (Gewurtz et al., 2009). Gerwurtz's study also found higher PFCA contamination in houses with new carpet and in carpet stores, suggesting that emissions from new carpets contribute to increased indoor PFCA levels.

Due to the highly stable nature of PFCAs, few mechanisms exist, short of eliminating sources completely, by which PFCAs can be removed from the indoor environment. Given its high source strength and duration, its potential contribution to indoor dust, and its close proximity to humans, treated carpeting may contribute to human exposure to PFCAs directly, e.g., dermal contact and hand-to-mouth transfer, and indirectly via the inhalation of suspended particles from treated carpet.

1.2 Goal and Objective

The main goal of this study was to quantify the efficiency of common carpet cleaning methods ― steam cleaning and hot water extraction ― in the removal of PFCAs from residential and commercial carpet that was manually treated with stain-protection solutions. Our aim in the study was to determine if these cleaning techniques are viable methods for reducing indoor exposure to PFCAs. These results will be valuable to policy makers and manufacturers for risk management purposes and may be of particular interest to people who wish to reduce the levels of PFCA in their household environments.

2. Materials and Methods

2.1 Test Facility

Carpet cleaning tests were conducted in a research house located in Cary, NC. The research house is a three-bedroom, ranch-style house with a crawl space, a central, forced-air heating system that uses natural gas, and an electric air-conditioning system. The total floor area is 126 m^2, and the house has a total volume of approximately 300 m^3. Additional information on the test house was provided by Tichenor et al. (1990) and Sparks et al. (1991). The two rooms of the house employed for this work were the front corner bedroom (FCBR) and the master bedroom (MBR), as shown in Figure 2.1. For this study, the carpeted areas of the MBR and FCBR measured 13.4 m^2 and 12.2 m^2, respectively.

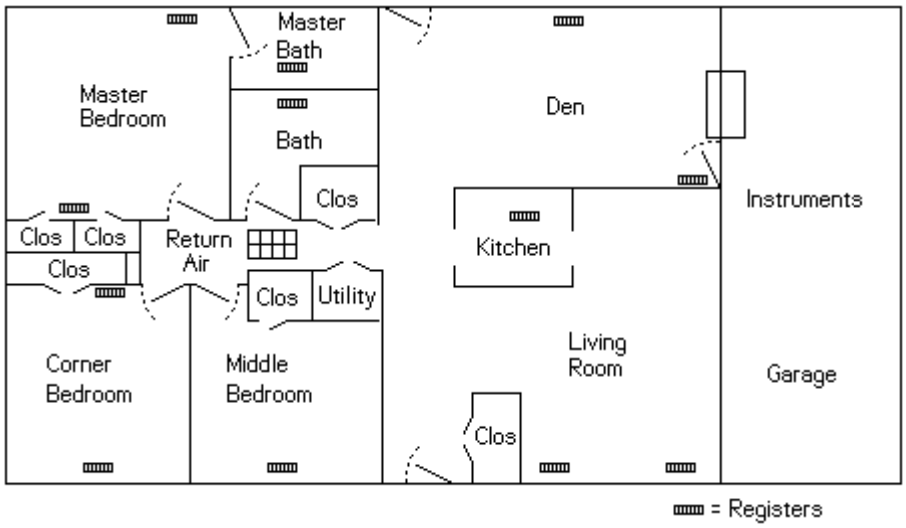

Figure 2.1. Floor plan for the research house in Cary, NC

The air exchange rate inside the house was monitored but not reported for these experiments. An OPTO 22 data acquisition system (OPTO 22, Temecula, CA) continuously logged temperature and humidity data in the FCBR and den. Air exchange rate, temperature, and humidity were not controlled as critical parameters, but temperature and relative humidity were recorded and reported. Additional conditions in the research house are listed in Table 2.1.

Table 2.1. Conditions in the research house

Parameter	Status
Exterior doors and windows	Closed
Interior doors	Open
Ceiling fans	On low
Heating/air conditioning fan	On
Heating/air conditioning registers	Open

2.2 Test Materials

2.2.1 Carpet Selection for Research House Experiments

The following two carpet types were chosen for the research house experiments: 1) residential carpet that was Green-Label Certified by the Carpet and Rug Institute (CRI) with a pile yarn content of 100% polytrimethylene terephthalate (PTT) – a product from recycled plastic bottles – with a textured cut pile and a woven polypropylene backing and 2) commercial carpet made with Antron® fiber, a nylon 6,6, hollow-filament fiber with a soil-resistant treatment incorporated into the fiber. The residential carpet fiber, also called Triexta®, shares the chemical structure of polyester; however, unlike traditional polyester carpet, Triexta® has extreme durability, high stain resistance, and a much softer texture. The fiber used for the commercial carpet is considered to be one of the most durable in the industry, and it has been utilized extensively in schools and offices. A solid urethane carpet pad was selected as the underpad for each type of carpet. Each of these products was screened to determine the content of PFCAs prior to installation. A 1-g sample of fiber from each type of carpet and 1 g of the solid memory foam backing carpet pad were analyzed to determine their PFCA content before the experiments were initiated. All of the samples from all of the products were found to contain less than 8 ng/g of PFCAs.

2.2.2 Professional Carpet Treatment Solutions

Two carpet stain-protection treatment solutions (CT-1 and CT-2) were selected using the following criteria: (1) shown to contain high levels of PFCAs (greater than 10 times the PQL); and (2) analytical data with good recovery for the recovery internal standards.

Table 2.2 lists the components of the selected treatment products as documented in each product's material safety data sheet (MSDS). The documentation indicated that both treatments are dispersible in water. The PFCA content in these treatment solutions are reported in Section 3.2 below.

Table 2.2. Components of carpet treatments CT-1 and CT-2

Chemical	Content (%)	
	CT-1	CT-2
Polymethylmethacrylate (CAS# 9011-14-7)	1.3 – 2.5	4.7
NJ Trade Secret Registry #00850201001-5155P	1 – 1.5	–
NJ Trade Secret Registry #00850201001-5259P	0 – 1.3	–
Citric Acid (CAS# 77-92-9)	0.5 – 1	–
Water (CAS# 7732-18-5)	90 – 97	95

2.2.3 Carpet Cleaning Machines

Two carpet cleaning machines were chosen to clean the carpets after treatment with a carpet stain-protection treatment: 1) a residential cleaning machine (CM-1), the Rug Doctor Mighty Pro model MP C-20 and 2) a portable professional steam cleaner (CM-2), the Century 400 Ninja Warrior. Figure 2.2 shows the Rug Doctor (front) and the Ninja Warrior (rear). The residential machines, which can be rented at most grocery and home improvement stores, use hot tap water for the extraction process with no additional heating during the cleaning process. A 28-psi (1.93×10^5 Pa) vacuum pump is used to extract the applied water, and a brush vibrates during the process. The professional steam cleaner has an 1850-watt, in-line heater. Steam at a pressure of approximately 150 psi (1.03×10^6 Pa) soaks the carpet fibers, and dual 2-stage vacuum motors extract the residue from the carpet. The professional cleaning machine does not agitate the carpet fibers.

Figure 2.2. Residential and commercial carpet cleaning machines

2.2.4 Carpet Cleaning Detergents

Two detergents were selected to test the removal of PFCAs from carpets, i.e., a residential carpet cleaning detergent (CD-2) recommended for use with the residential machine (CM-1) and a commercial carpet cleaning detergent (CD-1) chosen for use with the commercial machine (CM-2). The residential detergent was purchased at a local grocery store, and the commercial detergent was purchased from a local distributor of janitorial supplies. According to the material safety data sheets (MSDSs), CD-1 contains 3 to 6% of dipropylene glycol methyl ether (CAS# 34590-94-8) while CD-2 contains 1 to 5% of sodium 2-ethylhexyl sulfate (CAS# 126-92-1) and <1% of branched tridecylalcohol (CAS# 69011-36-5). Prior to use, each cleaner was screened for PFCAs. The residential detergent measured most PFCAs below the quantification limits of the instrument, with perfluorotridecanoic acid (C_{13}) having the highest concentration at 3 ng/mL. The commercial detergent had only trace levels of C_8 through C_{10}, and all were below the practical quantification limits.

2.3 Experimental Design

All experiments were conducted at the research house in Cary, NC. The FCBR was used primarily for the residential carpet, and the MBR was used for both residential and commercial carpet. The residential carpet was tested with both types of cleaning machines and detergents to represent what might be used in a typical household, while the commercial carpet was tested with only the commercial cleaning machine and the commercial detergent. An initial scouting test (Experiment 3) was performed with the residential carpet and CM-1 to finalize testing procedures before beginning the planned experiments. To ensure the presence of detectable PFCAs, all experiments involved the application of a commercial carpet stain-protection treatment solution containing PFCAs (CT-1 or CT-2). Following the application of the treatment and a subsequent 48-hour drying period, a series of three carpet cleanings was performed. A drying period of at least 48 hours followed each cleaning before carpet samples were collected. The first set of tests used only hot water or steam for the cleaning process to determine the efficiency of each carpet cleaning machine's method of removing the applied PFCAs. Additionally, four experiments, one of which was a duplicate test, were conducted using cleaning detergents (CD-1 or CD-2) in conjunction with a carpet cleaning machine to assess any additional PFCA removal. The complete experimental test matrix is summarized in Table 2.3.

Table 2.3. Experimental matrix for testing of carpet care treatments

Experiment	Research House Location	Carpet Type [c]	Treatment Type	Cleaning Machine	Detergent
1	MBR	C	CT-1	CM-2	None
2	MBR	C	CT-2	CM-2	None
3[a]	FCBR	R	CT-1	CM-1	None
4	FCBR	R	CT-1	CM-2	None
5	FCBR	R	CT-2	CM-2	None
6	MBR	C	CT-2	CM-2	CD-1
7	FCBR	R	CT-1	CM-1	CD-2
8[b]	MBR	R	CT-1	CM-1	CD-2
9	FCBR	R	CT-2	CM-2	CD-1

[a] Initial scouting test.
[b] Duplicate of Experiment 7.
[c] C = Commercial; R = Residential.

2.4 Test Procedure

2.4.1 Layout of Carpet Sampling Sections

The carpet in the MBR measured 3.66 × 3.66 meters, and the carpet in the FCBR measured 3.66 × 3.20 meters. For each room, a grid was created in order to evenly gauge five sampling sections, A-E, as shown in Figure 2.3. Once each section was located, a small piece of tape the size of a dime was placed in its center. This was the only indicator placed on the carpet itself and acted as a reference point for sampling (Section 2.5.1).

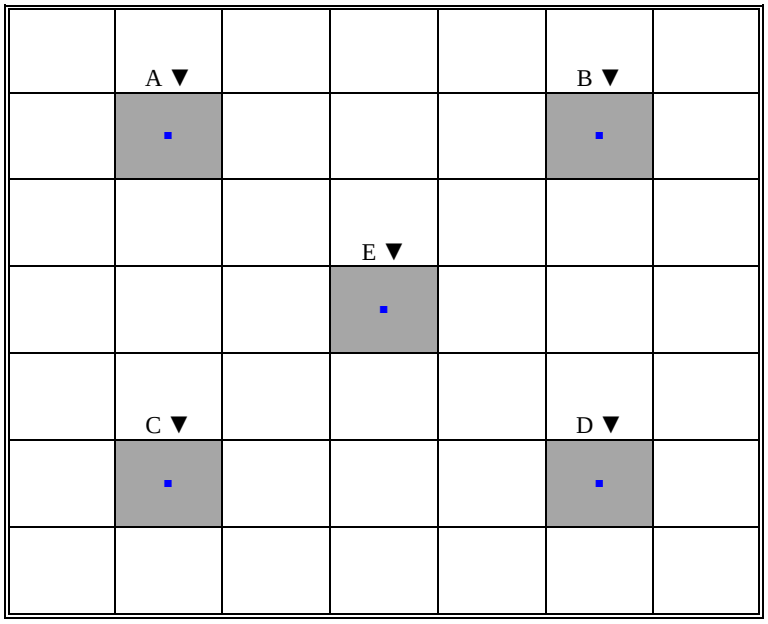

Figure 2.3. Diagram of generic carpet sampling quadrant

2.4.2 Dilution and Application of the Carpet Treatment

The dilution of the carpet treatment varied slightly for each set of tests. The treatment (CT-1) dilution for the scouting test (Experiment 3) was 1:1 distilled water to treatment solution to ensure measurable concentrations of PFCA through three cleanings. The manufacturer's recommendation was 4:1, and the resulting concentrations of PFCAs from the 1:1 dilution on the carpet indicated that a lower concentration of application treatment would be more applicable for this study; therefore, a dilution of 4:1 was used for all subsequent applications with both products. An exact measurement for the dilution was not critical because the dried product on the carpet fiber was collected and extracted for analysis before each test.

To start the application, the diluted carpet stain-protection treatment solution was placed in a commercial sprayer recommended in the product use guide for application of the treatment. The sprayer was modified by adding a pressure gauge to better control the application rate and uniformity of the application. Then, the sprayer was inserted into the "application cart," an apparatus manufactured in-house (Figure 2.4). The application cart was on wheels in order to provide a more uniform application of the solution, and it held the nozzle of the sprayer approximately 0.6 m from the ground.

Figure 2.4. Application cart

First, the carpet treatment was applied according to Figure 2.5. For each path, the applicator was pumped to a pressure of 10 psi (6.89×10^4 Pa), the spray nozzle was turned on, and the cart was pulled in the direction of the arrows for approximately 24 s. At the end of each path, the spray nozzle was turned off. A grid on the wall allowed for proper path widths based on the swath of the sprayer; the MBR had seven paths, while the FCBR had eight.

						Start
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
↓	↓	↓	↓	↓	↓	↓
End (Door)	↓	↓	↓	↓	↓	↓

Figure 2.5. Diagram of first application process

Once the application shown in Figure 2.5 was completed, a second, S-shaped application was performed, as shown in Figure 2.6. During this application, the process was not stopped at the end of each path. Instead, the application was done in a continuous motion, only stopping to pump the applicator to 8-10 psi (5.52×10^4 - 6.89×10^4 Pa) when necessary. After application the carpet was allowed to dry for at least 48 hour before carpet samples were collected.

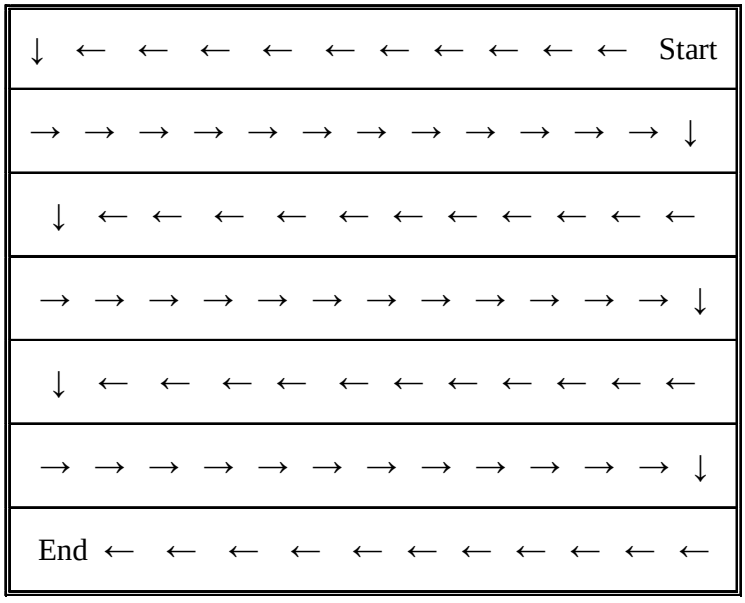

Figure 2.6. Diagram of second application process

2.4.3 Cleaning of Carpets

The manufacturer's instructions were followed for each carpet cleaning machine. When using the commercial cleaner (CM-2), the carpets were cleaned in paths that were approximately 3-3.5 ft (0.91-1.07 m) wide with about 1 in (2.54 cm) of overlap. Figure 2.7 demonstrates this process, where each section of the schematic represents a path, and the arrows indicate the direction of cleaning. For each path, there were four passes with the carpet cleaning apparatus with the cleaning solution being expelled and vacuumed up in the first pass and any remaining solution being vacuumed up in the final three passes, which, in turn, dried the carpet. Each 3-ft pass was timed for approximately10 seconds. The residential cleaner (CM-1) followed a similar cleaning process, except that 6-ft-wide paths were used.

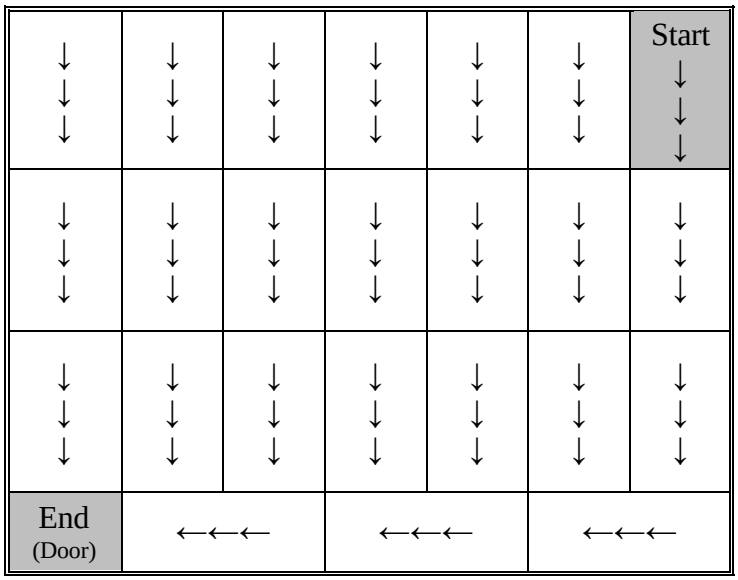

Figure 2.7. Diagram of carpet cleaning using commercial cleaner CM-2 (not to scale)

2.5 Sampling Methods

2.5.1 Collection of Carpet Samples

A carpet sample from each of the five sampling locations (Figure 2.3) was collected according to Table 2.4, using a 82-mm-diameter carpet cutter (05-174 Instant Repair Tool, Crain Cutter Company, Milpitas, CA). Methanol was used to clean the tool between samples to prevent cross-contamination. Each sample was placed in an individually-labeled plastic bag and transferred to the EPA campus for processing and analysis. Each 82-mm-diameter carpet sample was processed by removing the fibers from the backing and placing them in an aluminum foil dish. Each set of carpet samples was placed in a separate dessicator. After a minimum of 16 hours, the fiber sample was removed and mixed. A 1-g sub-sample was transferred to a 50-mL polypropylene centrifuge tube (Evergreen Scientific, Los Angeles, CA), and a second 1-g sub-sample was weighed and placed in a plastic bag. Once all individual samples from the stage had been processed, the plastic bag containing the remaining 5 g of carpet from quadrants A-E were used to compile five composite fiber samples. The bag was shaken to ensure that all fibers were distributed evenly, and five individual 1-g sub-samples of composite carpet fiber were removed from the plastic bag and placed in separate centrifuge tubes. In summary, each stage of the experiment produced a total of 10 samples, each weighing 1 g, i.e., five individual fiber samples (A-E) and five composite fiber samples composed of fibers from all five sampling locations. The individual sample data were used only to evaluate the uniformity of the application of the carpet treatment, while the composite samples were used to evaluate cleaning efficiency.

Table 2.4. Carpet fiber collection strategy

Stage of Experiment	Number of Samples Collected from Carpet	Number of Fiber Samples Processed for Analysis [a]	Number of Composite Fiber Samples Processed for Analysis [b]
Prior to Treatment Application (Initial Sampling)	5	5	5
Post-Treatment Application (Application Sampling)	5	5	5
Post-cleaning, Round 1 (Clean 1 Sampling)	5	5	5
Post-cleaning, Round 2 (Clean 2 Sampling)	5	5	5
Post-cleaning, Round 3 (Clean 3 Sampling)	5	5	5

[a] One ~1-g sample per sampling location.
[b] Five ~1-g samples taken from an equal mixture of fiber samples from all five locations.

Figure 2.8 shows a residential carpet after all sampling stages were completed.

Figure 2.8. Residential carpet after all sampling stages

2.5.2 Wipe Sampling

Wipe samples were collected only during Experiment 3, the scouting test, to evaluate over-spray during the application process. These data illustrate any possible exposure from wall surfaces in a typical home. Using ASTM Method D 6661, *Field Collection of Organic Compounds from Surfaces Using Wipe Sampling* (ASTM, 2010), as a general guideline, a 10 cm × 10 cm template

was used to outline the wipe area using painters tape. Four wipe areas were sampled on each of the four walls in the center of the carpet length, four inches above the baseboard molding. For each wipe sample, approximately 2 mL of methanol were applied to a piece of cotton gauze. The gauze was pressed firmly to the wall, and the sample area was wiped vertically with minimal overlap between strokes. Then, the sample area was wiped horizontally. The gauze used for the wipe was stored in a 50-mL polypropylene centrifuge tube and later extracted using the same procedure that was used for the residential carpet samples, which is described in Section 2.6.1. Background wipe samples were collected at each location before application of the carpet treatment.

2.6 Sample Analysis

2.6.1 Extraction of Residential Carpet Samples

To each of the residential fiber samples prepared in Section 2.5, 45 mL of HPLC-grade methanol (pre-screened for PFCAs) and 100 µL of recovery check standard (2 ng/µL each of perfluoro-*n*-[1, 2-$^{13}C_2$] decanoic acid Wellington Laboratoies, Canada) were added. The samples were extracted for 24 ± 2 hr using a Nutating Mixer (Model VSN-5, PRO Scientific, Inc., Oxford, CT). The extract was transferred to a 170-mL borosilicate glass blow-down tube. The original sample vial was rinsed three times with ~3 mL of methanol, and each of the rinse liquids was transferred to a 170-mL concentration tube. A spatula was used to agitate the samples during rinsing. Next, the sample was concentrated to approximately 1.5 mL in a heated (50 °C) nitrogen atmosphere by using a RapidVap N_2 Evaporation System (Model 791000, LabConco, Kansas City, MO), which was previously modified at the factory to remove all PTFE parts and coatings.

The blow-down sample was transferred to a 10-mL volumetric flask through a 0.1-µm Anotop syringe filter (Whatman International, Madestone, England). The tube was rinsed five times with a solution consisting of 60% (v/v) methanol and 40% (v/v) 2 mM ammonium acetate aqueous solution (hereafter referred to as 60:40 solution). The rinse liquids were filtered and combined with the blow-down sample in the volumetric flask. After adding 100 µL of the internal standard solution (0.5 ng/µL each of perfluoro-*n*-[1, 2, 3, 4-$^{13}C_4$] octanoic acid), the sample was brought to10 mL with 60:40 solution and sonicated for 10 min. The sample solution was transferred to a 15-mL polypropylene tube and stored at 4 °C until LC/MS/MS analysis.

2.6.2 Extraction of Commercial Carpet Samples

After extraction, due to clogging of the 0.1-µm Anotop filter, the sample first had to be first filtered through a Corning 0.22-µm cellulose acetate, low-binding filter and into a polystyrene tube. The 170-mL concentration tube was rinsed five times, and the extract was passed through the 0.22-µm Corning filter. Then, the sample extract was transferred through a 0.1-µm Anotop syringe filter into a 10-mL volumetric flask. The filter tube was rinsed five times with 60:40 solution into the flask. After adding 100 µL of the internal standard solution, the sample was brought to volume with 60:40 solution and sonicated for 10 min. The sample solution was transferred to a 15-mL polypropylene tube and stored at 4 °C until LC/MS/MS analysis.

2.6.3 Extraction of Liquid Samples

Approximately 1 mL of the liquid sample was weighed, spiked with 1 mL of a 2 ng/µL recovery check standard solution, and diluted to 10 mL with 60:40 solution. The diluted samples were sonicated for 10 min and then filtered with a 0.1-µm Anotop syringe filter. After filtration, 1 mL of the filtrate was transferred into a 10-mL volumetric flask and spiked with 100 µL of the internal standard. The resulting solution was sonicated for 10 min, transferred to a polypropylene tube, and stored at 4 °C until LC/MS/MS analysis. Note: for samples with levels of PFCAs above the calibration range, a second dilution of 1:10 was needed before adding the recovery check standard.

2.6.4 QC Sample Preparation

A set of five quality control (QC) samples was prepared for every batch of carpet extractions, i.e., a field blank, a solvent blank, and three recovery internal standard blanks. The field blank consisted of 45 mL of methanol and 100 µL of recovery internal standard, and it went through the same extraction process as the residential carpet samples described in Section 4.6.1. The solvent blank consisted of 60:40 solution with 100 µL of internal standard, brought to volume in a 10-mL volumetric flask. Each recovery internal standard blank contained 60:40 solution with 100 µL internal standard and 100 µL recovery internal standard, brought to volume in a 10-mL volumetric flask. All samples including field blank samples were sonicated, transferred to 15-mL polypropylene tubes, and stored at 4 °C with the corresponding batch of extraction samples until LC/MS/MS analysis. The analyte content in the solvent blank was subtracted from all samples and field blanks if it exceeded the practical quantification limit (PQL). The analyte content in the field blanks was required to be below the PQL.

2.6.5 Sample Analysis

Sample quantification was conducted using an Agilent 1100 HPLC equipped with an Applied Biosystems API 3200 Triple Quadrupole Mass Spectrometer with a Turbo V ion-spray interface. A C18 reversed-phase guard column and analytical C18 reversed phase column were used for analyte separation. Samples were injected at a flow rate of 0.250 mL/min and maintained at 50 °C. The initial gradient mobile-phase composition was 25% mobile phase B, where mobile phase A was 95% 2 mM aqueous ammonium acetate-5% methanol, and mobile phase B was 95% methanol-5% 2 mM aqueous ammonium acetate, held for 0.5 min. A linear gradient was used from 25% to 85% B over 4.5 min, then increased to 100% B over 0.10 min and held for 2 min. Then, a linear gradient decreased mobile phase B to 25% over 2 min, where it was held for 3 min. PFCAs were observed in the negative ion mode, and both primary and secondary ion transitions were collected for each analyte. The instrument was calibrated for 11 PFCA homologues (Table 2.5) plus the recovery check standards at eight concentration levels in the concentration range of 0.3 to 100 ng/mL with triplicate injections. This procedure followed methods detailed in Liu et al. (2009).

Table 2.5. Analyte names, abbreviations, chemical formulas, molecular weights (g/mol), and Chemical Abstracts Service registration numbers (CAS #)

Analyte	Abbreviation	Formula	MW	CAS #
Perfluorobutyric acid	PFBA – C4	$C_4HF_7O_2$	214.04	375-22-4
Perfluoropentanoic acid	PFPeA – C5	$C_5HF_9O_2$	264.04	2706-90-3
Perfluorohexanoic acid	PFHxA – C6	$C_6HF_{11}O_2$	314.05	307-24-4
Perfluoroheptanoic acid	PFHpA – C7	$C_7HF_{13}O_2$	364.05	375-85-9
Perfluorooctanoic acid	PFOA – C8	$C_8HF_{15}O_2$	414.06	335-67-1
Perfluorononanoic acid	PFNA – C9	$C_9HF_{17}O_2$	464.07	375-95-1
Perfluorodecanoic acid	PFDA – C10	$C_{10}HF_{19}O_2$	514.07	335-76-2
Perfluoroundecanoic acid	PFUdA – C11	$C_{11}HF_{21}O_2$	564.08	2058-94-8
Perfluorododecanoic acid	PFDoA – C12	$C_{12}HF_{23}O_2$	614.09	307-55-1
Perfluorotridecanoic acid	PFTrDA – C13	$C_{13}HF_{25}O_2$	664.11	72629-94-8
Perfluorotetradecanoic acid	PFTeDA – C14	$C_{14}HF_{27}O_2$	714.12	376-06-7
Perfluoro-*n*-[1,2-$^{13}C_2$] hexanoic acid [a]	13C-PFHxA	$^{13}C_2{}^{12}C_4HF_{11}O_2$	316.04	n/a
Perfluoro-*n*-[1,2,3,4-$^{13}C_4$] octanoic acid [b]	13C-PFOA	$^{13}C_4{}^{12}C_4HF_{15}O_2$	418.03	n/a
Perfluoro-*n*-[1,2-$^{13}C_2$] decanoic acid [a]	13C-PFDA	$^{13}C_2{}^{12}C_8HF_{15}O_2$	516.07	n/a

[a] Recovery check standard.
[b] Internal standard.

2.7 Quality Assurance and Quality Control

A Category II quality assurance project plan (QAPP) was developed and approved before the start of the project and detailed the Measurement Quality Objectives (MQOs) for the project, which are summarized in Table 2.6. For this study conducted at the EPA research house, the data quality objective for the precision of solvent extraction was relaxed to ± 30% to incorporate more information to better interpret the desired goal of this project. All data that fell outside the original MQOs listed below are highlighted in the data tables presented in Appendix A.

Table 2.6. Measurement quality objectives

Measurement	Parameter	Objective	Method
Calibration of LC/MS/MS	Coefficient of determination (r^2) for calibration curve	> 0.99	Linear regression
PFCA quantification by LC/MS/MS	Instrument detection limit	≤0.2 ng/mL	EPA method (40 CFR 136, 1986)
	Accuracy	85-115%	Internal audit program Daily calibration check standards
	Precision	20%	RSD for duplicate injections
	Agreement of primary and secondary ions	25%	Percent deviation (in highest 10% of samples)
Solvent extraction	Precision	20% [a]	RSD for replicate extractions
	Accuracy	80-120%	Recovery internal standard
	System blank	< IDL	Extraction w/o AOC[b] sample
Weight of AOC samples	Accuracy	±2 mg	NIST-traceable weights

[a] For data completeness, some extraction results presented below had precisions between 20% and 30%; data with precisions of greater than 30% were discarded.
[b] Articles of Commerce.

2.7.1 Uniformity of Carpet Protector Treatment Solution

The variability of the carpet treatment application and the inconsistency of the cleaning machine process (temperature, time of cleaning in single location, and operator variations) made the collection of truly duplicate carpet samples impossible. Individual samples from each of the five sampling locations were collected at each stage of the experiment, as indicated previously in Figure 2.3. Sampling in five different locations accounted for potential variations in both the treatment application and carpet cleaning. These five samples were analyzed individually and also combined to create a large mass composite carpet fiber sample. The accumulation and mixing of sub-samples from different locations eliminated application and cleaning variability and lack of uniformity in treatment applications. Five 1-g samples were removed from the composite samples after mixing. These replicate samples served as proxy duplicates. Figure 2.9 presents the results of the average of the five individual samples collected from positions A-E post-application from each experiment to demonstrate variations in the application of the treatment. A high standard deviation for the averages of the individual samples is an indicator of application variability. These data from individual samples are presented in Appendix A. The composite samples, as a physical average of carpet conditions, ideally would reduce these inconsistencies in PFCA deposition and removal.

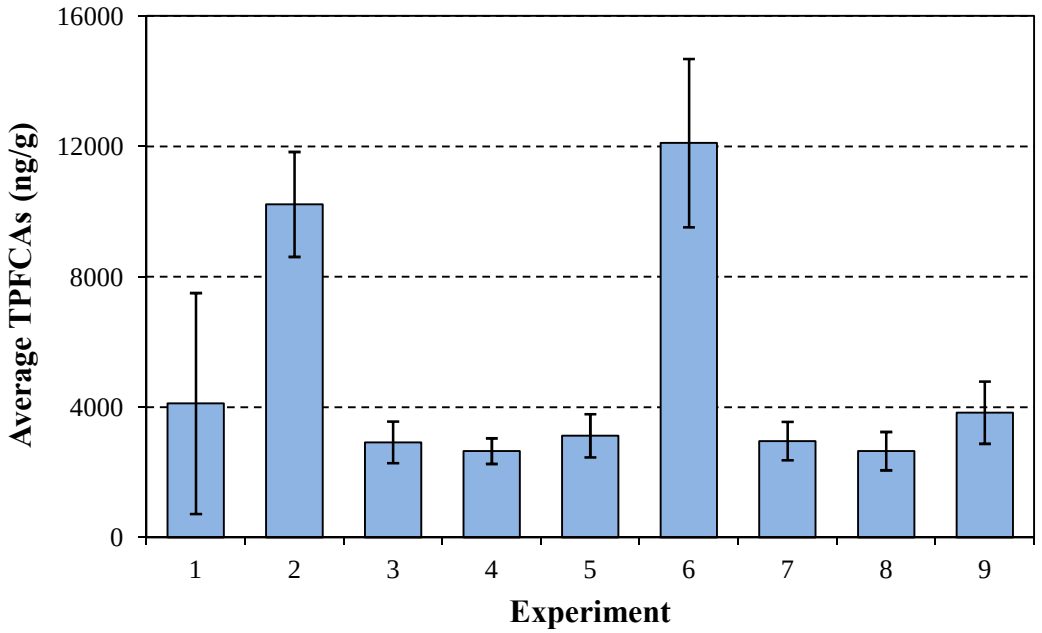

Figure 2.9. Average of individual quadrant samples for post-application PFCA concentrations in each experiment and associated standard deviations

2.7.2 Calibration of LC/MS/MS

The instrument was calibrated for 11 PFCA homologs (Table 2.5) plus the recovery check standards at eight concentration levels in the concentration range of 0.3 to 100 ng/mL with triplicate injections. The instrument detection limits (IDLs) for individual PFCAs in the injected sample were ≤ 0.2 ng/mL. The practical quantification limit (PQL) for the injection sample was < 1 ng/mL, which is equivalent to 1 ng/g for carpet extracts and 10 ng/g for liquid samples.

Initially, standards were prepared from individual analytes (Liu et al., 2009). However, in March 2010 calibrations were performed using a composite standard mix distributed by the manufacturer that contained the all of the analytes of interest. This change was due to the availability of a composite standard and the resulting simplification of the standard preparation process.

The acceptance criterion for the calibration curve was that the coefficient of determination (r^2) be no less than 0.99. The average of the r^2 values for 14 compounds over seven calibrations was 0.995 ± 0.003, giving 100% completeness for the MQO for calibration.

An internal audit program (IAP) standard was analyzed for each of the seven calibrations. The IAP standard contained at least three of the calibrated PFCAs using a different chemical source (Oakwood Laboratories or Aldrich) and was prepared by someone other than the analyst who prepared the calibration standards. The analyst who conducted the calibration received the IAP standard without knowing the concentrations. IAP standards were analyzed after each calibration as a measurement of calibration verification. The average of the percent accuracy for each analyte

ranged from 76% to 115%. Only two data points out of 26 did not meet the MQO criterion for acceptance of ± 15%, giving 92% completeness for this QA criterion.

2.7.3 Daily Calibration Checks

Daily calibration check (DCC) standards, approximately 10 ng/mL for each analyte, were analyzed to evaluate the performance of the LC/MS/MS. The DCC was conducted at the beginning of each analytical sequence and at the end of the sequence or, for extended sequences, after 24 hours of instrumental analysis. Analytical results of a sample batch were considered acceptable only if the percent recovery of the DCC was within 100 ± 15% and the percent relative standard deviation (%RSD) of triplicate injections of the DCC was within ± 15%. The MQO for DCC recovery was relaxed for this study to 100 ± 30%, and the percent relative standard deviation (%RSD) of triplicate injections of the DCC was instead required to be within ± 30%. Data that fell outside the original MQO for DCCs are highlighted in the data tables in Appendix A. Data that fell outside the relaxed MQOs were not used in this report. We collected 952 data points for the DCC measurements with only 55 points falling outside the relaxed MQO, giving 94% completeness using the relaxed MQO.

2.7.4 Contamination Checks

All purchased and prepared solvents, glassware, and the HPLC system were checked routinely for PFCA contamination. Also, a solvent blank was prepared with each set of standards and samples to assess the solvent and the HPLC system. Solvents and blanks were rejected if they contained the analytes of interest at concentrations higher than the IDL for individual PFCAs in the injection sample of ≤0.2 ng/mL.

2.7.5 Weight Measurements

Two balances were employed for weight measurements during this project, i.e., 1) a five-place microbalance and 2) a three-place pan balance. The microbalance was used early in the project for weighing the calibration standards for the LC/MS/MS. Both balances are calibrated every 12 months. To monitor the daily performance of the balances, two Class A weights were weighed at the beginning and end of each weigh session. The precision of each balance for this project was determined by averaging the measurements for each class A weight and determining the standard deviation for those measurements. The accuracy was determined by comparison of the class A weight measurement to the certified value of the weight. Table 2.7 presents these MQO measurements.

Table 2.7. MQOs for weight measurements

Parameter	Microbalance	Pan Balance
Precision (n > 10)	10 mg: 9.997 ± 0.038 mg 20 g: 19.999 ± 0.00014 g	1 g: 1.000 ± 0.001 g 10 g: 10.002 ± 0.001 g
Accuracy %	10 mg: 99.97% 20 g: 99.99%	1 g: 100.05% 10 g: 100.01%

3. Results

3.1 Summary of Experimental Conditions

The research house conditions for the nine experiments are summarized in Table 3.1.The relative humidity and air exchange rate were measured but not controlled during the research house experiments. After each application and subsequent cleaning, the relative humidity was elevated for several hours during the drying period for the carpets. The temperature was controlled by the conventional heating and air conditioning system in the house. The HVAC fan was operated continuously, and the ceiling fans in the FCBR, MBR, den, and living room were kept on low speed, blowing upward. Measurements for relative humidity and temperature were monitored in the FCBR and den by a Vaisala INTERCAP humidity and temperature transmitter recording to an OPTO DAS. The air exchange rate inside the house was not reported for any of the experiments. The only purpose of presenting the environmental data in Table 3.1 is to show that the carpet cleaning experiments were conducted under typical indoor environmental conditions. The data was not used anywhere else in this report.

Table 3.1. Environmental parameters recorded during the cleaning experiments

Experiment [a]	Temperature, °C [b]			Relative Humidity, % [b]		
	Average [c]	High	Low	Average [c]	High	Low
1 and 4	22 ± 1 [d] 24 ± 0.5 [e]	24 25	20 22	59 ± 6 54 ± 4	77 65	48 44
2 and 5	22 ± 1 [d] 24 ± 1 [e]	24 26	20 22	61 ± 4 56 ± 2	75 65	55 51
3	21 ± 1 [d] 19 ± 1 [e]	26 22	19 18	24 ± 6 25 ± 4	58 40	14 18
6 and 9	21 ± 1 [d] 23 ± 0.5 [e]	23 25	19 21	59 ± 6 52 ± 4	75 63	47 42
7 and 8	NA [f]	NA [f]	NA [f]	NA [f]	NA [f]	NA [f]

[a] Tests are grouped together by the start date for each test (one test being conducted in the FCBR and the other in the MBR). Test 3 was the initial scouting test conducted in the FCBR.
[b] Measurement locations: front corner bedroom (FCBR) and den.
[c] Mean ± standard deviation for n > 2500 (Sample number varied with each experiment).
[d] Transmitter in the FCBR.
[e] Transmitter in the Den.
[f] Not available — Data lost due to power failure at the house.

3.2 Extractable PFCA Content in Carpet Treatment Solutions

The levels of PFCAs quantified in the undiluted carpet protector treatments CT-1 and CT-2 are presented in Figure 3.1. The error bars show ±1 standard deviation with n = 4 for CT-1 and n = 6

for CT-2. The treatments were analyzed for 11 PFCAs; however, the levels of PFTeDA (C14) in the treatment solutions were below the limit of detection. The total PFCA content for CT-1 and CT-2 was, respectively, 6360 and 7500 ng/g.

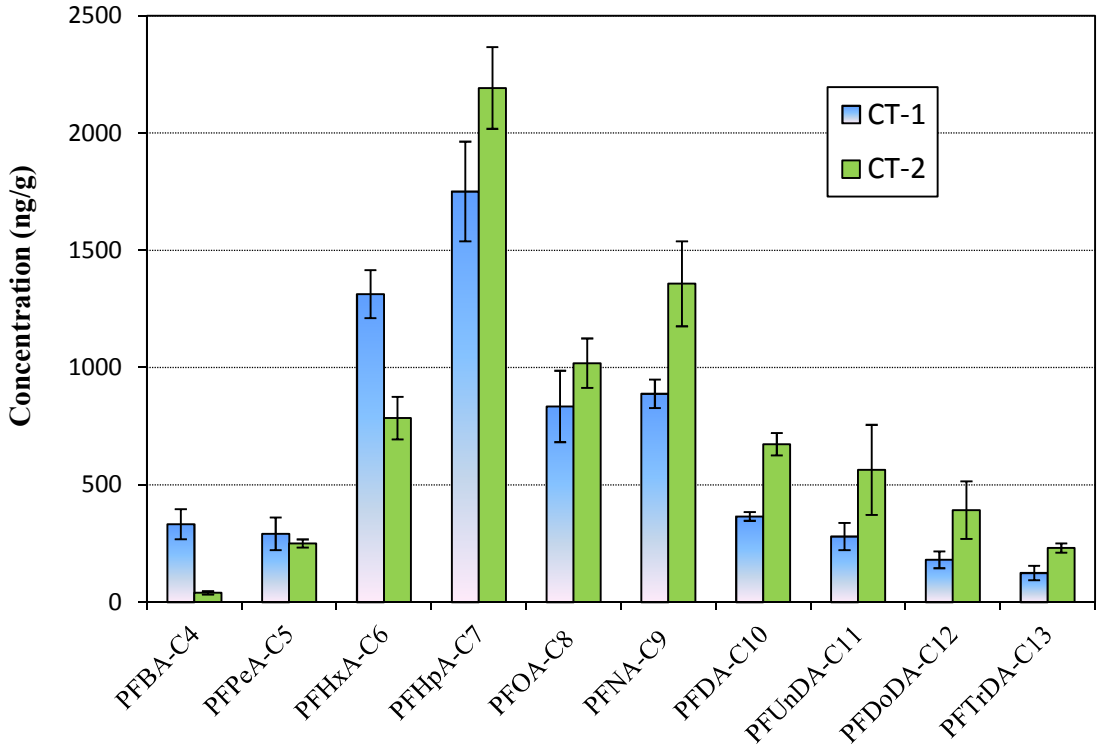

Figure 3.1. Extractable PFCA content in carpet treatment solutions CT-1 and CT-2

(The error bars represent ±1 SD; n = 4 for CT-1 and n = 6 for CT-2)

3.3 Extractable PFCA Content in Wipe Samples Taken from the Walls

The results from the wipe samples collected during Experiment 3 to evaluate overspray during the application process are presented in Table 3.2. Background wipe samples were collected from each of the four locations prior to the application of the carpet stain-protection treatment. Three of the four background samples showed no evidence of PFCAs on the walls prior to application. The fourth background sample was lost during analysis. The concentration of PFCAs measured on the wall varied significantly from location to location, ranging from 2.3 ng/cm^2 to 47.4 ng/cm^2. This exposure source is minimal when compared to the post-application concentration on the carpet of approximately 4000 ng/cm^2 of carpet area, as measured from the protectant application rate on the carpet.

Table 3.2. Results of wall wipe samples collected during Experiment 3

Wipe Samples	Wipe A, ng/cm^2	Wipe B, ng/cm^2	Wipe C, ng/cm^2	Wipe D, ng/cm^2
PFBA-C4	0.8	1.0	~~0.1~~ [b]	1.4
PFPeA-C5	0.5	0.7	~~0.0~~ [b]	1.0
PFHxA-C6	2.5	3.3	0.2	5.3
PFHpA-C7	6.9	8.6	0.4	*10.9* [b]
PFOA-C8	3.8	4.7	0.2	6.1
PFNA-C9	4.5	5.9	0.3	7.8
PFDA-C10	2.9	3.9	0.2	4.9
PFUnDA-C11	2.4	3.2	0.1	4.1
PFDoDA-C12	1.8	2.2	~~0.2~~ [b]	2.7
PFTrDA-C13	1.2	1.4	~~0.1~~ [b]	1.5
PFTeDA-C14	~~1.8~~[b]	~~1.9~~[b]	~~0.5~~ [b]	~~2.0~~
Total PFCA, ng/cm^2	29.1	36.8	2.3	47.7
Perfluoro-*n*-[1,2-13C2] decanoic acid (recovery check standard)	113%	117%	120%	121% [a]

[a] Did not pass MQO of ± 20% but is presented with the relaxed MQO.
[b] ~~nn~~ = values below quantification limit; *nn* = values above calibration range.

3.4 Extractable PFCA Content in Carpet Samples

The concentrations of individual PFCAs and total PFCA in composite carpet fiber samples for Experiments 1 through 9 are presented in Appendix A, Tables A.1 through A.9. The results of total PFCAs are presented graphically in this section in Figures 3.2 through 3.10. The composite samples represent fibers collected from each of the five sampling locations compiled into one sample and then divided into five replicate samples. The data in each graph represent the reportable data using both the QAPP standard MQOs and the relaxed MQOs from each experiment. The relaxed data are highlighted in the data tables in Appendix A. The error bars represent the variability between replicate samples and are equal to ± one standard deviation.

In the following graphs, each experiment is coded with the parameters of the experiment as presented in Table 4.3. The resulting code is presented as: Type of carpet: R = residential or C = commercial; carpet stain-protection treatment: 1 or 2; carpet cleaning machine: 1 or 2; and carpet detergent: 0 = no detergent, 1 or 2 [i.e., Experiment 1 (C-1-2-0) = Commercial carpet, CT1, CM1, and no detergent].

It was noticed that the commercial carpet (see Figures 3.2, 3.3, and 3.7) retained more PFCAs after treatment than the residential carpet did.

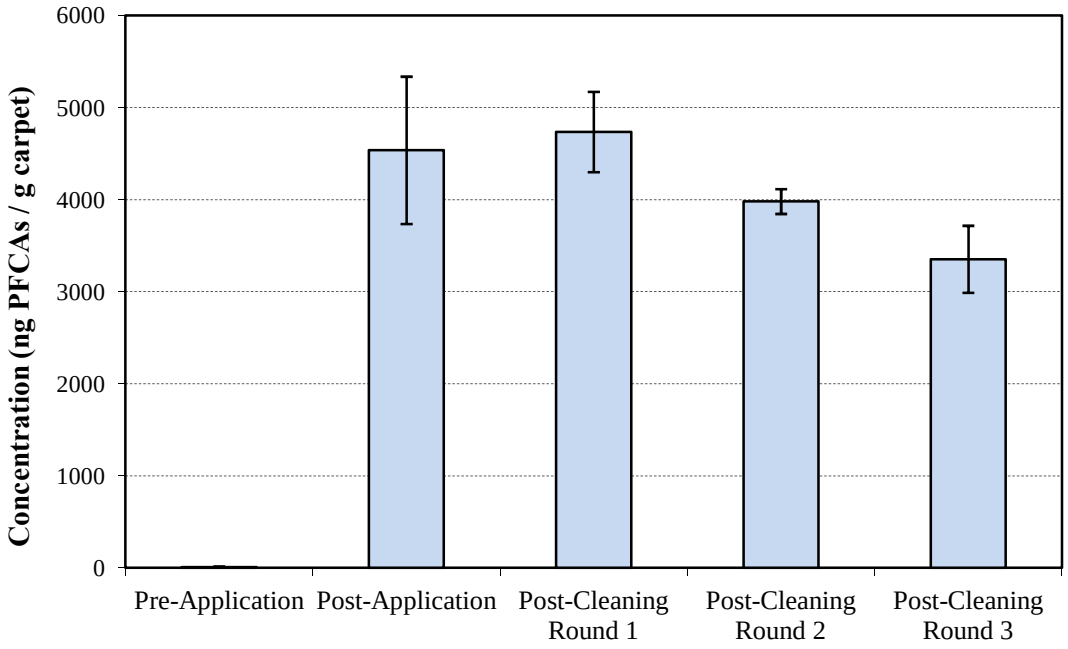

Figure 3.2. Average total PFCAs in composite carpet fiber samples for Experiment 1 (C-1-2-0)

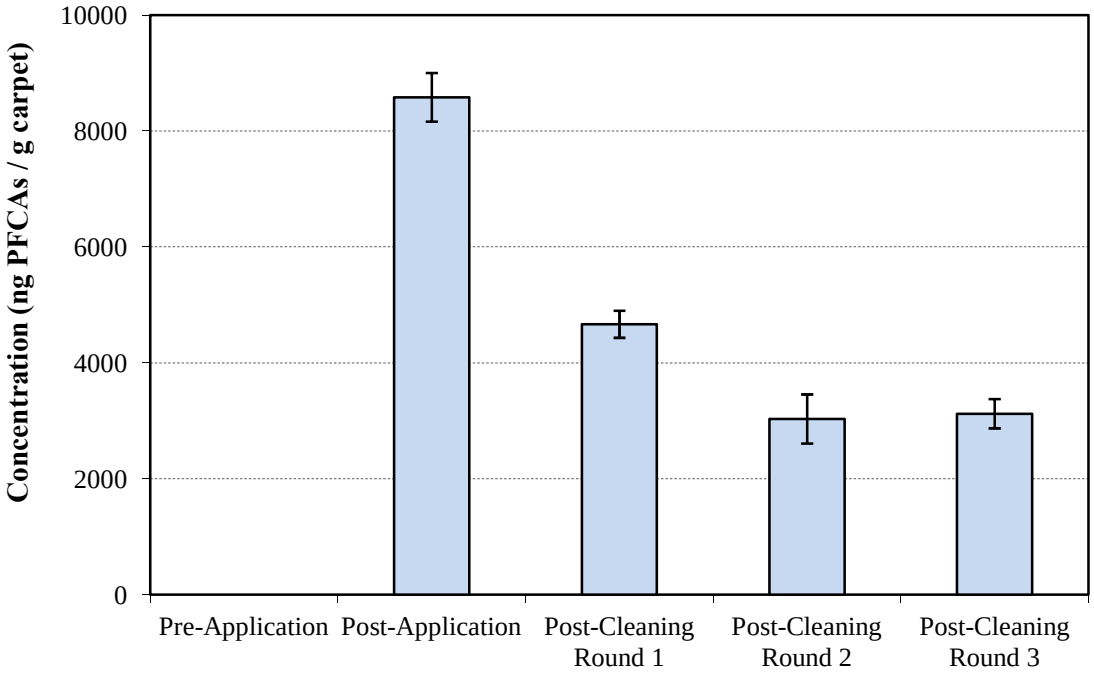

Figure 3.3. Average total PFCAs in composite carpet fiber samples for Experiment 2 (C-2-1-0)

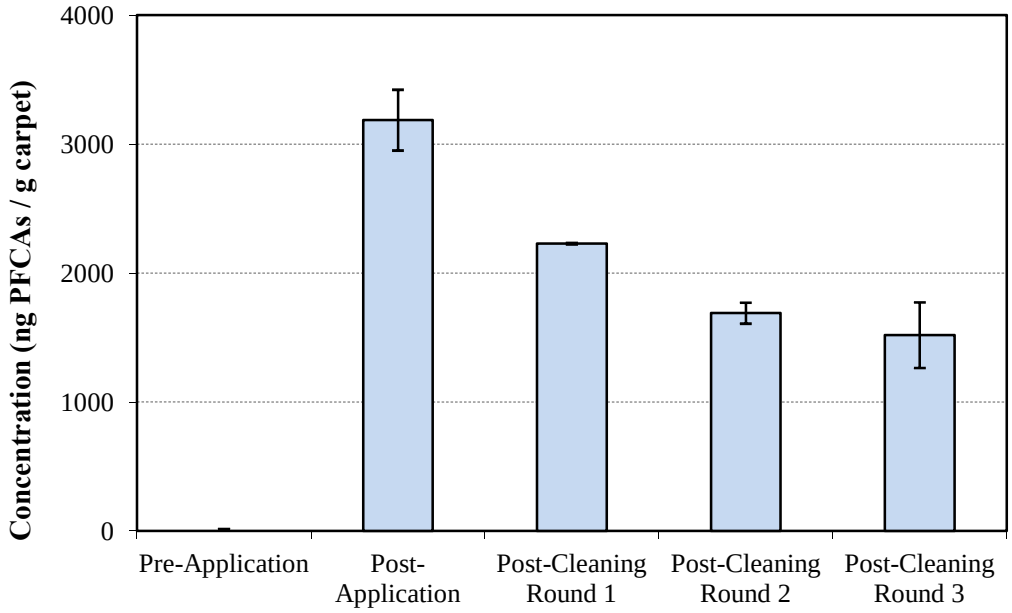

Figure 3.4. Average total PFCAs in composite carpet fiber samples for Experiment 3 (R-1-1-0)

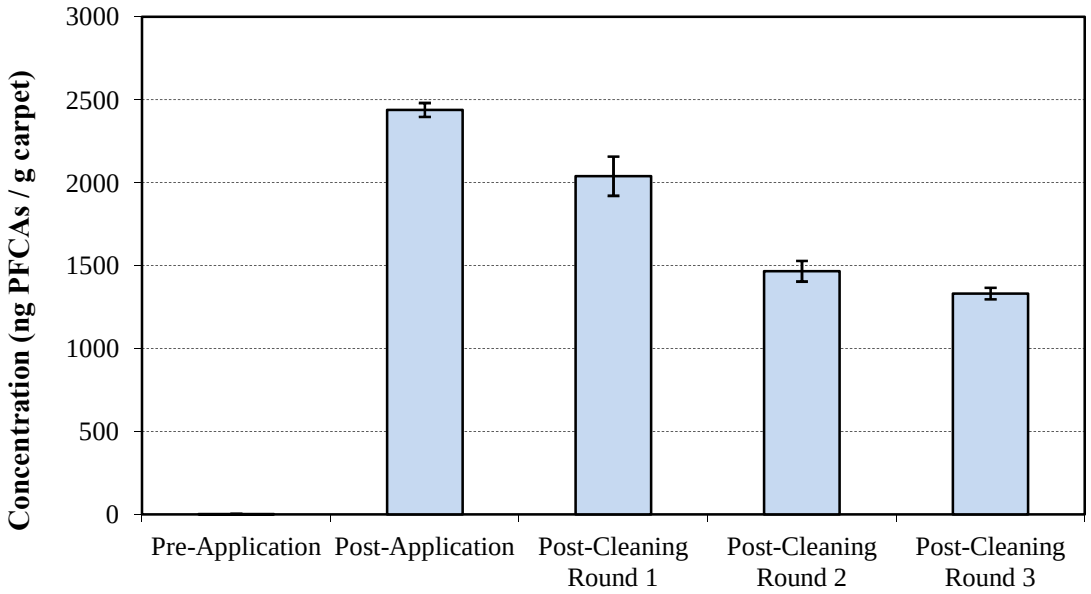

Figure 3.5. Average total PFCAs in composite carpet fiber samples for Experiment 4 (R-1-2-0)

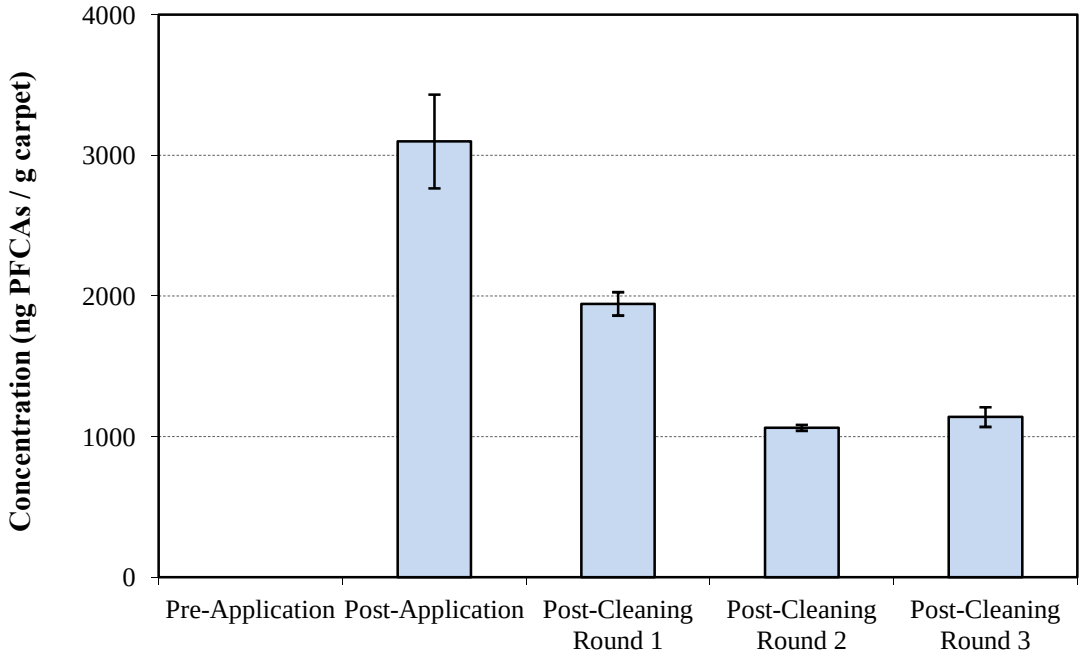

Figure 3.6. Average total PFCAs in composite carpet fiber samples for Experiment 5 (R-2-2-0)

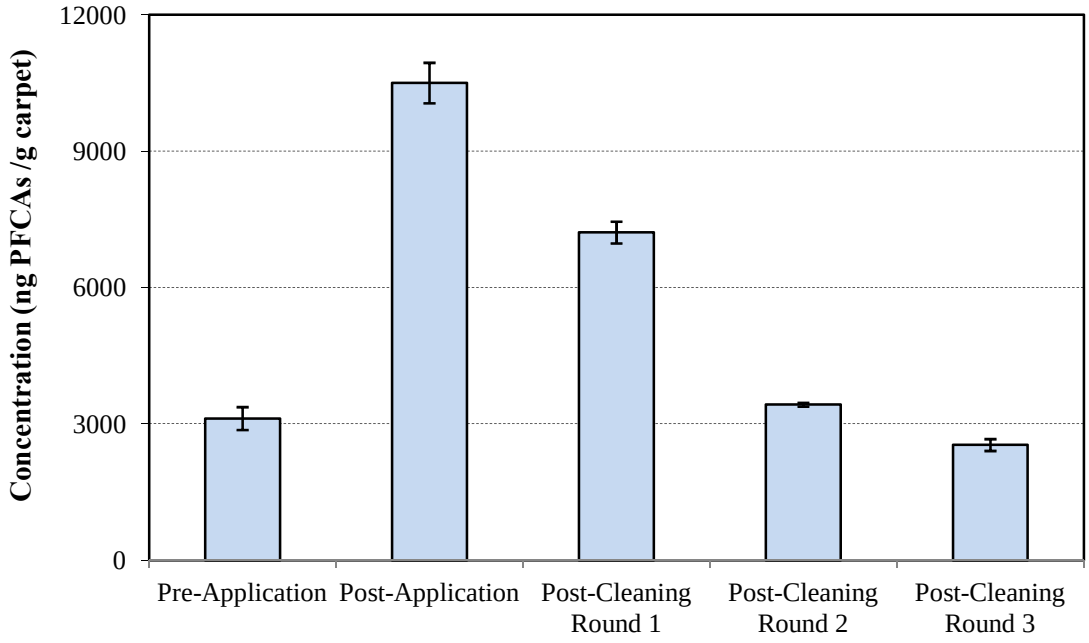

Figure 3.7. Average total PFCAs in composite carpet fiber samples for Experiment 6 (C-2-2-1)

[Re-use of carpet from Experiment 2]

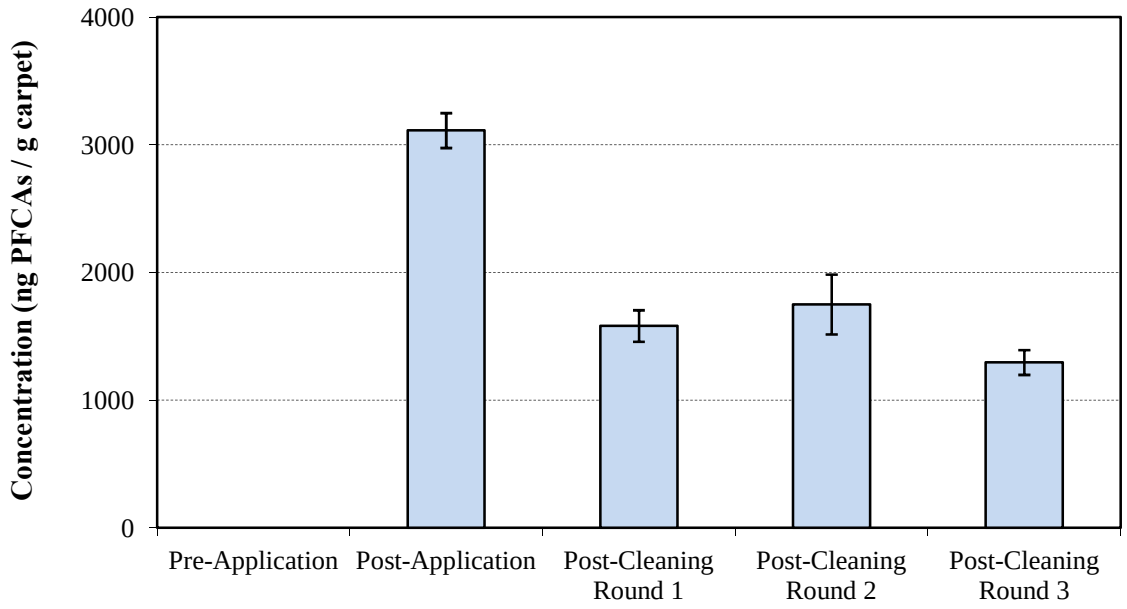

Figure 3.8. Average total PFCAs in composite carpet fiber samples for Experiment 7 (R-1-1-2)

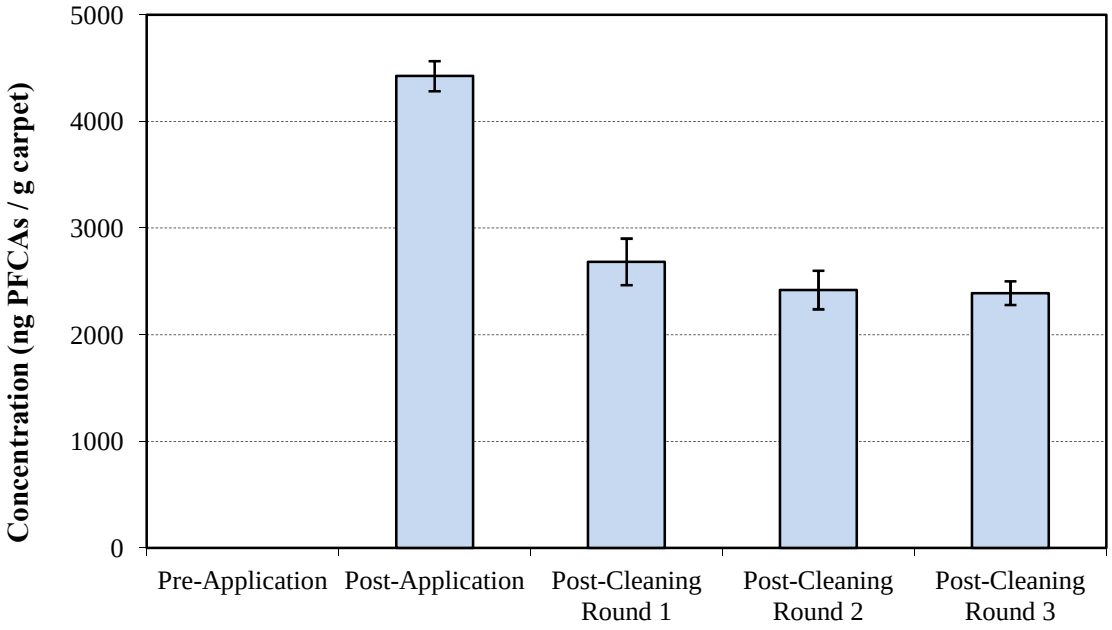

Figure 3.9. Average total PFCAs in composite carpet fiber samples for Experiment 8 (R-1-1-2), duplicate of Experiment 7

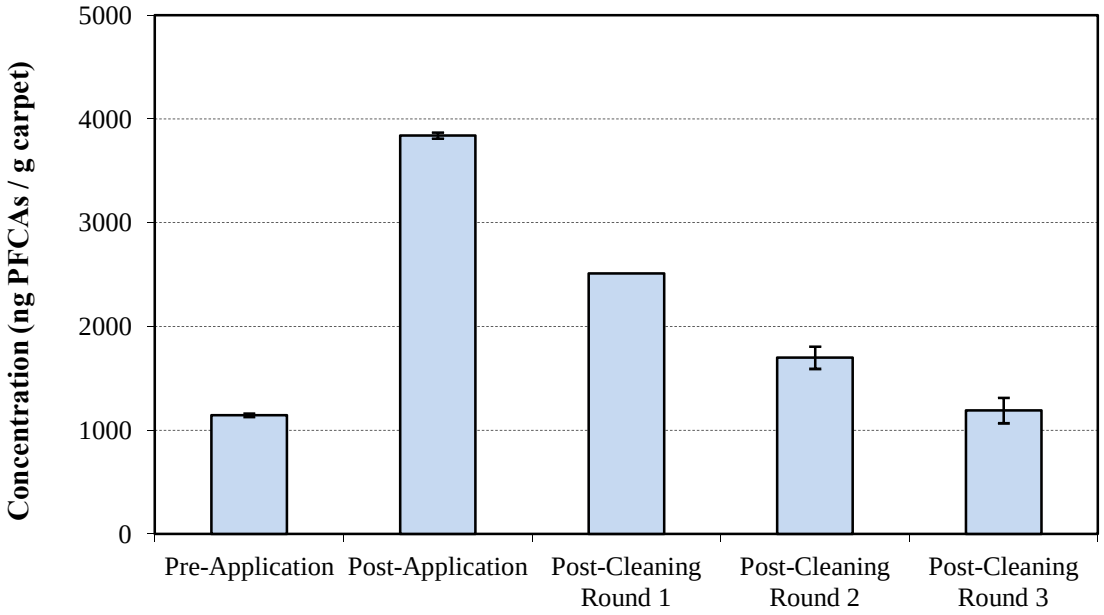

Figure 3.10. Average total PFCAs in composite carpet fiber samples for Experiment 9 (R-2-2-1)

[Re-use of carpet from Experiment 5]

3.5 Percent Removal of PFCAs by Cleaning

For each experiment, the percent removal of total PFCAs after the final cleaning was calculated by comparison of the total PFCA concentration after application of the carpet treatment to the total PFCA concentration after the third round of cleaning. These data are presented in Appendix A, Tables A1 through A9. Figure 3.11 is a graphic representation of these results.

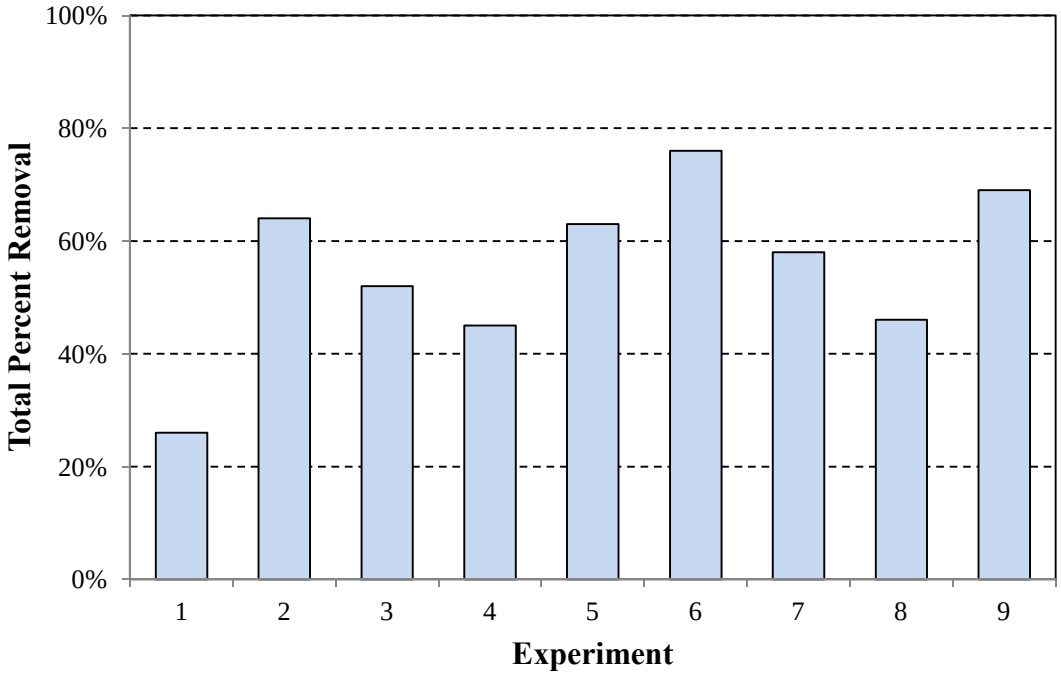

Figure 3.11. Calculated percent removal of total PFCAs for each experiment

4. Discussion

4.1 Removal Efficiency of Speciated Extractable PFCAs in Carpet Samples

A relative comparison of the post-application concentration of the speciated carpet treatment to the concentration after the third cleaning for each experiment as percent removal of individual compounds PFPeA-C5 through PFDoDA-C12 is presented in Table 4.1. PFBA-C4 and PFTrDA-C13 are not presented due to incomplete data sets. Three of the four experiments with detergent (7, 8, and 9) and two of the experiments without detergent (4 and 5) present a clear trend that the higher removal efficiency was associated with the lower molecular weight compounds rather than high molecular weight compounds. This trend is confirmed in the comparison of duplicate experiments 7 and 8. However, in experiments 1, 2, 3, and 6, the trend indicated that the removal efficiencies were more uniform. No clear conclusion about speciated removal efficiency could be determined from these data.

Table 4.1. Percent removal efficiency of speciated compounds

Experiment	1 (C120)	2 (C220)	3 (R110)	4 (R120)	5 (R220)	6 (C221)	7 (R112)	8 (R112)	9 (R221)
PFPeA-C5	29%	63%	57%	100%	NR	60%	89%	79%	84%
PFHxA-C6	29%	70%	70%	100%	89%	70%	94%	91%	91%
PFHpA-C7	28%	62%	58%	52%	76%	64%	80%	73%	81%
PFOA-C8	44%	67%	40%	27%	38%	73%	47%	43%	61%
PFNA-C9	25%	70%	38%	18%	39%	81%	13%	30%	64%
PFDA-C10	28%	64%	59%	22%	84%	81%	73%	64%	58%
PFUnDA-C11	3%	59%	44%	5%	4%	NR	4%	23%	79%
PFDoDA-C12	19%	59%	49%	14%	NR	89%	43%	20%	70%

4.2 Overall PFCA Removal Efficiency

For the nine cleaning tests conducted, the overall PFCA removal efficiency after three rounds of cleaning ranged from 26% to 76% (Table 4.2). Repeated cleanings with and without detergent did not return either carpet to its pre-application concentration of < 8 ng/g of TPFCAs. On average, each round of carpet cleaning removed approximately 20% of the total PFCAs. The overall removal efficiency of the PFCAs could be attributed to the differences in the carpet fibers of each of the products. Although there was high variability in the percent of PFCAs removed in each round of cleaning (Appendix A), certain trends were evident. In general, the first round of carpet cleaning resulted in the largest decrease in PFCAs. The decrease in PFCAs from the second to the third cleaning continued to show removal of PFCAs, albeit with less efficiency, almost indicating a leveling off of PFCA removal with each cleaning system.

Table 4.2. Composite carpet sample data summarizing average percent reduction in PFCAs by experiment

Experiment	Experimental ID	Total Removal, %
1	C-1-2-0	26%
2	C-2-2-0	64%
3	R-1-1-0, scouting	52%
4	R-1-2-0	45%
5	R-2-2-0	63%
6	C-2-2-1	76%
7 [a]	R-1-1-2	58%
8 [a]	R-1-1-2	46%
9	R-2-2-1	70%

[a] Duplicate experiments.

4.3 Comparison of Duplicate Experiments

Experiments 7 and 8 were performed using the same residential carpet, treatment, cleaning machine and detergent and represents the only duplicate experiment that was conducted. Experiment 8 had slightly higher initial PFCA concentrations than Experiment 7. Analysis for average PFCA removal following each round of cleaning showed less than 30% variance between the duplicate experiments, which was within the ± 30% deviation indicated in the relaxed MQOs for this investigation, suggesting that these data are reproducible.

4.4 Factors Affecting the Efficiency of PFCA Removal

To investigate the data more closely, average reductions in PFCAs were examined by experimental variable. These data are presented in Table 4.3 and Figure 4.1. Although it is not possible to draw statistically relevant conclusions due to the large number of variables relative to the number of experiments and the cross-correlation of the variables, some preliminary conclusions can be drawn.

A detergent, either CD-1 or CD-2, was used in Experiments 6 through 9. The detergent was used in addition to the carpet cleaning machine potentially to enhance PFCA removal. As compared to Experiments 1 through 4, the experiments that used detergent exhibited no statistically significant increases in percent removals of PFCAs after three rounds of cleaning. As compared by detergent (none vs. 1 or 2), CD-1 does appear to have removed a greater amount of PFCAs, 73% as compared to ≥50% for both CD-2 and no detergent, but percent removals for most individual experiments ranged from 45% to 76% regardless of detergent use. A larger number of replicate experiments would be needed to determine with greater certainty the effects of detergent application.

Table 4.3. Average percent reduction in PFCAs by experimental variable for composite samples

Variable	Type	Total Removal
Carpet	Residential	56%
	Commercial	55%
Treatment	CT-1	46%
	CT-2	68%
Carpet Cleaning Machine	CM-1	52%
	CM-2	57%
Detergent	CD-1	73%
	CD-2	52%
	No Detergent	50%

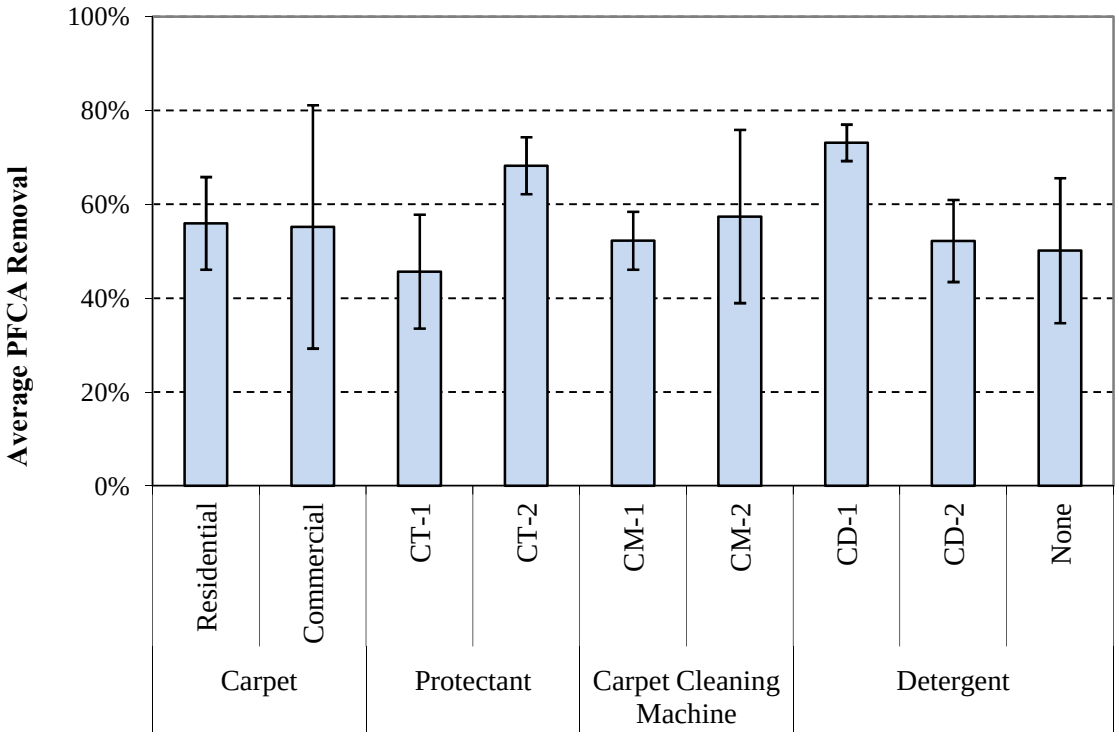

Figure 4.1. Average percent reduction in PFCAs by experimental variable for composite samples

The concentrations presented in these results represent PFCAs available via methanol extraction, which may not equal the level of PFCAs available under actual normal use conditions for carpet. For example, it has been shown that extraction of PFCAs from carpets using either water or sweat (laboratory-prepared sweat simulant), which are more common household solvents than methanol, recovered only about 10% to 65% of the PFCAs that were recovered with up to 24 hours of methanol extraction (Mawn et al., 2005). As treated carpet ages, wear and fiber degradation may also increase the availability of PFCAs through methods such as dust formation or transfer to other surfaces.

4.5 Composite vs. Individual Samples

The analysis of individual quadrant samples was conducted only to show the uniformity of the application of the carpet treatment, as presented in Section 2.7.1, Figure 2.9. Complete uniformity of application is not possible with the manual application procedure that was recommended by the manufacturer of the carpet treatment products. Thus, the discussion focused only on the data obtained from composite carpet samples.

4.6 Extractable PFCA Content Collected from Walls

The collection of wipe samples from the wall surfaces was conducted during the initial scouting experiment as a point of interest and does not concern the central discussion of PFCA removal from carpets. However, these samples do demonstrate that PFCAs are, in fact, deposited on the surface of the walls during the process of applying a carpet treatment (Table 3.2). Although the reported concentration of PFCAs on the surfaces of the walls was several orders of magnitude lower than that applied to the carpet, the presence of PFCAs on the walls after applying a carpet treatment should still be noted.

5. Conclusions and Recommendations

The efficiency of PFCA removal from treated carpet by hot water extraction and stream cleaning was evaluated under close-to-realistic conditions in a research house. On average, each carpet cleaning event removed approximately 20% of total PFCAs. At this removal efficiency, it takes three, seven, and ten rounds of cleaning to remove, respectively, 50%, 80%, and 90% of total PFCAs.

The average American home has about 1000 square feet (93 m^2) of carpet. Previous research has shown that treated carpets represent one of the largest sources of PFCAs in homes. Additionally, PFCAs are highly stable, with the potential for a long residence time indoors, and can also bind to house dust, making it difficult to remove the PFCAs from the indoor environment by any available mechanisms. The use of a carpet cleaning machine may be a risk management option for people who wish to reduce their indoor exposure to PFCAs. However, as mentioned above, this method is only modestly effective in removing PFCAs from treated carpet.

Using the household hot water machine in four experiments with the residential carpet, reductions of PFCA averaged between 45% and 58% after three rounds of cleaning. The removal efficiency using the commercial cleaning machine on the commercial carpet had the highest variability. The results from two experiments using the commercial cleaning machine with a steam extraction indicated only a 26% reduction of PFCAs for carpet treatment 1 (CT-1), but there was a reduction of 64% for carpet treatment 2 (CT-2). Differences in the carpet fibers of each product may have also contributed to the overall removal efficiency of PFCAs. No significant difference in the reduction of PFCAs was observed when detergent was added to the carpet cleaning machine. With the commercial steam cleaner using a commercial detergent, a slight improvement was noted during one experiment in which an effective removal of more than 70% of total PFCA was obtained. However, the difference was not statistically significant.

Further work is needed in the following areas to better understand human exposure to PFCAs from treated carpeting: (1) PFCA transfer mechanisms from carpets to indoor air, household surfaces, and dust; (2) the relationship between PFCA-treated carpet and inhalation exposure with regard to particle resuspension; (3) determination of the significance of dermal exposure; and (4) effective risk management measures for reducing PFCA levels in houses with PFCA-treated carpet. It is also recommended that market monitoring be continued to determine if all the carpet-treatment products on today's market are virtually PFCA-free.

Acknowledgments

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Appendix A: Data

Data for total PFCA content in carpet fibers for all the experiments are presented below. Both individual carpet fiber samples and composite fiber samples, five per type, were collected at five stages of each experiment. These stages included before the application of a carpet treatment solution, after the application of a carpet treatment solution, and after each of three rounds of cleaning with a carpet cleaning machine. The PFCA concentrations (as mean ± standard deviation) in composite carpet fiber samples for Experiments 1 through 9 are presented in Tables A1- A9. Averaged PFCA concentrations at each step for individual samples are not presented, but post-application data are given in Tables A10-A18 to demonstrate the lower data quality for individual samples and to give an indication of application uniformity, as discussed previously in Section 2.7.1.

In the following tables, each experiment is coded with the parameters of the experiment, as presented in Table 2.3. The resulting code is presented as: Type of carpet: R = residential or C = commercial; carpet stain-protection treatment: 1 or 2; carpet cleaning machine: 1 or 2; and carpet detergent: 0 = no detergent, 1, or 2 [i.e., Experiment 1 (C-1-2-0) = Commercial carpet, CT1, CM1, and no detergent]. BDL indicates that a result was below the instrument detection limit of 0.2 ng/mL, while NR indicates that data could not be reported because the results did not meet data quality requirements or were not obtained. Italicized values indicate that the results are above the highest calibration concentration of 100 ng/mL. PFTeDAwas not found above the practical quantification limit (PQL) and, therefore, was not reported.

Data highlighted in bold font did not pass one or more of the QAPP-stated MQOs, but they passed the relaxed MQOs of ± 30% established for this project.

Table A.1. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 1 (C-1-2-0).

	Pre-Application n=5	Post-Application n=3	Post-Cleaning 1 n=2	Post-Cleaning 2 n=5	Post-Cleaning 3 n=4	Total PFCA removal, %
PFBA-C4	BDL	63.4 ± 13	**55.6 ± 6.6**	52.3 ± 2.8	49.5 ± 1.8	22%
PFPeA-C5	BDL	55.1 ± 10	**82.7 ± 9.2**	43.2 ± 3.0	39.3 ± 1.1	29%
PFHxA-C6	BDL	399 ± 91	**349 ± 46**	310 ± 9.6	24 ± 17	29%
PFHpA-C7	BDL	1150 ± 201	**1530 ± 140**	*1000* ± 57	819 ± 77	28%
PFOA-C8	7.5 ± 1.0	809 ± 126	**828 ± 89**	560 ± 17	450 ± 39	44%
PFNA-C9	BDL	934 ± 118	**1030 ± 155**	864 ± 69	703 ± 89	25%
PFDA-C10	BDL	514 ± 88	**363 ± 8.9**	446 ± 37	373 ± 63	28%
PFUnDA-C11	BDL	382 ± 94	**227 ± 6.1**	413 ± 25	373 ± 42	3%
PFDoDA-C12	BDL	235 ± 66	**201 ± 10**	195 ± 8.5	190 ± 31	19%
PFTrDA-C13	**BDL**	(NR)	**71.8 ± 1.1**	91.6 ± 6.6	73.4 ± 10	(NR)
Total PFCAs	7.5 ± 1.0	4540 ± 801	**4740 ± 437**	3980 ± 135	3350 ± 364	26%

Table A.2. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 2 (C-2-2-0).

	Pre-Application n=5 [a]	Post-Application n=5	Post-Cleaning 1 n=3	Post-Cleaning 2 n=4	Post-Cleaning 3 n=4	Total PFCA removal, %
PFBA-C4	**BDL**	(NR)	(NR)	(NR)	(NR)	(NR)
PFPeA-C5	**BDL**	73.2 ± 4.8	35.4 ± 4.0	26.8 ± 2.4	27.0 ± 2.3	63%
PFHxA-C6	**BDL**	411 ± 41	158 ± 4.4	115 ± 15	122 ± 12	70%
PFHpA-C7	**BDL**	*2520* ± 90	*1340* ± 18	916 ± 99	962 ± 68	62%
PFOA-C8	**BDL**	*1480* ± 140	744 ± 68	492 ± 78	495 ± 37	67%
PFNA-C9	**BDL**	*1980* ± 113	*1060* ± 86	602 ± 85	591 ± 51	70%
PFDA-C10	**BDL**	*1090* ± 81	610 ± 47	377 ± 74	391 ± 34	64%
PFUnDA-C11	**BDL**	549 ± 29	287 ± 7.7	191 ± 31	227 ± 28	59%
PFDoDA-C12	**BDL**	488 ± 60	271 ± 6.7	207 ± 45	198 ± 21	59%
PFTrDA-C13	**BDL**	(NR)	154 ± 14	98.8 ± 8.6	105 ± 11	(NR)
Total PFCAs	**BDL**	8580 ± 471	4660 ± 234	3030 ± 424	3120 ± 252	64%

[a] Recovery standards for these samples exceeded the measurement quality objective (MQO) but data were all BDL and presented for completeness.

Table A.3. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 3 (R-1-1-0).

	Pre-Application n=3	Post-Application n=2	Post-Cleaning 1 n=3	Post-Cleaning 2 n=3 [a]	Post-Cleaning 3 n=3 [a]	Total PFCA removal, %
PFBA-C4	BDL	135 ± 29	95.5 ± 2.8	**83.6 ± 7.9**	**59.0 ± 11**	**56%**
PFPeA-C5	BDL	112 ± 15	73.2 ± 2.8	**62.1 ± 3.1**	**48.1 ± 8.9**	**57%**
PFHxA-C6	BDL	524 ± 53	309 ± 14	**189 ± 9.1**	**159 ± 27**	**70%**
PFHpA-C7	BDL	716 ± 84	483 ± 46	**342 ± 26**	**298 ± 56**	**58%**
PFOA-C8	BDL	384 ± 16	290 ± 18	**249 ± 14**	**232 ± 36**	**40%**
PFNA-C9	BDL	447 ± 23	315 ± 15	**283 ± 13**	**278 ± 48**	**38%**
PFDA-C10	BDL	257 ± 11	187 ± 15	**127 ± 5.4**	**106 ± 19**	**59%**
PFUnDA-C11	BDL	279 ± 10	225 ± 15	**159 ± 9.1**	**155 ± 25**	**44%**
PFDoDA-C12	BDL	197 ± 26	143 ± 0.92	**112 ± 6.2**	**99.8 ± 18**	**49%**
PFTrDA-C13	BDL	136 ± 15	107 ± 1.7	**82.1 ± 5.9**	**82.2 ± 9.5**	**39%**
Total PFCAs	BDL	3190 ± 236	2230 ± 6.1	**1690 ± 81**	**1520 ± 255**	**52%**

[a] Recovery standards for these samples were between 45-70% but data included for completeness.

Table A.4. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 4 (R-1-2-0).

	Pre-Application n=5	Post-Application n=4	Post-Cleaning 1 n=5	Post-Cleaning 2 n=5	Post-Cleaning 3 n=3	Total PFCA removal, %
PFBA-C4	BDL	55.1 ± 3.9	35.7 ± 4.0	BDL	BDL	100%
PFPeA-C5	BDL	53.0 ± 1.6	41.0 ± 5.5	BDL	BDL	100%
PFHxA-C6	BDL	413 ± 22	334 ± 43	BDL	BDL	100%
PFHpA-C7	0.821 ± 1.4	668 ± 24	564 ± 36	381 ± 21	319 ± 9.7	52%
PFOA-C8	BDL	349 ± 28	292 ± 16	273 ± 14	256 ± 3.6	27%
PFNA-C9	BDL	367 ± 5.3	303 ± 23	303 ± 15	302 ± 8.9	18%
PFDA-C10	BDL	177 ± 14	156 ± 14	139 ± 13	139 ± 14	22%
PFUnDA-C11	BDL	195 ± 15	168 ± 14	165 ± 12	185 ± 15	5%
PFDoDA-C12	BDL	85.8 ± 1.9	79.2 ± 8.1	75.9 ± 6.3	73.7 ± 3.2	14%
PFTrDA-C13	BDL	73.9 ± 1.6	66.6 ± 9.5	**56.4 ± 6.5**	56.7 ± 3.1	23%
Total PFCAs	0.821 ± 1.4	2440 ± 42	2040 ± 119	1470 ± 62	1330 ± 35	45%

Table A.5. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 5 (R-2-2-0).

	Pre-Application n=5	Post-Application n=5	Post-Cleaning 1 n=4	Post-Cleaning 2 n=5 [a]	Post-Cleaning 3 n=5	Total PFCA removal, %
PFBA-C4	BDL	10.8 ± 0.88	BDL	(NR)	(NR)	100% [b]
PFPeA-C5	BDL	59.2 ± 4.7	29.8 ± 1.1	(NR)	(NR)	50% [b]
PFHxA-C6	BDL	252 ± 24	143 ± 5.5	**BDL**	**26.0 ± 1.7**	**89%**
PFHpA-C7	BDL	870 ± 64	480 ± 29	**227 ± 8.1**	**213 ± 23**	**76%**
PFOA-C8	BDL	389 ± 6.9	261 ± 15	**168 ± 2.8**	**240 ± 12**	**38%**
PFNA-C9	BDL	583 ± 40	367 ± 18	**230 ± 10**	353 ± 22	39%
PFDA-C10	BDL	312 ± 48	180 ± 11	**68.7 ± 9.7**	49 ± 10	84%
PFUnDA-C11	BDL	229 ± 25	228 ± 11	**177 ± 10**	219 ± 19	4%
PFDoDA-C12	BDL	173 ± 13	171 ± 9.8	**124 ± 5.4**	(NR)	1% [b]
PFTrDA-C13	**BDL**	95.0 ± 6.0	(NR)	(NR)	(NR)	(NR)
Total PFCAs	BDL	3100 ± 333	1940 ± 83	**1060 ± 21**	1144 ± 70	63%

[a] Recovery standards for reported data only 64% but data included for completeness.

[b] Total PFCA % removal as calculated from the results from the first cleaning. All others calculated from the third cleaning.

Table A.6. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 6 (C-2-2-1). Re-use of carpet from Experiment 2.

	Pre-Application n=4 [a]	Post-Application n=5 [b]	Post-Cleaning 1 n=5	Post-Cleaning 2 n=4	Post-Cleaning 3 n=2	Total PFCA removal, %
PFBA-C4	(NR)	27.7 ± 3.4	21.4 ± 3.0	**12.7 ± 1.3**	**11.5 ± 1.5**	**59%**
PFPeA-C5	27.0 ± 2.3	84.7 ± 9.0	61.3 ± 6.7	**30.4 ± 2.1**	**33.5 ± 4.5**	**60%**
PFHxA-C6	122 ± 12	577 ± 38	384 ± 40	**200 ± 3.2**	**171 ± 22**	**70%**
PFHpA-C7	962 ± 68	*2640* ± 179	*2010* ± 176	***1200* ± 39**	**958 ± 135**	**64%**
PFOA-C8	495 ± 37	*1700* ± 116	*1190* ± 67	**595 ± 30**	**454 ± 0.14**	**73%**
PFNA-C9	591 ± 51	*2330* ± 49	*1480* ± 23	**615 ± 27**	**445 ± 15**	**81%**
PFDA-C10	391 ± 34	*1460* ± 106	*1000* ± 59	**415 ± 8.1**	**276 ± 5.9**	**81%**
PFUnDA-C11	227 ± 28	(NR)	438 ± 16	**181 ± 8.5**	**75.3 ± 6.0**	**(NR)**
PFDoDA-C12	198 ± 21	669 ± 99	377 ± 12	**123 ± 38**	**71.4 ± 0.09**	**89%**
PFTrDA-C13	105 ± 11	**380 ± 16**	243 ± 13	**50.1 ± 5.4**	**40.7 ± 4.9**	**89%**
Total PFCAs	3120 ± 252	*10450* ± 445	7210 ± 239	**3420 ± 43**	**2540 ± 130**	**76%**

[a] Pre-Application data are the results of Post-Cleaning 3 for Experiment 2.

[b] Initial post-application results are higher than previous experiments due to the re-use of the carpet from Experiment 2.

Table A.7. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 7 (R-1-1-2). Experiments 7 and 8 considered replicate tests.

	Pre-Application n=5	Post-Application n=5	Post-Cleaning 1 n=5	Post-Cleaning 2 n=5	Post-Cleaning 3 n=5	Total PFCA removal, %
PFBA-C4	BDL	85.4 ± 4.1	27.5 ± 1.0	28.8 ± 5.5	19.0 ± 1.9	79%
PFPeA-C5	BDL	79.9 ± 5.4	12.6 ± 0.47	16.7 ± 1.0	9.11 ± 1.4	91%
PFHxA-C6	BDL	537 ± 29	69.9 ± 2.1	60.9 ± 8.8	29.8 ± 1.6	95%
PFHpA-C7	BDL	809 ± 36	276 ± 18	263 ± 31	163 ± 22	81%
PFOA-C8	BDL	417 ± 27	311 ± 13	324 ± 26	220 ± 17	48%
PFNA-C9	BDL	495 ± 42	436 ± 39	516 ± 53	430 ± 32	13%
PFDA-C10	BDL	288 ± 20	142 ± 29	159 ± 23	79.0 ± 4.4	72%
PFUnDA-C11	BDL	**258 ± 17**	214 ± 32	281 ± 39	**247 ± 20**	**4%**
PFDoDA-C12	BDL	83.0 ± 6.4	47.5 ± 3.1	60.7 ± 7.2	47.6 ± 3.5	45%
PFTrDA-C13	BDL	(NR)	(NR)	62.3 ± 11	53.2 ± 5.1	(NR)
Total PFCAs	BDL	3110 ± 136	1580 ± 123	1770 ± 234	1300 ± 97	58%

Table A.8. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 8 (R-1-1-2). Experiments 7 and 8 considered replicate tests.

	Pre-Application n=5	Post-Application n=5	Post-Cleaning 1 n=5	Post-Cleaning 2 n=5	Post-Cleaning 3 n=4	Total PFCA removal, %
PFBA-C4	**BDL**	78.1 ± 3.5	30.6 ± 1.6	**20.3 ± 1.8**	**15.5 ± 0.70**	**80%**
PFPeA-C5	BDL	**63 ± 2.5**	22.9 ± 1.1	17.5 ± 1.5	13.1 ± 0.76	**79%**
PFHxA-C6	BDL	384 ± 16	76.4 ± 9.3	49.3 ± 4.5	34.2 ± 1.5	91%
PFHpA-C7	BDL	904 ± 28	343 ± 25	320 ± 21	241 ± 10	73%
PFOA-C8	BDL	548 ± 10	314 ± 21	347 ± 30	310 ± 15	43%
PFNA-C9	BDL	482 ± 32	394 ± 22	340 ± 25	340 ± 21	30%
PFDA-C10	BDL	234 ± 4.9	148 ± 15	84.8 ± 6.8	84.2 ± 3.8	64%
PFUnDA-C11	BDL	*1620* ± 91	*1240* ± 132	*1150* ± 102	*1250* ± 73	**23%**
PFDoDA-C12	BDL	63.2 ± 0.59	58.2 ± 6.9	50.4 ± 0.30	**49.9 ± 3.5**	**20%**
PFTrDA-C13	BDL	49.8 ± 6.8	59.8 ± 4.4	49.3 ± 3.6	**50.5 ± 3.6**	-1%
Total PFCAs	BDL	4430 ± 142	2680 ± 218	2370 ± 209	2390 ± 111	46%

Table A.9. Average extractable PFCAs (ng/g) and percent of original amount removed in composite carpet fiber samples at each experimental stage for Experiment 9 (R-2-2-1). Re-use of carpet from Experiment 5.

	Pre-Application n=5 [a]	Post-Application n=5	Post-Cleaning 1 n=1	Post-Cleaning 2 n=5	Post-Cleaning 3 n=3	Total PFCA removal, %
PFBA-C4	(NR)	9.29 ± 0.74	**7**	**2.84 ± 0.58**	**3.24 ± 2.7**	65%
PFPeA-C5	(NR)	56.8 ± 2.5	**34**	**20.5 ± 0.50**	**8.56 ± 0.55**	84%
PFHxA-C6	**26.0 ± 2.0**	296 ± 6.5	**98**	**45.0 ± 0.96**	**27.7 ± 0.55**	91%
PFHpA-C7	**213 ± 23**	*1060* ± 37	**624**	**415 ± 11**	199 ± 18	81%
PFOA-C8	**240 ± 12**	607 ± 26	**370**	**263 ± 60**	236 ± 8.4	61%
PFNA-C9	353 ± 22	835 ± 33	**522**	**368 ± 18**	304 ± 32	64%
PFDA-C10	49.0 ± 10	328 ± 25	**173**	**113 ± 24**	137 ± 37	58%
PFUnDA-C11	219 ± 19	377 ± 17	**314**	**241 ± 12**	193 ± 12	79%
PFDoDA-C12	(NR)	286 ± 11	**208**	**141 ± 22**	**85 ± 26**	70%
PFTrDA-C13	(NR)	(NR)	**159**	**88.9 ± 27**	**72.9 ± 20**	(NR)
Total PFCAs	1144 ± 70	3840 ± 25	**2510**	**1700 ± 107**	1190 ± 123	69%

[a] Pre-Application data are the results of Post-Cleaning 3 for Experiment 5.

Table A.10. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 1 (C-1-2-0).

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	63.8	67.5
PFPeA-C5	57.1	61.9
PFHxA-C6	324	287
PFHpA-C7	977	818
PFOA-C8	684	544
PFNA-C9	794	629
PFDA-C10	479	398
PFUnDA-C11	341	270
PFDoDA-C12	213	166
PFTrDA-C13	179	152
Total PFCAs	4110	3390

Table A.11. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 2 (C-2-2-0).

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	23.6	4.3
PFPeA-C5	94.9	31
PFHxA-C6	515	117
PFHpA-C7	*2960*	472
PFOA-C8	*1680*	274
PFNA-C9	*2310*	344
PFDA-C10	*1300*	190
PFUnDA-C11	619	109
PFDoDA-C12	472	60
PFTrDA-C13	245	44
Total PFCAs	10220	1610

Table A.12. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 3 (R-1-1-0).

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	112	27
PFPeA-C5	98.9	28
PFHxA-C6	490	134
PFHpA-C7	634	154
PFOA-C8	355	85
PFNA-C9	445	97
PFDA-C10	240	56
PFUnDA-C11	245	34
PFDoDA-C12	169	23
PFTrDA-C13	126	16
Total PFCAs	2920	640

Table A.13. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 4 (R-1-2-0).

	Post-Application n=4	Standard Deviation n=4
PFBA-C4	63.9	13
PFPeA-C5	56.9	13
PFHxA-C6	413	83
PFHpA-C7	730	115
PFOA-C8	407	45
PFNA-C9	384	68
PFDA-C10	203	15
PFUnDA-C11	200	41
PFDoDA-C12	108	18
PFTrDA-C13	85.0	11
Total PFCAs	2650	392

Table A.14. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 5 (R-2-2-0).

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	12.0	1.7
PFPeA-C5	67.1	9.8
PFHxA-C6	273	59
PFHpA-C7	906	182
PFOA-C8	421	104
PFNA-C9	564	147
PFDA-C10	336	56
PFUnDA-C11	243	53
PFDoDA-C12	192	41
PFTrDA-C13	102	22
Total PFCAs	3120	663

Table A.15. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 6 (C-2-2-1).[a]

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	37.2	11
PFPeA-C5	158	74
PFHxA-C6	672	171
PFHpA-C7	*3090*	660
PFOA-C8	*1920*	424
PFNA-C9	*2670*	602
PFDA-C10	*1660*	264
PFUnDA-C11	721	115
PFDoDA-C12	681	148
PFTrDA-C13	493	125
Total PFCAs	12100	2580

[a] Re-use of carpet from Experiment 2.

Table A.16. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 7 (R-1-1-2). Experiments 7 and 8 considered replicate tests.

	Post-Application n=4	Standard Deviation n=4
PFBA-C4	113	51
PFPeA-C5	95.2	46
PFHxA-C6	524	86
PFHpA-C7	753	141
PFOA-C8	365	69
PFNA-C9	460	91
PFDA-C10	272	47
PFUnDA-C11	293	33
PFDoDA-C12	69.5	51
PFTrDA-C13	44.2	12
Total PFCAs	2960	586

Table A.17. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 8 (R-1-1-2). Experiments 7 and 8 considered replicate tests.

	Post-Application n=5	Standard Deviation n=5
PFBA-C4	79.9	15
PFPeA-C5	63.3	15
PFHxA-C6	342	86
PFHpA-C7	674	157
PFOA-C8	416	100
PFNA-C9	471	100
PFDA-C10	253	62
PFUnDA-C11	243	43
PFDoDA-C12	61.0	13
PFTrDA-C13	49.9	10
Total PFCAs	2650	589

Table A.18. Average extractable PFCAs (ng/g) post-application in individual carpet fiber samples for Experiment 9 (R-2-2-1). [a]

	Post-Application n=4	Standard Deviation n=4
PFBA-C4	7.52	5.1
PFPeA-C5	51.7	18
PFHxA-C6	271	68
PFHpA-C7	965	269
PFOA-C8	585	169
PFNA-C9	801	234
PFDA-C10	355	88
PFUnDA-C11	369	78
PFDoDA-C12	282	67
PFTrDA-C13	136	26
Total PFCAs	3830	954

[a] Re-use of carpet from Experiment 5.