# Use of Scanning Electron Microscopy / Energy Dispersive Spectroscopy (SEM/EDS) Methods for the Analysis of Small Particles Adhering to Carpet Fiber Surfaces as a Means to Test Associations of Trace Evidence in a Way that is Independent of Manufactured Characteristics

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## Abstract

Very small particles (VSP) are ubiquitous in our environment and are virtually ignored by forensic science. These particles range in size from an order of magnitude smaller than conventional trace evidence, down to the molecular level (now routinely exploited through DNA analysis). We move about in a soup that is a combination of VSP that provides an extraordinary, largely untapped resource for forensic associations and source attribution. This project was an initial, highly successful effort to exploit VSP for one specific application.

An innovative instrumental trace evidence analysis approach was developed and tested for the recovery and quantitative SEM/EDS analysis of VSP adhering to the surfaces of carpet fibers – one of the most common types of trace evidence examined in crime laboratories.

Program goals were: (1) to develop methods to quantitatively remove VSP from carpet fibers and prepare them for SEM/EDS analysis, and (2) to exploit existing computer-assisted SEM/EDS methods to test whether the resulting VSP profiles are useful to quantitatively associate shed fibers with a source carpet. Specific program objectives were to:

- Develop suitable methods as described
- Use these methods to determine VSP profiles within source carpets
- Analyze VSP adhering to single fibers from the source carpets, and determine if their VSP profiles were consistent with an unbiased statistical sampling from the source carpet
- Explore between-item variation for VSP profiles using a broader qualitative survey of carpets

Appropriate methods were developed and used to assess within-carpet variability using VSP from three different areas on each of nine carpets. Carpet area VSP profiles were defined by a set of ten carpet fibers and the profiles of individual fibers from these areas were compared. Between-item variation was explored using a survey of VSP profiles on an additional 12 carpets.

Program goals and objectives were met. The regular occurrence of hundreds to thousands of VSP on individual carpet fibers was demonstrated. The quantity and character of VSP was sufficient to associate fibers with their carpet area of origin. The hypothesis of a strictly quantitative relationship among VSP, as measured using environmental particle profiles, was strongly rejected. These environmental particle profiles were found to be unsuitable to assess VSP variability. An alternative method was developed based on Target Particle Types (TPTs) defined by their elemental profiles as measured by computer-assisted SEM/EDS. Within-carpet and between-carpet variations showed a roughly even distribution for most TPTs and between-carpet variations showed a wide range in types and quantities of VSP.

The usefulness of VSP to link of carpet fiber evidence has been established. There is now a clearly achievable potential to use VSP for independent, quantitative testing of the common origin of carpet fibers. To unlock this potential, a set of follow-on research steps have been outlined and are ready to be undertaken.

The specific application of VSP to carpet fibers is ready for development, but the proof of principle resulting from this research is of much broader significance for the future of trace

evidence analysis. It is a breakthrough providing the impetus and direction for a fundamental change in the way that forensic trace evidence is conceptualized, analyzed and used in the criminal justice system. The results of this research are likely extendable, with minor modifications, to other trace evidence types, and are expected to contribute significantly for those types of trace evidence that have long been considered of low evidential value. Furthermore, entirely new approaches to trace evidence are enabled by exploiting VSP profiles, such as comparing different types of trace evidence with one another and comparing VSP defined by crime scene or suspect environments to that on virtually any item of physical evidence.

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## Executive Summary

Our research has demonstrated that hundreds to thousands of very small particles (VSP) cling to the surfaces of individual carpet fibers. We have developed methods to remove these particles, analyze them, and use them to link the fibers to the carpet from which they came.

This project focused on one specific trace evidence application (carpet fibers) and one specific instrumental particle analysis method (computer-assisted SEM/EDS) to explore and test a fundamentally different approach to trace evidence analysis. In doing so, a set of reasonable assumptions and choices were made that can themselves be tested and refined for this specific application.

As proof of principle our findings are a highly significant breakthrough for the future of trace evidence analysis. They demonstrate a way to remove fundamental limitations of class associations and provide the impetus and direction for fundamental change in the way forensic trace evidence is conceptualized, analyzed and used in the criminal justice system. Further research is encouraged to allow the independent, quantitative testing of common origin using populations of VSP.

Problem and Purpose

There is a fundamental limitation to the probative value of many of the most common types of trace evidence (e.g., fibers, glass, paint) because their characteristics are determined by their manufacture. As mass-produced commodities, probative value is limited to class associations. Multiple-transfer cases shatter this limitation. These are cases where a set of different trace evidence materials, found on a suspect, correspond to sources at a crime scene, and/or the reverse: where a set of trace evidence materials, found at the crime scene, correspond to suspect-related sources. The well-known case of Atlanta child murders provides an excellent example, where fibers corresponding to the trunk of Wayne Williams' car, his bedroom carpet, his bedspread, and his blanket were all found together on multiple victims. When the possibilities of correlation can be discounted, the probative value can become extraordinarily high, even when probabilities for the occurrence of individual trace evidence types are modest, or subject to inherent imprecision in their estimates. The co-occurrence of multiple events of modest frequency is the foundation for all highly probative types of physical evidence, including DNA and fingerprint identifications. The potential is also there for trace evidence.

This research is part of an effort to radically improve trace-evidence analysis, systematically addressing the fundamental limitations affecting the strength and measurement of probative value, by exploiting the very small particles (VSP) that are present on virtually every material or object. These VSP "ride piggy-back" on conventional trace evidence. They occur in complex mixtures and include a tremendous variety of particles that are acquired when manufactured materials are exposed to alternative environments. These particles reflect cumulative exposures and conditions; they will be highly characteristic of the local environment and their presence, identity and relative quantities, provide an untapped source of individuality for conventional trace evidence.

There is a tremendous potential here: every trace evidence case becomes a multiple-transfer case, with the adhering fine particles providing an independent quantitative means to test hypotheses of common origin. Consider: if *these* carpet fibers came from *that* carpet, then the multivariate occurrence of a quantitative profile of VSP, present on *that* carpet, ought to be present, subject to statistical sampling, on *these* fibers.

To unlock this potential, research is required that (1) determines which VSP have useful forensic performance characteristics, (2) develops suitable methods for detection and measurement, and (3) provides data on variation and occurrence that enable reliable statistical interpretation.

The research described in this report develops and tests this approach using the elemental analysis of populations of VSP occurring on the surface of carpet fibers. Carpets are common in indoor domestic and many commercial environments, and they are ubiquitous in automobiles. Carpet fibers are easily shed, easily transferred, easily recognized on careful examination, and easily recovered. Carpet fibers are a particularly suitable candidate for this approach: they are subject to long-term exposures, they have a tremendously large exposed surface area, and they are designed to trap small particles.

Methods for computer-assisted SEM/EDS analysis of particles in the size range of interest have been intensively developed in the past ten years for workplace and environmental monitoring. In crime laboratories, the methods have been in use since the 1980s for identification of gunshot residue particles (GSR), based on a combination of their elemental composition and morphology.

For initial investigations we used existing computer-assisted particle analysis protocols and classification criteria designed for environmental applications, as the methods are rigorously standardized and the particle classification criteria are explicitly defined. Methods for removal of particles from fibers and their transfer to SEM stubs needed to be developed.

The goals of the present research were to (1) to develop the methods needed to quantitatively remove VSP from carpet fibers and prepare them for SEM/EDS analysis, and (2) to exploit existing computer-assisted SEM/EDS methods to test whether the resulting fine particle profiles are useful to quantitatively associate shed fibers with a source carpet.

Our specific hypothesis was that carpet fibers from a source carpet will bear a measurable VSP profile that is a function of an unbiased statistical sampling of the VSP population present on the source carpet. To test this hypothesis and put the results in context, we defined four objectives:
(1) Develop methods, compatible with existing fiber analysis protocols, and using currently available crime laboratory resources, to quantitatively remove and analyze the VSP adhering to carpet fibers.
(2) Use these methods to determine within-item variation (VSP profiles within the source carpet).
(3) Analyze VSP adhering to fibers that have been shed from the source carpets, and determine if the profiles occurring on these fibers are consistent with an unbiased statistical sampling from the source carpet population.
(4) Conduct a broader qualitative survey of carpet VSP profiles to explore between-item variation.

Research Design

Carpets were selected from residences, vehicles and workplaces with an emphasis on those occurring most commonly in forensic casework: nylon and polyester fibers with trilobal or circular cross sections. Twenty-one carpets were selected and sampled. These carpets were not meant to be representative of a population, but to cover a range of settings so that a range of within- and between-carpet variation could be studied.

Fibers were cut directly from looped piles in the carpets. Carpets used for analysis of within-item variation (nine of the 21) were sampled from three areas, spaced from one to three feet apart.

A method to recover VSP from carpet fibers was developed after testing five alternative washing liquids and ten alternative methods of agitation. Completeness of particle removal was assessed by SEM imaging of fibers. The finalized method used pre-filtered reagent grade ethanol with sonication in a micro-centrifuge tube for ten minutes. This was followed by filtration to recover the particles, drying and mounting for computer-assisted SEM analysis. Process blanks were included alongside each batch of samples processed (pre-filtered ethanol sonicated for ten minutes in an empty tube and otherwise treated in an identical manner).

Standardized computer-assisted SEM/EDS analyses were performed by Gateway Analytical, LLC. Analyses were conducted on a total of 120 samples, along with 12 process blanks. Twenty-seven samples were sets of ten fibers from the nine carpets used to study within-carpet variation (one set from each area sampled). The 10-fiber sets were used to represent the population of VSP at that area of the carpet. Eighty-one individual fibers, three from each of the 27 areas, were used to represent individual shed fibers from these areas. Twelve additional carpets were sampled (ten fibers from each) and were used to represent populations of particles for those carpets.

The computer-assisted SEM/EDS analysis was performed on an Aspex Corporation PSEM Explorer SEM-EDS system using the Automated Feature Analysis (AFA) program within the Aspex Corporation Perception software. We adopted an existing methodology with a mix of rarely encountered and commonly occurring elements among a set of 28. The number of elements (among those detectable by EDS) is not restricted by the SEM/EDS or by the software and the assortment of elements can be changed. The number of elements selected affects the overlap of x-ray lines, but ambiguities are resolved by the x-ray detection software using known correlated x-ray emissions for the specific elements. The specific operational settings of the software restricted detection to 4000 particles and 28 elements: Sodium (Na), Magnesium (Mg), Aluminum (Al), Silicon (Si), Sulfur (S), Chlorine (Cl), Calcium (Ca), Titanium (Ti), Chromium (Cr), Manganese (Mn), Iron (Fe), Nickel (Ni), Copper (Cu), Zinc (Zn), Bromine (Br), Strontium (Sr), Zirconium (Zr), Silver (Ag), Tin (Sn), Antimony (Sb), Barium (Ba), Tungsten (W), Gold (Au) and Mercury (Hg).

Environmental Particle Groups were determined following pre-defined classification rules based on percentages of total x-ray counts for specified elements. Ten alternative criteria were applied in sequence until a criterion was met. The specific criteria specified up to four elements. Most were based on the presence of one or two elements; with x-ray count thresholds as low as 10%. Frequencies of Environmental Particle Groups were determined for each of the 120 sample

analyses and 12 sample process blanks. Within-item variation was assessed using maximum-likelihood estimation and chi-square testing.

As an alternative to Environmental Particle Groups, Target Particle Types (TPTs) were defined based on frequently occurring elemental profiles (> 1%) in the 27 10-fiber sets used to study within-carpet variation. A set of 68 mutually-exclusive TPTs were defined after grouping of closely related and overlapping elemental profile classes. Frequencies of each of the TPTs were then determined for each of the 120 sample analyses and 12 sample process blanks. Within- and between-item variation was assessed by analysis and comparison of the TPTs, including the presence or absence of TPTs and their relative abundance in samples (proportionality and rank).

Findings and Conclusions

All of the findings in this project are based on the analysis of carpets covering a limited range of characteristics (primarily trilobal, nylon fibers) and present in a limited set of environments. Accordingly, the conclusions made from these findings would apply only to these types of carpets. They serve as a model and as an initial study. As with any research, the initial efforts define a starting point. Further work on other carpet types can then be considered in reference to these data and the findings can then be either generalized or more definitively qualified.

There were nine specific findings:

1. Very small particles (VSP) present on the surfaces of individual carpet fibers can be recovered nearly completely and prepared for computer-assisted SEM/EDS analysis by extraction with reagent ethanol and filtration onto polycarbonate filters.

The method is effective, employs non-toxic materials, and is easy to apply. Practitioner review of the method established that it could be easily incorporated as a preliminary washing step prior to polarized light microscopy. With the development of this method, the first of the project objectives was achieved: to develop methods, compatible with existing fiber analysis protocols, and using currently available crime laboratory resources, to quantitatively remove and analyze the VSP adhering to carpet fibers.

2. Hundreds to thousands of VSP routinely occur on the surface of individual carpet fibers.

VSP isolated from individual fibers and analyzed by computer-assisted SEM/EDS varied in number from less than a hundred to greater than 4000 (the maximum number examined). After allowing for particles occurring in process blanks, there is an average (n = 81) of over 500 ethanol-insoluble VSP on the surface of a single carpet fiber. The confirmed presence of VSP in this quantity on carpet fibers enables research on the best means to analyze and interpret them, unlocking their extraordinary potential to enhance probative value and independently test hypotheses of common origin.

3. VSP on individual carpet fibers, when classified using criteria developed for environmental applications, cannot be considered as an unbiased statistical sampling of a VSP population on the carpet itself.

The hypothesis that the measured Environmental Particle Profiles on individual fibers represent an unbiased statistical sampling of the Environmental Particle Profiles on sets of ten fibers was strongly rejected. The specific program goal was met: to exploit existing computer-assisted SEM/EDS methods to test whether the resulting VSP profiles are useful to quantitatively associate shed fibers with a source carpet.

4. Environmental Particle Groupings have significant weaknesses for investigation of VSP variation and were found to be unsuitable.

The principal weaknesses are that many particle compositions have ambiguous group classifications and are based on only a small percentage of the particle's composition. The classification ambiguities in the environmental classification scheme are resolved by sequentially applying the classification criteria. Once a particle fits a class it is removed from consideration in the remaining classes. This approach is a reasonable one when targeting specific types of particles, with a defined priority, in a given environmental problem. In practice, an initial classification scheme is used essentially as a presumptive or screening test, to identify candidate particles that might meet a targeted particle type of environmental concern. Images and x-ray spectra of these individual particles, or a sampling of them, can then be examined as part of "confirmatory testing." Looking at only a small portion of a particle's composition is also reasonable for the environmental task; if particles don't meet the presumptive screening criteria, they are conclusively *not* of interest and are disregarded. For the investigation of VSP variation, however, the ambiguously defined, overlapping environmental classes are poorly suited.

5. Clearly defined, mutually exclusive Target Particle Types (TPTs), based on the commonly occurring elemental profile groupings within samples, were found to be useful for study of the within- and between-carpet variations in VSP occurrence.

The deficiencies of the Environmental Particle Groupings were overcome by defining Elemental Profile Groupings based on the four highest x-ray counts of the 28 elements detected by the computer-assisted SEM/EDS procedure. Groupings occurring at the highest levels in 27 carpet area samples were used to define 68 mutually exclusive TPTs which were then used to study within- and between-carpet variation.

6. Among different areas of the same carpet, most TPTs showed comparable occurrence, or comparable absence. Some TPTs were localized.

Qualitatively, within-carpet variation for the TPTs was usually very low (~ 75% of the time), but occasionally ranged to very high for some TPTs and some areas (~ 13% of the time). This indicates an underlying, roughly even distribution of most particle types, together with occasional localized particle types. The findings do not support a single homogenized VSP distribution on fibers from a carpet, but they conclusively demonstrate that there are many particle types that occur with a sufficiently even distribution to be a useful for characterization.

The finding of some highly localized variability is not an unreasonable or unexpected result, given the likelihood of local carpet exposures to soiling or staining.

7. Different carpets vary widely in the TPTs and quantities of VSP adhering to their fiber surfaces.

The TPTs used to assess VSP variation among different carpets differed in their occurrence and discrimination potential. Eight of the TPTs occurred at high levels in only one to a few of the 21 carpets studied and another four TPTs occurred at moderate to high levels in several carpets, but at very low levels in most. Three other TPTs occurred over a wide range of levels across the full set of carpets. These 15 TPTs, in particular, are highly discriminating among the set of carpets.

Considering the profiles of VSP observed in each of the carpets, 12 of the 21 carpets are easily distinguished from one another based on qualitative differences and large differences in the frequencies of specific TPTs. The remaining eight carpets show a broad range of frequencies among five specific TPTs. These findings provide a compelling demonstration of the potential of VSP to discriminate among areas from different carpets.

8. When sufficient particles are recovered, individual fibers show highly characteristic patterns of TPTs that closely correspond to those from their originating carpet area.

Very low particle totals were found on some individual fibers, but there was an average of over 700 per fiber. With few exceptions, when totals were over 1000, the TPT occurrences from individual fibers closely followed those from their originating area. The patterns were highly characteristic and showed both qualitative similarity, and similarities in rank and proportion. With lower totals (e.g. 300 to 800 particles), many fibers still showed reasonable similarities with their originating area.

TPTs allowed meaningful comparisons of the occurrences of VSP on individual carpet fibers. For the present methods, a quantitative relationship among the full set of TPTs was not observed. However, the frequent occurrence of similarities in rank and proportionality, in separate experiments using multiple carpets and multiple areas within each carpet, establishes the proof of principle: VSP profiles on shed fibers can be measured, and they regularly correspond to reference samples taken from the area of the source carpet from which they came.

9. Using a set of TPTs, VSP adhering to the surface of individual carpet fibers can be recovered, analyzed by computer-assisted SEM/EDS and used to associate these fibers with the carpet and carpet area from which they came.

This finding is supported by the overall program results, including:

- Development and use of a practical method for the recovery of VSP from individual carpet fibers
- Demonstration of the regular occurrence of VSP on individual carpet fibers, in quantity and character sufficient to associate them with their carpet area of origin
- Development of a method suitable for the study of VSP variation, using a computer-assisted SEM/EDS analysis method based on TPTs

- Establishing that within-carpet variations show a roughly even distribution for most TPTs
- Establishing that between-carpet variations show a wide range in types and quantities of VSP, as demonstrated by TPT profiles

Implications for Policy and Practice

1. The usefulness of VSP to remove fundamental limitations to the probative value of carpet fiber evidence has been demonstrated, providing the impetus and direction for fundamental change in the way that forensic trace evidence is conceptualized, analyzed and used in the criminal justice system.

2. The results of this research are likely extendable, with minor modifications, to other trace evidence types, and are expected to contribute significantly for those types of trace evidence that are have long been considered of low evidential value.

3. An entirely new approach to trace evidence is enabled: comparing different types of trace evidence with one another by way of their adhering VSP.

4. An additional, high priority use for existing crime laboratory SEM/EDS analytical capabilities and related practitioner skills can now be anticipated, guiding the allocation of laboratory resources.

5. A need can be anticipated for policies and practices for evidence collection and processing of crime scenes that are sensitive to requirements for the preservation and analysis of VSP.

Implications for Further Research

The establishment of useful VSP profiles on the surface of individual carpet fibers provides both the impetus and direction for follow-on research. This research can be broadly divided as (1) that focused on developing the specific application of computer-assisted SEM/EDS analysis of VSP on carpet fibers, and (2) that focused on expanding the VSP approach to other applications.

For the specific carpet fiber application, proof of principle has been established with the development of practical methodologies, establishment of workable within-item variability and the measurement of corresponding profiles on individual fibers. The focus for this application now shifts to direct follow-on steps, the most important of which are:

- determining which TPTs have the best forensic performance characteristics for carpets
- rigorously measuring within and between variability for these TPTs
- optimizing the analytical protocol
- determining how susceptible shed fibers are to contamination and loss of VSP profiles
- development and validation of quantitative methods for the testing the origin of carpet fibers using VSP

For expansion of the VSP approach, additional alternative research directions are

- application to other trace evidence types

- application of other instrumental analysis methods
- the use of VSP to associate an environment with an object
- more general development and validation of quantitative methods for the use and interpretation of VSP

## I. Introduction

There is a fundamental limitation to probative value of many of the most common types of trace evidence (e.g., fibers, glass, paint) because their characteristics are determined by their manufacture. As mass-produced commodities, probative value is limited to class associations. This has long been appreciated [1-4], and most recently emphasized in the summary assessments in the NRC report [5]. Furthermore, determination of the evidential value of these class associations is extraordinarily problematic. Surveys conducted to determine frequencies of random occurrence of alternative class characteristics are an excellent foundation for expert opinion, but do not allow for quantitative interpretations due to ill-defined populations, the lack of a foundation for randomness within a population, changes in manufacturing practices over time, and variations among analytical methods.[2,3,6] Absent multiple transfers and exceptional circumstances,[7,8] probative value remains difficult to determine and limited by the possibility, or the suggestion of the possibility, that the evidence came from an alternative mass-produced item.

Multiple-transfer cases shatter this limitation. These are cases where a set of different trace evidence materials, found on a suspect, correspond to sources at a crime scene, and/or the reverse: where a set of trace evidence materials, found at the crime scene, correspond to suspect-related sources. When the possibilities of correlation can be discounted, the probative value can become extraordinarily high, even when probabilities for the occurrence of individual trace types are modest, or subject to inherent imprecision in their estimates. The co-occurrence of multiple events of modest frequency is the foundation for all highly probative types of physical evidence, including DNA and fingerprint identifications, and is an inherent aspect of some types of trace evidence, including the comparison of multi-layered structural paints and soil.

This research is part of an effort to radically improve trace-evidence analysis, systematically addressing the fundamental limitations affecting the strength and measurement of probative value, by exploiting the very small particles (VSP) that are present on virtually every material object.[6]

Four founding principles guide this effort:

1. Mixtures of particles have a great potential to proceed systematically toward individualization by providing enhanced probative value via their joint occurrence, with individual particle types occurring at modest, estimable frequencies with testable correlations.

2. Particles are always present in mixtures.

3. As we consider very small particles (VSP), their abundance increases, along with the complexity of the mixture.

4. VSP occurring on the surfaces of commonly used trace evidence types can be recovered, identified, and quantified leading to independent testing of the hypotheses of common origin.

The first principle has already been discussed, with specific reference to DNA, fingerprints and multiple-transfer evidence as precedence.

Given the value of mixtures, the second principle guides us to broaden our perspective and recognize that mixtures are always there. The traditional focus of forensic particle trace evidence work is on one of a small set of particle types, such as fibers, glass, paint, or hair. Target particles such as these are defined by casework circumstances, and corresponding particles are sought as evidence of transfer and contact.[9] The significance of these particles is that (1) their transfer can be reasonably predicted, based on a hypothesis of contact and (2) they can be efficiently detected among the mixtures of particles that are always present. Other co-occurring particles, smaller, or without a discrete crime scene or suspect source, are largely ignored as "noise."

The third principle guides us to focus on smaller particles. As we consider them, we have more particles, and the mixture becomes more complex. There is a nearly ten-thousand-fold dimensional gap between conventional trace evidence types and those routinely recovered and analyzed by conventional DNA analysis. The biggest of these are those seen by higher power light and electron microscopy – the respirable or near respirable dusts, which are traditionally ignored in forensic investigations, with the notable exception of gunshot residue (GSR).[10]

The fourth principle states that we can recover, identify and quantify and make practical use of VSP that adhere to the surfaces of conventional trace evidence particles. These VSP "ride piggy-back" on conventional trace evidence. They occur in complex mixtures and include a tremendous variety of particles that are acquired when manufactured materials are exposed to alternative environments. These particles reflect cumulative exposures and conditions; they will be highly characteristic of the local environment [11] and their presence, identity and relative quantities, provide an untapped source of individuality for conventional trace evidence. There is a tremendous potential here: every trace evidence case becomes a multiple-transfer case, with the adhering fine particles providing an independent quantitative means to test hypotheses of common origin. Consider: if *these* carpet fibers came from *that* carpet, then the multivariate occurrence of a quantitative profile of fine particles, present on *that* carpet, ought to be present, subject to statistical sampling, on *these* fibers.

To unlock this potential, research is required that (1) determines which VSP have useful forensic performance characteristics, (2) develops suitable methods for detection and measurement, and (3) provides data on variation and occurrence that enables reliable statistical interpretation.

The research described in this report develops and tests this approach using the elemental analysis of populations of VSP occurring on the surface of carpet fibers. Carpets are common in indoor domestic and many commercial environments, and they are ubiquitous in automobiles. Carpet fibers are easily shed, easily transferred, easily recognized on careful examination, and easily recovered. The transfer and persistence of fibers have received the most attention of any trace evidence type,[12] and methods for the forensic analysis of fibers are well developed,[13] including national consensus guidelines for morphology, optical properties, composition, and dyes/pigmentation,[14] as well as for examiner training.[15] Overall, fibers are a mature form of trace evidence that has been exploited frequently as associative evidence. Carpet fibers are also

the most likely candidates, among conventional trace evidence types, to be suitable for this approach: they are subject to long-term exposures, they have a tremendously large exposed surface area, and they are designed to trap small particles. Attempts to clean are seldom thorough, and they have the likely effect of homogenizing the "dust profile."

Methods for computer-assisted SEM/EDS analysis of particles in the size range of interest have been intensively developed in the past ten years for workplace and environmental monitoring,[16-18] and have become increasingly efficient and increasingly focused on particle identification, rather than characterization as methods of chemical imaging have developed.[19,20] In crime laboratories, the methods have been in use since the 1980s (first manually, with progressively more computer assistance) for targeted identification of gunshot residue particles (GSR), based on a combination of their elemental composition and morphology.[10, 21-24] The summary of the FBI Laboratory's Gunshot Residue Symposium[23] provides an excellent sense for the extent of activity in this area. The future of these computer-assisted combinations of chemical and microscopical analysis, as applied to forensic trace evidence analysis, has been foreshadowed by Roux.[25] It is a good fit, with respect to existing laboratory skills and utilization of existing equipment, to extend computer-assisted SEM/EDS analyses to fine particles adhering to the surfaces of conventional trace evidence materials.

For initial investigations, it was reasonable to use existing computer-assisted SEM/EDS particle analysis protocols and classification criteria, even though they were designed for environmental applications, as the methods are rigorously standardized and the particle classification criteria are explicitly defined.[18] For efficient analysis of fine particles from carpet fibers using computer-assisted SEM/EDS, methods for particle removal and transfer to SEM stubs must be developed.

The goals of the present research were to (1) to develop the methods needed to quantitatively remove VSP from carpet fibers and prepare them for SEM/EDS analysis, and (2) to exploit existing computer-assisted SEM/EDS methods to test whether the resulting fine particle profiles are useful to quantitatively associate shed fibers with a source carpet.

Our specific hypothesis was that carpet fibers from a source carpet will bear a measurable fine particle profile that is a function of an unbiased statistical sampling of the fine particle population present on the source carpet. To test this hypothesis and put the results in context, we defined four objectives:

(1) Develop methods, compatible with existing fiber analysis protocols, and using currently available crime laboratory resources, to quantitatively remove and analyze VSP adhering to carpet fibers.
(2) Use these methods to determine within-item variation (VSP profiles within the source carpet).
(3) Analyze VSP adhering to fibers that have been shed from the source carpets, and determine if the profiles occurring on these fibers are consistent with an unbiased statistical sampling from the source carpet population.
(4) Conduct a broader qualitative survey of carpet particle profiles to explore between-item variation.

## II. Methods

A. Carpet Sample Selection

Carpets were selected for three purposes:

Development of protocols for recovery of particles from fiber surfaces
Analysis of within-carpet variation
Survey of a broader set of carpets

*1. Carpet Selection for Development of Protocols for Recovery of Particles from Fiber Surfaces*

Practitioner review of proposed generic fiber types for development of particle recovery protocols revealed an overwhelmingly dominant incidence of Nylon fibers among carpet fibers occurring in casework (75% or more), and an overwhelmingly dominant incidence of trilobal cross-sections (85 to 90%). Trilobal cross section designs are also specifically meant to trap debris, and thus provide an appropriate "worst case" medium for development of particle recovery methods. Accordingly, trilobal nylon carpet fibers were chosen for the testing of particle recovery protocols. Beige, $TiO_2$-delustered trilobal nylon carpet fibers (Figure 1) were collected from the bottom step of the household entry way stairs from the residence of one of the SFI staff.[1]

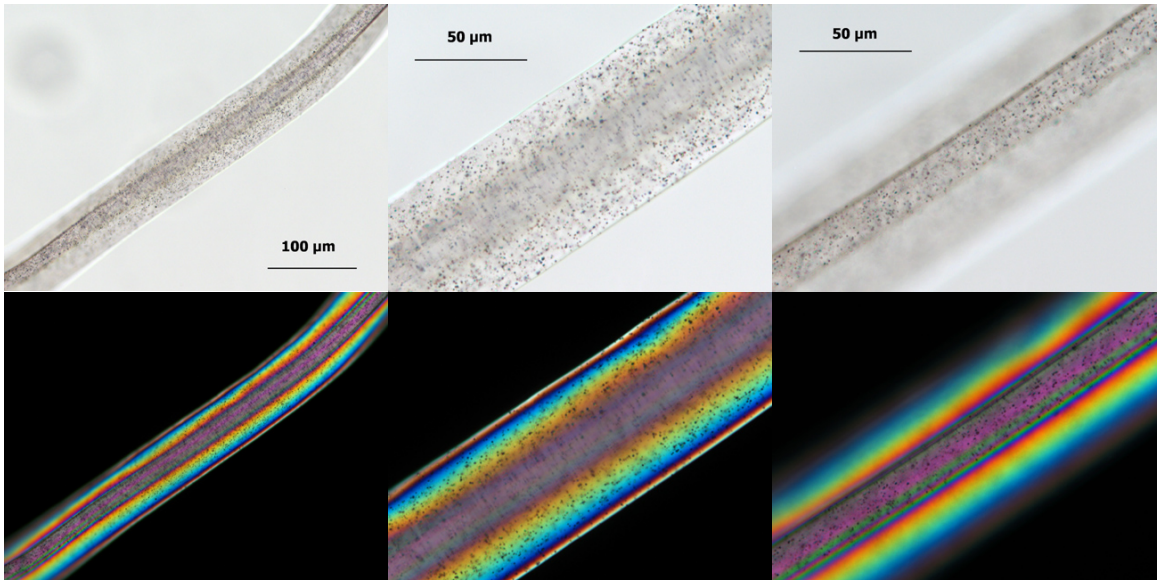


Figure 1. Polarized light microscopy of a representative trilobal Nylon fiber used for development of particle recovery protocols shown at two different magnifications with plane polarized light (above) and in-between crossed polars (below). The photomicrographs in the right column have the focus raised to illustrate the fiber's trilobal cross section. The mounting medium is $n_D$ 1.570.

[1] 13539 Lavender Mist Lane, Centreville, VA, also designated as Carpet A.

*2. Carpet Selection for Analysis of Within-Carpet Variation*
To cover a reasonable breadth of settings, nine carpets were used for the study of within-carpet variation. Represented were three residential carpets, three automobile carpets and three carpets from workplace settings. Carpets in SFI staff residences, vehicles and family workplace settings were selected for convenience. These carpets were not meant to be representative of a population, but to cover a range of settings so that a range of within-carpet variation could be studied. Fiber types and cross-sections were determined by polarized light microscopy[14,15] and were reviewed by practitioners for reasonable representation of types encountered in casework. Of these nine carpets, six were composed of nylon fibers (cross sections: five trilobal, one square with hollow filaments) and three were composed of polyester fibers (cross sections: two circular, one trilobal). Table 1 provides a listing of these carpets. Description and documentation of the carpet locations and fiber types is given in *Appendix A. Carpet Sampling for Within-Item Variation.*

*3. Carpet Selection for the Broader Survey of Carpets*
Twelve additional carpets were selected to allow better understanding of between-item variation and to serve as a foundation for the design of follow-on studies. Practitioner input was that fiber analyses related to commercial or workplace carpets are much less frequently encountered in forensic casework, compared to automobile and residential carpets. Their assessment was also that that fibers encountered in casework are fairly evenly split between residential and vehicle carpets, perhaps with slightly more residential carpets. Accordingly, we adjusted the scope of the types of carpet fibers considered to include round cross sections and the broader survey of carpets was limited to residential and vehicular carpets (seven residential and five vehicular). For convenience, carpets were selected from the residences and vehicles of SFI staff, relatives and neighbors. Again, these carpets were not meant to be representative of a population, but to cover a broader range of settings so that a range of between-carpet variation could be appreciated. Of these 12 carpets, 11 were composed of trilobal nylon fibers and one was composed of very dark fibers of circular cross section. Table 2 provides a listing of these carpets. Description and documentation of the carpet locations and fiber types is given in *Appendix B. Additional Carpet Sampling for Between-Item Variation.*

B. Sampling of Fibers from Carpets
Sampling fibers by contact was initially considered, similar to the experiments conducted by Roux et al.[26] This method was rejected after practitioner review and replaced by direct cutting of looped piles from the carpets. Direct cutting was the method used by practitioners in casework, having been adopted to ensure that fibers were actually from the carpet source, as opposed to loose fibers, that could be alleged to have come from another source. Lengths of cut fibers were not individually measured, but ranged from approximately 10 to 15mm in long.

Carpets used for analysis of within-item variation were sampled from three areas, spaced from one to three feet apart. Homogenized dust samples (by vaccum) from each of the carpet locations will also be taken for reference purposes. Collection locations for each of the carpet samples are described and illustrated in *Appendix A. Carpet Sampling for Within-Item Variation* and *Appendix B. Additional Carpet Sampling for Between-Item Variation.*

Table 1. Carpets Selected for Analysis of Within-Carpet Variation[2]

| Carpet Designation | Fiber Composition | Fiber Cross Section | Type of Location | Description |
|---|---|---|---|---|
| A | Nylon | Trilobal | Residence | Base of the stairs near front door |
| BH | Nylon | Trilobal | Residence | Basement hallway |
| PH | Polyester | Trilobal | Residence | Top of stairs near front door |
| BV | Polyester | Round | Vehicle | 2010 Toyota RAV4 driver side footwell |
| F | Polyester | Round | Vehicle | 2002 Ford Focus trunk area |
| PV | Nylon | Trilobal | Vehicle | 2005 Ford Taurus passenger side footwell |
| MT | Nylon | Square Hollow Filament | Workplace | Middle school lecture hall |
| PL | Nylon | Trilobal | Workplace | Office building second floor hallway |
| W | Nylon | Trilobal | Workplace | Office building hallway near entrance |

[2] Description and documentation of the carpet locations and fiber types is given in *Appendix A. Carpet Sampling for Within-Item Variation.*

Table 2. Additional Carpets Selected for Analysis of Between-Carpet Variation[3]

| Carpet Designation | Fiber Composition | Fiber Cross Section | Type of Location | Description |
|---|---|---|---|---|
| R1 | Nylon | Trilobal | Residence | Living room |
| R2 | Nylon | Trilobal | Residence | Basement |
| R3 | Nylon | Trilobal | Residence | Basement |
| R4 | Nylon | Trilobal | Residence | Living room |
| R5 | Nylon | Trilobal<br>Thin lobed, Y | Residence | Top of stairs, second floor |
| R6 | Nylon | Trilobal | Residence | Living room |
| R7 | Nylon | Trilobal | Residence | Base of the stairs near front door |
| V1 | Nylon | Trilobal | Vehicle | 2008 Honda CRV middle front footwell |
| V2 | Nylon | Trilobal | Vehicle | 2006 Lexus LX70 trunk area |
| V3 | Nylon | Trilobal | Vehicle | 2001 Honda Odyssey trunk area |
| V4 | Not Determined | Round | Vehicle | 2009 Ford Focus trunk area |
| V5 | Nylon | Trilobal | Vehicle | 2008 Toyota Highlander front passenger footwell |

[3] Description and documentation of the carpet locations and fiber types is given in *Appendix B. Additional Carpet Sampling for Between-Item Variation.*

C. SEM Imaging and Qualitative Assessment of Particle Abundance on Fiber Surfaces

Fibers were mounted on carbon tape affixed to an aluminum SEM stub and lightly carbon-coated using a Cressington Auto Carbon Coater 108c with carbon evaporation conducted at 3.8 volts for 6 seconds. The fibers were then imaged using a Tescan Vega II scanning electron microscope with an accelerating voltage of 5 kV and a probe current of PC = 10. No charging was observed and no spontaneous scattering of particles was observed.

D. Isolation of Particles from Fibers (Protocol Development)

Five alternative fluids were tested using a set of alternative agitation methods and agitation times. The five fluids were acetone, ethanol, hexanes, 5% aqueous ethanol, and 5% aqueous sodium hexametaphosphate. Acetone is the liquid medium recommended for preparing soil and dust samples for automated SEM analysis.[27] Ethanol is an effective deflocculant that has long been used to separate aggregates of fine particles adhering to each other, especially for preparation of palynological samples.[28] Hexanes were used as a representative non-polar liquid to test their relative effectiveness, given that all of the others being tested were polar in nature. Dilute aqueous alcohol solutions are routinely used in our soil separation methods and are recommended for deflocculation during settling velocity separations.[29] Dilute (5%) aqueous sodium hexametaphosphate is commonly used by soil scientists to deflocculate clay and silt particles for separation by settling velocity and is used as a deflocculant in the ASTM Standard Test Method for Particle-Size Analysis of Soils.[30]

The methods of agitation were sonication, vortex mixing, and a combination of the two. Sonication was performed using a Cole Palmer model 08849-00 ultrasonic cleaner, using the single, fixed setting. Durations of sonication were limited to a maximum of ten minutes, as there is strong evidence that with sonication times exceeding ten minutes particles as hard as quartz may begin to break apart into smaller grains.[31] Table 3 shows agitation conditions used with each fluid.

Table 3. The Set of Agitation Conditions used for Each Fluid During Development of the Particle Isolation Protocol

| Sonication Only | Vortex Mixing Only | Combinations of Sonication and Vortex Mixing |
|---|---|---|
| 10 seconds | 10 seconds | 5 seconds vortexing<br>10 seconds sonication<br>5 seconds vortexing |
| 30 seconds | 60 seconds | 5 seconds vortexing followed by two repetitions of:<br>30 seconds sonication<br>5 seconds vortexing |
| 60 seconds | | 5 seconds vortexing followed by ten repetitions of:<br>60 seconds sonication and<br>5 seconds vortexing |
| 5 minutes | | |
| 10 minutes | | |

After each agitation experiment, the fiber was removed from the solvent using clean, fine-tipped forceps and gently placed on carbon tape mounted on an aluminum SEM stub. The agitation experiments and fiber mounting were conducted in a clean bench to minimize environmental contamination. Following particle removal processes and mounting for SEM, fibers were imaged by SEM to determine whether all adhering particles had been removed or not. For each fiber, an attempt was made to document the dirtiest areas (those with the most adhering particles), the cleanest areas (those with the fewest particles), and those with an "average" or typical level of cleanliness as observed on the fiber. At least eight images were taken documenting the appearance of each fiber.

E. Isolation of Particles from Fibers (Finalized Method)[4]

All procedures were conducted on the clean bench. Prior to starting, a significant volume (roughly 100 mL) of reagent grade ethanol (99.5%) was filtered through a polycarbonate filter (Millipore Isopore™ Membrane Filters, 0.4 μm HTTP). All test tubes, transfer pipets, forceps and razor blades were pre-washed with the particle-free ethanol.

Approximately 0.5 mL of pre-filtered reagent grade ethanol (99.5%) was transferred to a 1.5 mL micro-centrifuge tube using a transfer pipet. The fiber(s) to be analyzed were placed inside the tube using forceps. The tube was then sonicated in an ultrasonic cleaner (Cole Palmer model 08849-00) for ten minutes. After sonication the fiber(s) were removed from the tube using forceps, leaving behind a particle suspension in ethanol.

A polycarbonate filter (Millipore Isopore™ Membrane Filters, 0.4 μm HTTP) was placed on a support pad and then directly on a vacuum filtration apparatus (with no upper funnel attached). The vacuum was applied to the filter and then a small square region (roughly 5 mm by 5 mm) was cut out of the center of the filter using a Teflon-coated razor blade and left in place. A wide-tipped (1.5mm ID) transfer pipet was used to re-suspend the fine particles in the ethanol and then suction up the ethanol particle suspension. The suspension was slowly transferred from the pipet to the small square cut out of the filter while the vacuum pulled the ethanol through the filter. The filter was left on the support pad to dry, after which the small square was transferred to carbon tape on an SEM stub using forceps. The SEM stub was carbon-coated using a Cressington Auto Carbon Coater 108c with carbon evaporation conducted at 3.8 volts for 6 seconds and evaluated for particle dispersal by imaging in Tescan Vega II scanning electron microscope using an accelerating voltage of 5 kV and a probe current of PC = 10. In all instances the particles were well-dispersed, with the typical appearance as in Figure 2.

A process control blank was prepared alongside each batch of samples processed (pre-filtered ethanol sonicated for ten minutes in an empty tube and otherwise treated in an identical manner).

[4] One of the research products is this method, and it is described in detail in *Appendix C. Illustrated Fine Particle Removal and Mounting Method.*

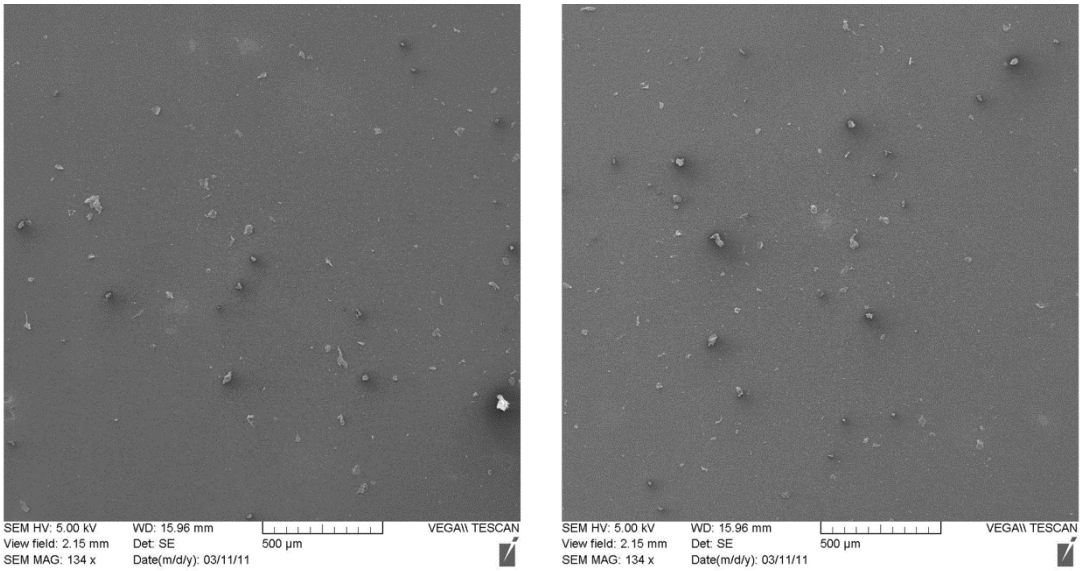


Figure 2. Typical appearance of the prepared carbon-coated SEM stubs upon inspection prior to computer-assisted SEM/EDS analysis. The particles are well-dispersed.

F. Computer-Assisted SEM/EDS Analysis of Particles

Standardized computer-assisted SEM/EDS analyses were performed by Gateway Analytical, LLC. Analyses were conducted on a total of 120 samples, along with 12 process blanks. These samples are summarized in Table 4. The 27 sets of ten fibers were collected from the nine carpets listed in Table 1 (one set from each area sampled). The 10-fiber sets were used to represent the population of particles at that area of the carpet. The 81 individual fibers, three from each of the 27 areas, were used to represent individual shed fibers from these areas. The 12 samples from each of the carpets in Table 2 each consisted of ten fibers and were used to represent populations of particles for those carpets. The 12 process blanks were those run alongside the processing of each batch of fiber samples. The sample designation scheme for the fiber samples from the Carpets in Table 1 is shown in Table 5, using Carpet A as an example.

Table 4. Summary of Samples and Process Blanks

| | |
|---|---|
| Carpets Selected for Analysis of Within-Item Variation (Table 1) | |
| 27 samples | sets of ten fibers from each of the three areas on each of the nine carpets |
| 81 samples | 3 individual fibers from each of the three areas on each of the nine carpets |
| Carpets Selected for Broader Survey of Carpets (Table 2) | |
| 12 samples | sets of ten fibers from one area on each carpet |
| Process Blanks | |
| 12 samples | one process blank for each batch of fiber samples processed |

Table 5. Summary of Sample Designations for Fiber Samples from Carpet A

| Sample Designation | Description |
|---|---|
| A1-10 | Area 1 of Carpet A, 10 Fiber Sample |
| A1-1, A1-2, A1-3 | Area 1 of Carpet A, individual fiber samples 1, 2, and 3 |
| A2-10 | Area 2 of Carpet A, 10 Fiber Sample |
| A2-1, A2-2, A2-3 | Area 2 of Carpet A, individual fiber samples 1, 2, and 3 |
| A3-10 | Area 3 of Carpet A, 10 Fiber Sample |
| A3-1, A3-2, A3-3 | Area 3 of Carpet A, individual fiber samples 1, 2, and 3 |

The computer-assisted SEM/EDS analysis was performed on an Aspex Corporation PSEM Explorer SEM-EDS system using the Automated Feature Analysis (AFA) program within the Aspex Corporation Perception software. Analysis was performed under high vacuum conditions utilizing a 20.0kV accelerating voltage, backscatter electron detector (BSED), working distance of approximately 15mm-17mm, and spot size of approximately 37%. The magnification for the analysis was 2,000X with the number of electronic fields set at a maximum of 5 x 5. A grid dimension of 256 x 256 was used with a dwell time per pixel of 8µs for searching and 16µs for measuring. The size criteria for analysis were a minimum size of 0.3µm and a maximum size of 50.0µm. The maximum number of particles analyzed, as set by program parameters, was 4000. EDS parameters had a nominal duration of 3s, a maximum of 6s, a minimum count of 300 and a target count of 2500. An EDS Copper calibration check was performed prior to, and following each analysis. Operational settings restricted detection to the 28 elements listed in Table 6, which are given along with the x-ray lines used for detection. These elements were chosen to follow an existing environmental analysis protocol with a mix of rarely encountered and commonly occurring elements among a set of 28. The number of elements (among those detectable by EDS) is not restricted by the SEM/EDS or by the software and the assortment of elements can be changed. The number of elements selected affects the overlap of x-ray lines, but ambiguities are resolved by the x-ray detection software using known correlated x-ray emissions for the specific elements. Specific x-ray windows used for elemental detection for each of the elements are given in *Appendix E-12. X-Ray Count Windows*.

Raw datasets for each computer-assisted SEM/EDS run consisted of summary statistics along with individual particle analysis data. Summary statistics were: total number of fields analyzed, total search area, total number of particles analyzed, and number of particles classified into each of ten pre-defined Environmental Particle Groups. Individual particle data were a particle index number, a set of particle size and shape parameters, the four elements in the particle's EDS spectrum having the highest x-ray counts, and the corresponding four x-ray counts. Particle size and shape parameters were the average, maximum and minimum particle diameters, the aspect ratio, area and perimeter. Classification among the ten pre-defined Environmental Particle Groups was based on percentages of total x-ray counts for specified elements. Alternative criteria were applied in sequence until a criterion was met. The specific criteria are given in Table 7.

Table 6. The 28 Elements Detected by the Automated EDS Procedure

| | | | |
|---|---|---|---|
| Sodium (Kα, Kβ) | Magnesium (Kα, Kβ) | Aluminum (Kα, Kβ) | Silicon (Kα, Kβ) |
| Sulfur (Kα, Kβ) | Chlorine (Kα, Kβ) | Calcium (Kα, Kβ) | Titanium (Kα, Kβ) |
| Chromium (Kα, Kβ) | Manganese (Kα, Kβ) | Iron (Kα, Kβ) | Nickel (Kα, Kβ) |
| Copper (Kα, Kβ) | Zinc (Kα, Kβ) | Bromine (Lα, Lβ) | Strontium (Lα, Lβ) |
| Zirconium (Lα, Lβ, Lγ) | Silver (Lα, Lβ, Lγ) | Tin (Lα, Lβ, Lγ) | Antimony (Lα, Lβ, Lγ) |
| Barium (Lα, Lβ, Lγ) | Tungsten (Mα) | Gold (Mα) | Mercury (Mα) |
| Lead (Mα) | Bismuth (Mα) | Lanthanum (Lα, Lβ, Lγ) | Cerium (Lα, Lβ, Lγ) |

Table 7. Sequentially Applied Criteria for Environmental Particle Groupings

| Application Order | Environmental Particle Group | Percent of Total X-ray Counts for the 28 Elements in Table 6 |
|---|---|---|
| 1 | Cl/Ca/S | Cl>=10 and Ca>=10 and S>=10 |
| 2 | Na/Cl | Na>=10 and Cl>=10 and Ca<10 and S<10 |
| 3 | Al/Si | Al>=10 and Si>=20 |
| 4 | Ca/S | Ca>=10 and S>=10 |
| 5 | Fe-rich | Fe>=10 |
| 6 | Ca-rich | Ca>=60 |
| 7 | Si-rich | Si>=40 |
| 8 | Al-rich | Al>=20 |
| 9 | Na-rich | Na>10 |
| 10 | Misc | all other particles |

G. Analysis of Environmental Particle Groupings

Frequencies for the Environmental Particle Groupings obtained from the computer-assisted SEM/EDS analyses were analyzed using the method of Kelley,[32] based on a multinomial distribution with maximum-likelihood estimation and chi-square testing. This statistical treatment assumes that a sample of size N (the particles adhering to the questioned carpet fiber) has been drawn from a population of interest (all of the particles adhering to fibers on the control carpet). As the sample is drawn, each of the particles is classified into one of *k* categories. The categories must be mutually exclusive and together include all elements in the population. Comparison between the questioned and known samples is based on the assumption that the frequencies of particles observed in the sample (questioned carpet fiber) are representative of the proportions in the population (control carpet), and that the population has been sampled randomly in on the questioned fiber. The control carpet particle group population frequencies were determined based on isolation and analysis of particles from a pooled sample of ten fibers from one carpet area. Questioned fiber particle group frequencies were those from individual fibers taken from the same carpet areas. Three individual fibers were analyzed for each area.

H. Within-Sample Elemental Profile Classes

For classification of more complete elemental profiles the four highest x-ray counts were normalized. Starting with the first particle, each successive particle was checked for qualitative and quantitative correspondence. Qualitative correspondence was determined by the presence, in the other particle, of any elements needed to account for at least 85% of the normalized x-ray counts. Quantitative correspondence was determined by normalized x-ray counts within a tolerance of 30% of one another, according to Equation 1, where $C_1$ and $C_2$ are normalized x-ray counts for the two corresponding elements. The set of corresponding particles defined the first group. This process was repeated for each ungrouped particle, testing the remaining ungrouped particles and defining further groupings. The values of 0.85 for the qualitative correspondence threshold and 30% for the quantitative x-ray count tolerance were chosen after consultation with subject matter experts[5] based on expected instrument detection thresholds and x-ray count variability.

Equation 1. $$0.30 > \frac{|(C_1 - C_2)|}{(C_1 + C_2) / 2)}$$

I. Selection, Definition and Frequency of Target Particle Types

Target Particle Types (TPTs) were defined based on those within-sample Elemental Profile Classes occurring frequently in samples from the nine carpets listed in Table 1. Analyses were conducted on sets of ten fibers from each of the three areas sampled for each of the nine carpets; a total of 27 analyses. Elemental Profile Classes occurring with a frequency of greater than 1% in any of the 27 analyses were selected. A set of mutually-exclusive TPTs were defined based on grouping of closely related and overlapping selected Elemental Profile Classes.[6] Frequencies of each of the TPTs were determined for each of the 120 sample analyses and 12 sample process blanks. Limits of Detection (LODs) for each of the TPTs were defined based as 3 times the sample standard deviation of the process blanks.

[5] David Exline of Gateway Analytical, LLC and Richard Brown of MVA Scientific Consultants, Inc.
[6] Details of the definition of the Target Particle Types are given as part of the Results.

## III. Results

A. Isolation of Particles from Fibers

Acetone, ethanol, hexanes, 5% aqueous ethanol, and 5% aqueous sodium hexametaphosphate were evaluated for their efficiency of fine particle removal from the surfaces of trilobal carpet fibers using a range of agitation conditions. SEM images documenting the results of the experimentation testing the particle removal processes can be found in *Appendix D: Fiber Particle Removal Documentation Dataset*.

*1. Acetone*

A noticeable number of particles remained adhering to the fibers after essentially all of the acetone agitation experiments. It was not clear whether sonication, vortex mixing, or a combination of the two was most effective, nor was there an apparent trend relating to agitation times. It was clear that acetone is not a good candidate for quantitative removal of small particles from fibers.

*2. Ethanol*

The ethanol agitation methods did a considerably better job of removing particles from fibers than either the acetone or hexanes, and a slightly better job than the 5% aqueous ethanol solution. Ethanol was comparable to (or slightly poorer than) a 5% aqueous sodium hexametaphosphate solution. The ten-minute sonication experiment for ethanol resulted in a very clean fiber, and was the most effective agitation method based on the cleanliness of the fiber after treatment. Vortex mixing alone was somewhat less effective than sonication alone. A combination of sonication and vortex mixing was not noticeably more effective than sonication alone, making the additional required time and effort unjustified. Ethanol is a good candidate for quantitative removal of small particles from fibers.

*3. Hexanes*

A noticeable number of particles remained adhering to the fibers after virtually all of the hexanes agitation experiments. Of the different agitation treatments, the ten-minute combination of vortex mixing and sonication resulted in the cleanest fiber, although it was not as clean as the fiber treated with ten-minute sonication in ethanol. Hexanes are not a good candidate for quantitative removal of small particles from fibers.

*4. 5% Aqueous Ethanol*

A noticeable but small number of particles remained adhering to the fibers after virtually all of the 5% ethanol agitation experiments. Of the different agitation treatments, the ten-minute combination of vortex mixing and sonication resulted in the cleanest fiber, and it was almost as clean as the fiber treated with ten-minute sonication in ethanol. The fibers treated with other agitation times and methods retained more particles than those treated with pure ethanol. 5% aqueous ethanol is not as good of a candidate for quantitative removal of small particles from fibers as pure ethanol.

*5. 5% Aqueous Sodium Hexametaphosphate*

The 5% aqueous sodium hexametaphosphate agitation experiments appeared to do a considerably better job of removing particles from fibers than either the acetone or the hexanes,

and a slightly better job than either the pure ethanol or the 5% aqueous ethanol solution. However, the 5% aqueous sodium hexametaphosphate left a residue on the fibers after washing. This residue is rich in sodium and phosphorus, and is expected to impact the SEM/EDS particle analysis by its presence on or among particles washed from the fibers. Particles of this composition (formed by drying) could potentially be washed away during filtration using a separate washing step, but this is an undesirable extra source of possible contamination.

These findings resulted in the finalized fine particle removal and mounting method referred to in the Methods section and described in detail in *Appendix C. Illustrated Fine Particle Removal and Mounting Method.*

B. Computer-Assisted SEM Analyses

The computer-assisted SEM analyses conducted on the 120 samples and 12 process blanks are provided in *Appendix E. Computer-Assisted SEM-EDS Analysis Dataset*. Individual spreadsheets are provided within 11 Excel Workbooks. Nine of these workbooks correspond to the nine carpets selected for analysis of within-carpet variation, as listed in Table 1. Worksheets bear sample designations following the scheme illustrated in Table 5. A tenth workbook corresponds to the additional carpets selected for analysis of between-carpet variation, as listed in Table 2. Worksheets bear the carpet designations. The final workbook contains spreadsheets corresponding to the process blanks. Worksheets bear the designations B0 (B-zero) through B11.

Particle numbers recovered and analyzed from the 39 sets of ten fibers are summarized in Table 8. Those from the 12 process blanks are summarized in Table 9 and those from the 81 individual fibers are summarized in Table 10. Descriptive statistics are found Tables 11 and 12.[7] The maximum number of particles analyzed, as set by program parameters, was 4000. For sets of ten fibers, this number was reached in 41% of the samples (16 of 39), compared to 5% of the single fibers (4 of 81), and none of the process blanks. On the average, sets of ten fibers showed 2780.4 particles (n = 39, SD = 1353.0); single fibers showed 720.7 particles (n = 81, SD = 943.8); and blanks showed 175.6 particles (n = 12, SD = 126.8). Particles were well dispersed on the SEM stubs as indicated by very low averages for the number of particles present per field (as documented in *Appendix E. Computer-Assisted SEM-EDS Analysis Dataset*). Most samples had an average of much less than one particle per field and no sample had an average of more than three.

Histograms of particle sizes for each of the 132 samples are given as part of *Appendix F. Particle Descriptive Statistics and Histograms*. Each sample has two histograms to allow examination of the distribution of the coarser as well as the finger particles. Figure 3 shows histograms from sample A3-10 as an example.

Environmental Particle Groupings for each of the sets of ten fibers sampled from each area of the nine carpets in Table 1 are summarized in Table 13, grouped by Residential, Vehicular and Workplace carpets. Tables with Environmental Particle Groupings for each of the carpet areas and each of the corresponding individual fibers are given in *Appendix G. Environmental Particle Groupings Dataset*.

[7] Full descriptive statistics can be found in the Excel file *Appendix F. Particle Numbers Descriptive Statistics*.

Table 8. Numbers of Particles Recovered and Analyzed from Sets of ten Fibers[8]

| Residential Carpets | | Vehicular Carpets | | Workplace Carpets | |
|---|---|---|---|---|---|
| Carpet A | 3001 | Carpet BV | 1233 | Carpet MT | 4000 |
| | 4000 | | 3613 | | 2750 |
| | 4000 | | 1431 | | 1564 |
| Carpet BH | 2948 | Carpet F | 445 | Carpet PL | 4000 |
| | 4000 | | 1179 | | 2169 |
| | 4000 | | 359 | | 3662 |
| Carpet PH | 1363 | Carpet PV | 4000 | Carpet W | 2896 |
| | 4000 | | 4000 | | 4000 |
| | 4000 | | 1005 | | 710 |
| Carpet R1 | 642 | Carpet V1 | 3422 | | |
| Carpet R2 | 4000 | Carpet V2 | 1803 | | |
| Carpet R3 | 4000 | Carpet V3 | 3485 | | |
| Carpet R4 | 3206 | Carpet V4 | 1030 | | |
| Carpet R5 | 4000 | Carpet V5 | 518 | | |
| Carpet R6 | 4000 | | | | |
| Carpet R7 | 4000 | | | | |

Table 9. Numbers of Particles Recovered and Analyzed from Process Blanks[8]

| Process Blanks | |
|---|---|
| Blank B0 | 138 |
| Blank B1 | 227 |
| Blank B2 | 89 |
| Blank B3 | 279 |
| Blank B4 | 215 |
| Blank B5 | 235 |
| Blank B6 | 89 |
| Blank B7 | 465 |
| Blank B8 | 241 |
| Blank B9 | 24 |
| Blank B10 | 32 |
| Blank B11 | 73 |

[8] Limited to 4000 particles by program settings.

Table 10. Numbers of Particles Recovered and Analyzed from Individual Fibers

| Residential Carpets | | | Vehicular Carpets | | | Workplace Carpets | | |
|---|---|---|---|---|---|---|---|---|
| Carpet A | Area 1 | 164 | Carpet BV | Area 1 | 754 | Carpet MT | Area 1 | 498 |
| | | 161 | | | 1107 | | | 127 |
| | | 490 | | | 307 | | | 123 |
| | Area 2 | 872 | | Area 2 | 1500 | | Area 2 | 122 |
| | | 1110 | | | 1757 | | | 190 |
| | | 1783 | | | 2121 | | | 440 |
| | Area 3 | 67 | | Area 3 | 521 | | Area 3 | 214 |
| | | 306 | | | 574 | | | 47 |
| | | 85 | | | 365 | | | 61 |
| Carpet BH | Area 1 | 244 | Carpet F | Area 1 | 437 | Carpet PL | Area 1 | 1492 |
| | | 345 | | | 293 | | | 1742 |
| | | 239 | | | 464 | | | 848 |
| | Area 2 | 421 | | Area 2 | 286 | | Area 2 | 505 |
| | | 312 | | | 129 | | | 494 |
| | | 691 | | | 397 | | | 518 |
| | Area 3 | 4000 | | Area 3 | 237 | | Area 3 | 375 |
| | | 504 | | | 213 | | | 692 |
| | | 234 | | | 166 | | | 454 |
| Carpet PH | Area 1 | 70 | Carpet PV | Area 1 | 809 | Carpet W | Area 1 | 514 |
| | | 59 | | | 438 | | | 464 |
| | | 205 | | | 604 | | | 535 |
| | Area 2 | 137 | | Area 2 | 4000 | | Area 2 | 419 |
| | | 147 | | | 4000 | | | 367 |
| | | 4000 | | | 3779 | | | 280 |
| | Area 3 | 504 | | Area 3 | 674 | | Area 3 | 320 |
| | | 711 | | | 950 | | | 258 |
| | | 1568 | | | 330 | | | 152 |

Table 11. Descriptive Statistics for Particle Numbers Recovered and Analyzed

| 10 Fiber sets | | Single Fibers | | Blanks | |
|---|---|---|---|---|---|
| Count | 39 | Count | 81 | Count | 12 |
| Mean | 2780.4 | Mean | 727.0 | Mean | 175.6 |
| Standard Error | 216.6 | Standard Error | 104.8 | Standard Error | 36.6 |
| Standard Deviation | 1353.0 | Standard Deviation | 943.0 | Standard Deviation | 126.8 |
| Median | 3422 | Median | 437 | Median | 176.5 |
| Minimum | 359 | Minimum | 47 | Minimum | 24 |
| Maximum | 4000 | Maximum | 4000 | Maximum | 465 |

Table 12. Descriptive Statistics by Carpet Type for Particle Numbers Recovered and Analyzed[9]

| 10-Fiber Sets[9] | | | | | |
|---|---|---|---|---|---|
| Residential | | Vehicular | | Workplace | |
| Count | 10 | Count | 8 | Count | 3 |
| Mean | 3428.5 | Mean | 2001.6 | Mean | 2861.2 |
| Standard Error | 327.4 | Standard Error | 427.0 | Standard Error | 218.8 |
| Standard Deviation | 1035.3 | Standard Deviation | 1207.6 | Standard Deviation | 378.9 |
| Median | 3833.5 | Median | 1947.7 | Median | 2771.3 |
| Minimum | 642 | Minimum | 518 | Minimum | 2535.3 |
| Maximum | 4000 | Maximum | 3485 | Maximum | 3277 |
| Individual Fibers | | | | | |
| Residential | | Vehicular | | Workplace | |
| Count | 27 | Count | 27 | Count | 27 |
| Mean | 700.6 | Mean | 1007.9 | Mean | 453.7 |
| Standard Error | 200.8 | Standard Error | 222.9 | Standard Error | 74.9 |
| Standard Deviation | 1043.1 | Standard Deviation | 1158.0 | Standard Deviation | 389.1 |
| Median | 312 | Median | 521 | Median | 419 |
| Minimum | 59 | Minimum | 129 | Minimum | 47 |
| Maximum | 4000 | Maximum | 4000 | Maximum | 1742 |

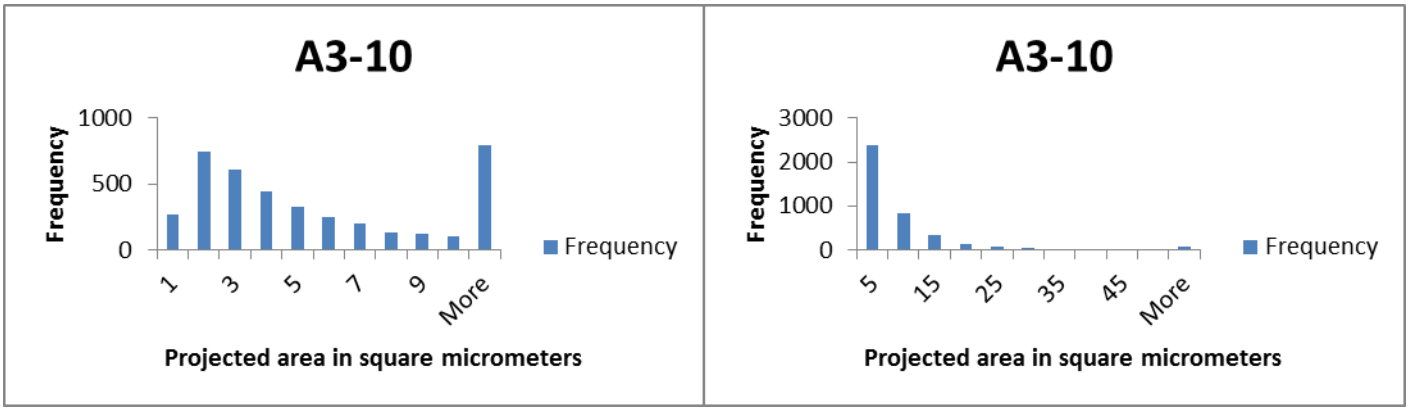


Figure 3. Particle size histograms for sample A3-10. Histograms for all 132 samples can be found in *Appendix F. Particle Descriptive Statistics and Histograms*.

[9] A single mean value was used for the nine carpets where three areas were sampled.

Table 13. Environmental Particle Groupings for the 27 Areas of the Nine Carpets in Table 1

| | Residential | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Carpet A | | | | | | Carpet BH | | | | | | Carpet PH | | | | | |
| | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | |
| Class | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. |
| Al/Si | 568 | 0.189 | 485 | 0.121 | 1425 | 0.356 | 273 | 0.093 | 817 | 0.204 | 219 | 0.055 | 61 | 0.045 | 39 | 0.010 | 277 | 0.069 |
| Al-rich | 72 | 0.024 | 39 | 0.010 | 125 | 0.031 | 28 | 0.009 | 86 | 0.022 | 44 | 0.011 | 27 | 0.020 | 14 | 0.004 | 39 | 0.010 |
| Ca/S | 617 | 0.206 | 324 | 0.081 | 304 | 0.076 | 85 | 0.029 | 84 | 0.021 | 291 | 0.073 | 73 | 0.054 | 77 | 0.019 | 154 | 0.039 |
| Ca-rich | 316 | 0.105 | 193 | 0.048 | 312 | 0.078 | 297 | 0.101 | 331 | 0.083 | 207 | 0.052 | 517 | 0.379 | 1971 | 0.493 | 1408 | 0.352 |
| Cl/Ca/S | 1 | 0.000 | 0 | 0.000 | 3 | 0.001 | 2 | 0.001 | 0 | 0.000 | 0 | 0.000 | 1 | 0.001 | 0 | 0.000 | 2 | 0.001 |
| Fe-rich | 275 | 0.092 | 35 | 0.009 | 347 | 0.087 | 276 | 0.094 | 508 | 0.127 | 1335 | 0.334 | 19 | 0.014 | 52 | 0.013 | 227 | 0.057 |
| Misc | 345 | 0.115 | 498 | 0.125 | 367 | 0.092 | 775 | 0.263 | 777 | 0.194 | 1296 | 0.324 | 286 | 0.210 | 1209 | 0.302 | 868 | 0.217 |
| Na/Cl | 26 | 0.009 | 3 | 0.001 | 39 | 0.010 | 20 | 0.007 | 18 | 0.005 | 3 | 0.001 | 53 | 0.039 | 26 | 0.007 | 78 | 0.020 |
| Na-rich | 486 | 0.162 | 2327 | 0.582 | 570 | 0.143 | 1080 | 0.366 | 1085 | 0.271 | 480 | 0.120 | 289 | 0.212 | 594 | 0.149 | 812 | 0.203 |
| Si-rich | 295 | 0.098 | 96 | 0.024 | 508 | 0.127 | 112 | 0.038 | 294 | 0.074 | 125 | 0.031 | 37 | 0.027 | 18 | 0.005 | 135 | 0.034 |
| Total | 3001 | 1.000 | 4000 | 1.000 | 4000 | 1.000 | 2948 | 1.000 | 4000 | 1.000 | 4000 | 1.000 | 1363 | 1.000 | 4000 | 1.000 | 4000 | 1.000 |
| | Vehicular | | | | | | | | | | | | | | | | | |
| | Carpet BV | | | | | | Carpet F | | | | | | Carpet PV | | | | | |
| | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | |
| Class | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. |
| Al/Si | 524 | 0.425 | 1494 | 0.414 | 563 | 0.393 | 137 | 0.308 | 198 | 0.168 | 105 | 0.292 | 1859 | 0.465 | 1692 | 0.42 | 334 | 0.332 |
| Al-rich | 24 | 0.019 | 68 | 0.019 | 32 | 0.022 | 7 | 0.016 | 13 | 0.011 | 8 | 0.022 | 149 | 0.037 | 131 | 0.03 | 17 | 0.017 |
| Ca/S | 23 | 0.019 | 54 | 0.015 | 14 | 0.010 | 6 | 0.013 | 8 | 0.007 | 7 | 0.019 | 10 | 0.003 | 147 | 0.04 | 12 | 0.012 |
| Ca-rich | 142 | 0.115 | 138 | 0.038 | 79 | 0.055 | 34 | 0.076 | 68 | 0.058 | 58 | 0.162 | 212 | 0.053 | 239 | 0.06 | 53 | 0.053 |
| Cl/Ca/S | 0 | 0.000 | 0 | 0.000 | 0 | 0.000 | 2 | 0.004 | 0 | 0.000 | 0 | 0.000 | 3 | 0.001 | 0 | 0.00 | 0 | 0.000 |
| Fe-rich | 110 | 0.089 | 424 | 0.117 | 156 | 0.109 | 63 | 0.142 | 148 | 0.126 | 67 | 0.187 | 605 | 0.151 | 819 | 0.20 | 302 | 0.300 |
| Misc | 146 | 0.118 | 348 | 0.096 | 285 | 0.199 | 59 | 0.133 | 399 | 0.338 | 49 | 0.136 | 413 | 0.103 | 476 | 0.12 | 113 | 0.112 |
| Na/Cl | 0 | 0.000 | 28 | 0.008 | 13 | 0.009 | 10 | 0.022 | 106 | 0.090 | 1 | 0.003 | 53 | 0.013 | 19 | 0.00 | 25 | 0.025 |
| Na-rich | 31 | 0.025 | 68 | 0.019 | 36 | 0.025 | 9 | 0.020 | 84 | 0.071 | 3 | 0.008 | 289 | 0.072 | 114 | 0.03 | 41 | 0.041 |
| Si-rich | 233 | 0.189 | 991 | 0.274 | 253 | 0.177 | 118 | 0.265 | 155 | 0.131 | 61 | 0.170 | 407 | 0.102 | 363 | 0.09 | 108 | 0.107 |
| Total | 1233 | 1.000 | 3613 | 1.000 | 1431 | 1.000 | 445 | 1.000 | 1179 | 1.000 | 359 | 1.000 | 4000 | 1.000 | 4000 | 1.00 | 1005 | 1.000 |

Table 13. (Continued)

| | Workplace | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Carpet MT | | | | | | Carpet PL | | | | | | Carpet W | | | | | |
| | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | | Area 1 | | Area 2 | | Area 3 | |
| Class | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. | Count | Freq. |
| Al/Si | 1191 | 0.298 | 1167 | 0.424 | 492 | 0.315 | 257 | 0.064 | 516 | 0.238 | 729 | 0.199 | 1002 | 0.346 | 1758 | 0.440 | 251 | 0.354 |
| Al-rich | 104 | 0.026 | 109 | 0.040 | 63 | 0.040 | 20 | 0.005 | 42 | 0.019 | 63 | 0.017 | 123 | 0.042 | 134 | 0.034 | 20 | 0.028 |
| Ca/S | 46 | 0.012 | 25 | 0.009 | 17 | 0.011 | 3097 | 0.774 | 468 | 0.216 | 1118 | 0.305 | 175 | 0.060 | 538 | 0.135 | 97 | 0.137 |
| Ca-rich | 656 | 0.164 | 138 | 0.050 | 82 | 0.052 | 159 | 0.040 | 176 | 0.081 | 562 | 0.153 | 176 | 0.061 | 233 | 0.058 | 103 | 0.145 |
| Cl/Ca/S | 0 | 0.000 | 2 | 0.001 | 0 | 0.000 | 0 | 0.000 | 10 | 0.005 | 7 | 0.002 | 5 | 0.002 | 8 | 0.002 | 2 | 0.003 |
| Fe-rich | 591 | 0.148 | 168 | 0.061 | 109 | 0.070 | 84 | 0.021 | 238 | 0.110 | 330 | 0.090 | 175 | 0.060 | 348 | 0.087 | 55 | 0.077 |
| Misc | 670 | 0.168 | 408 | 0.148 | 200 | 0.128 | 213 | 0.053 | 269 | 0.124 | 503 | 0.137 | 325 | 0.112 | 426 | 0.107 | 86 | 0.121 |
| Na/Cl | 5 | 0.001 | 28 | 0.010 | 16 | 0.010 | 0 | 0.000 | 5 | 0.002 | 11 | 0.003 | 86 | 0.030 | 1 | 0.000 | 0 | 0.000 |
| Na-rich | 61 | 0.015 | 246 | 0.089 | 363 | 0.232 | 31 | 0.008 | 71 | 0.033 | 91 | 0.025 | 507 | 0.175 | 17 | 0.004 | 9 | 0.013 |
| Si-rich | 676 | 0.169 | 459 | 0.167 | 222 | 0.142 | 139 | 0.035 | 374 | 0.172 | 248 | 0.068 | 322 | 0.111 | 537 | 0.134 | 87 | 0.123 |
| Total | 4000 | 1.000 | 2750 | 1.000 | 1564 | 1.000 | 4000 | 1.000 | 2169 | 1.000 | 3662 | 1.000 | 2896 | 1.000 | 4000 | 1.000 | 710 | 1.000 |

C. Statistical Testing of Environmental Particle Groupings

The specific hypothesis for the Environmental Particle Groupings was that carpet fibers from a source carpet will bear a measurable fine particle profile that is a function of an unbiased statistical sampling of the fine particle population present on the source carpet. Per the experimental design, the fine particle population on an area of the source carpet was represented by a set of ten fibers. Individual fibers taken from these same areas were used to represent individual evidentiary fibers, whose fine particle profiles were to be tested against the carpet fine particle population. Working with the computer-assisted SEM/EDS results in Appendix G, statistical analysis resulted in the clear rejection of the hypothesis. Tests of homogeneity resulted in values of the chi-square statistic far in excess of those expected under the null hypothesis; even at the highest levels of $\alpha$.[10] Details of the calculations of the chi-square statistic are given in *Appendix H. Environmental Groupings Within-Carpets Population Tests*. The hypothesis that the measured Environmental Particle Profiles on individual fibers represent an unbiased statistical sampling of the Environmental Particle Profiles on sets of ten fibers is strongly rejected.

D. Elemental Profile Groupings and Classes

Specific weaknesses of the Environmental Particle Groupings include: many particle compositions with ambiguous group classification, a group classification process that is dependent on the order of classification, and insensitivity to much of a particle's elemental profile. Alternative, Elemental Profile Groupings were designed to address these weaknesses and used to group particles of similar elemental compositions within samples.[11] Elemental Profile Groupings occurring with frequencies greater than 1% in each of the 27 10-fiber carpet area samples are given in *Appendix I. Elemental Particle Groupings Occurring with Frequencies Greater than One Percent.* As an example, Table 14 shows the data for Carpet A. The table gives element and normalized x-ray count data for individual particles that were used as the "seed" particle defining the group. (Membership in a group was determined by comparison to these specific particle characteristics.) In the table, shaded elements are those not needed to account for at least 85% of the normalized x-ray counts.

E. Definition of Target Particle Types

Sixty eight mutually exclusive Target Particle Types (TPTs) were defined by pooling and consolidation of particles represented in the Elemental Profile Groupings. Specific criteria for the TPTs were based on the qualitative presence of designated elements, and ranges and/or ratios in normalized x-ray counts for designated elements. These specific criteria are given in *Appendix J. Criteria for Target Particle Type Classifications*. Table 15 gives a descriptive summary of the TPTs.

[10] For example, of the 81 values of the chi-square statistic, only four were below 23.6, the value for $X^2_{.005}$ with 9 *df*

[11] See the Methods section for the steps used to define these classes and the Discussion section for explanation and discussion of the weaknesses.

Table 14. Example of Data in Appendix I: Carpet A Particles Defining Elemental Profile Groupings Occurring Above 1%*

| Area 1 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Group Size | | Four Elements with Highest X-Ray Counts | | | | Corresponding Normalized Counts | | | |
| Number | Percent | | | | | | | | |
| 197 | 6.6 | S | Ca | Bi | Hg | 0.44 | 0.27 | 0.16 | 0.14 |
| 83 | 2.8 | Ca | Na | Pb | Hg | 0.86 | 0.06 | 0.04 | 0.03 |
| 74 | 2.5 | Al | Si | Mg | Fe | 0.47 | 0.45 | 0.05 | 0.03 |
| 68 | 2.3 | Ca | Na | Mg | Zn | 0.83 | 0.09 | 0.05 | 0.02 |
| 46 | 1.5 | Mg | Ca | Na | Al | 0.46 | 0.42 | 0.07 | 0.05 |
| 43 | 1.4 | S | Ca | Na | Si | 0.44 | 0.34 | 0.16 | 0.06 |
| 42 | 1.4 | Si | Al | Sr | S | 0.61 | 0.26 | 0.08 | 0.05 |
| 41 | 1.4 | Ca | S | Na | Pb | 0.41 | 0.37 | 0.12 | 0.11 |
| 35 | 1.2 | Si | Al | Na | Ca | 0.37 | 0.32 | 0.17 | 0.14 |
| 34 | 1.1 | S | Bi | Ca | Hg | 0.39 | 0.27 | 0.22 | 0.12 |
| 33 | 1.1 | Si | S | Br | Mg | 0.83 | 0.06 | 0.06 | 0.05 |
| 32 | 1.1 | Si | Sr | S | Al | 0.83 | 0.09 | 0.04 | 0.04 |
| 31 | 1.0 | Si | Al | Na | Sr | 0.52 | 0.20 | 0.14 | 0.14 |

| Area 2 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Group Size | | Four Elements with Highest X-Ray Counts | | | | Corresponding Normalized Counts | | | |
| Number | Percent | | | | | | | | |
| 269 | 6.7 | Ca | Mg | Al | Zr | 0.45 | 0.41 | 0.08 | 0.06 |
| 198 | 5.0 | Ca | Na | Al | Mg | 0.47 | 0.41 | 0.07 | 0.05 |
| 140 | 3.5 | S | Na | Bi | Ca | 0.41 | 0.32 | 0.16 | 0.10 |
| 127 | 3.2 | Ca | Na | Al | Hg | 0.47 | 0.36 | 0.12 | 0.04 |
| 127 | 3.2 | Na | Ca | Al | Mg | 0.56 | 0.27 | 0.11 | 0.06 |
| 108 | 2.7 | Si | Al | S | Sr | 0.69 | 0.24 | 0.04 | 0.03 |
| 87 | 2.2 | Na | Hg | Zr | W | 0.50 | 0.19 | 0.18 | 0.13 |
| 61 | 1.5 | Ca | Si | Al | Na | 0.92 | 0.03 | 0.03 | 0.02 |
| 53 | 1.3 | Na | Ca | Hg | Br | 0.41 | 0.38 | 0.13 | 0.08 |
| 53 | 1.3 | S | Na | Hg | Pb | 0.38 | 0.32 | 0.17 | 0.14 |
| 52 | 1.3 | Na | Ca | S | Hg | 0.43 | 0.39 | 0.09 | 0.09 |
| 52 | 1.3 | Na | Ca | S | Al | 0.54 | 0.23 | 0.13 | 0.10 |
| 52 | 1.3 | S | Na | Bi | Ca | 0.45 | 0.24 | 0.22 | 0.09 |
| 52 | 1.3 | Na | S | Hg | Bi | 0.48 | 0.22 | 0.16 | 0.14 |
| 50 | 1.3 | Ca | Mg | Na | Al | 0.41 | 0.41 | 0.13 | 0.05 |
| 47 | 1.2 | Na | S | Hg | Pb | 0.43 | 0.37 | 0.10 | 0.10 |
| 45 | 1.1 | Na | S | Ca | Al | 0.50 | 0.29 | 0.14 | 0.08 |
| 45 | 1.1 | Ca | Mg | Na | S | 0.42 | 0.28 | 0.23 | 0.08 |
| 43 | 1.1 | Na | Ca | W | Hg | 0.41 | 0.36 | 0.13 | 0.10 |
| 43 | 1.1 | Na | Ca | Mg | Pb | 0.55 | 0.34 | 0.06 | 0.05 |

| Area 3 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Group Size | | Four Elements with Highest X-Ray Counts | | | | Corresponding Normalized Counts | | | |
| Number | Percent | | | | | | | | |
| 335 | 8.4 | Al | Si | Mg | Na | 0.49 | 0.38 | 0.07 | 0.06 |
| 128 | 3.2 | Si | Br | S | Cl | 0.87 | 0.05 | 0.04 | 0.03 |
| 98 | 2.5 | Ca | Mg | Al | Pb | 0.47 | 0.44 | 0.06 | 0.03 |
| 92 | 2.3 | Ca | Na | Al | W | 0.79 | 0.08 | 0.08 | 0.05 |
| 91 | 2.3 | Si | Al | Na | W | 0.51 | 0.29 | 0.16 | 0.05 |
| 90 | 2.3 | Si | Al | W | S | 0.59 | 0.31 | 0.05 | 0.04 |
| 80 | 2.0 | Si | Sr | Br | S | 0.74 | 0.14 | 0.08 | 0.04 |
| 68 | 1.7 | Si | Al | W | S | 0.70 | 0.19 | 0.07 | 0.04 |
| 66 | 1.7 | Si | Sr | S | Cl | 0.83 | 0.08 | 0.05 | 0.04 |
| 60 | 1.5 | Ca | Mg | Al | Si | 0.88 | 0.05 | 0.03 | 0.03 |
| 57 | 1.4 | Ca | Na | Bi | S | 0.73 | 0.12 | 0.10 | 0.04 |
| 56 | 1.4 | Si | Ca | Mg | Fe | 0.48 | 0.21 | 0.19 | 0.12 |
| 49 | 1.2 | Si | Al | Fe | Mg | 0.48 | 0.33 | 0.11 | 0.08 |
| 47 | 1.2 | Al | Si | Na | Mg | 0.52 | 0.30 | 0.13 | 0.04 |
| 45 | 1.1 | S | Ca | Bi | Hg | 0.40 | 0.38 | 0.12 | 0.11 |
| 42 | 1.1 | Na | S | Bi | Ca | 0.39 | 0.32 | 0.15 | 0.14 |

*Shaded elements are those not needed to account for at least 85% of the normalized x-ray counts.

Table 15. Descriptive Summary of Target Particle Types[12]

| Type | Designation | Normalized X-Ray Counts of | X-ray Count Ratios of | Restricted Levels of | Absence of |
|---|---|---|---|---|---|
| P1 | Al | Al | | | |
| P2 | Si | Si | | | |
| P3 | Si/Al Lower Si | Al, Si | Al, Si | | |
| P4 | Si/Al Medium Si | Al, Si | Al, Si | | |
| P5 | Si/Al Higher Si | Al, Si | Al, Si | | |
| P6 | Al/Si/Ca | Al, Ca, Si | Al, Si | Fe, Mg, Na, S, Sr, W | |
| P7 | Si/Al/Ca | Al, Ca, Si | | Fe, Mg, Na, S, Sr, W | |
| P8 | Al/Si/Fe Higher Fe | Al, Fe, Si | | | |
| P9 | Al/Si/Fe Lower Fe | Al, Fe, Si | Al, Si | | |
| P10 | Si/Al/Fe Lower Fe | Al, Fe, Si | Al, Si | | |
| P11 | Si/Al/Mg Higher Mg | Al, Mg, Si | Al, Si | Ca, Fe, Na, S, Sr, W | |
| P12 | Si/Al/Mg Lower Mg | Al, Mg, Si | Al, Si | Ca, Fe, Na, S, Sr, W | |
| P13 | Al/Si/Na | Al, Na, Si | Al, Si | | |
| P14 | Si/Al/Na Lower Si | Al, Na, Si | Al, Si | | |
| P15 | Si/Al/Na Higher Si | Al, Na, Si | Al, Si | Ca, Fe, Mg, S, Sr, W | |
| P16 | Al/Si | Al, Si | Al, Si | | |
| P17 | Al/Si/W | Al, Si, W | Al, Si | Ca, Fe, Mg, Na, S, Sr | |
| P18 | Si/Al/S | Al, Si, S | Al, Si | | |
| P19 | Si/Al/Sr | Al, Si, Sr | Al, Si | | |
| P20 | Si/Al/Ca/Na | Al, Ca, Na, Si | | | |
| P21 | Ca/Si/Mg | Ca, Mg, Si | Ca, Si | | |
| P22 | Si/Ca/Mg | Ca, Mg, Si | Ca, Si | | |
| P23 | Si/Mg | Mg, Si | Mg, Si | | |
| P24 | Si/Sr | Si, Sr | | | |
| P25 | Si/W | Si, W | | | |
| P26 | Ca | Ca | | | |
| P27 | Ca/Al | Ca, Al | | | |
| P28 | Ca/Bi | Ca, Bi | | | |
| P29 | Ca/Br | Ca, Br | | | |
| P30 | Ca/Fe/S | Ca, Fe, Si | Ca, Fe | | |
| P31 | Ca/Mg Higher Ca | Ca, Mg | | | |
| P32 | Ca/Mg Lower Ca | Ca, Mg | Ca, Mg | | |

[12] Full descriptions are given in *Appendix H. Criteria for Target Particle Type Classifications.*

Table 16. Descriptive Summary of Target Particle Types (Continued)

| Type | Designation | Normalized X-Ray Counts of | X-ray Count Ratios of | Restricted Levels of | Absence of |
|---|---|---|---|---|---|
| P33 | Ca/Mg/Al | Al, Ca, Mg | Ca, Mg | | |
| P34 | Ca/Mg/Na | Ca, Mg, Na | Ca, Mg | Al | |
| P35 | Ca/Na Lower Ca | Ca, Na | Ca, Na | | |
| P36 | Ca/Na Medium Ca | Ca, Na | Ca, Na | | |
| P37 | Ca/Na Higher Ca | Ca, Na | Ca, Na | | |
| P38 | Na/Ca | Ca, Na | | | |
| P39 | Ca/Na/Al Higher Ca | Al, Ca, Na | E2, E3[13] | | |
| P40 | Ca/Na/Al Lower Ca | Al, Ca, Na | Ca, Na | | |
| P41 | Na/Ca/Al | Al, Ca, Na | Ca, Na | | |
| P42 | Ca/Na/Br | Br, Ca, Na | E2, E3 | | |
| P43 | Ca/Na/(Hg,W) | Ca, Na, Hg, W | Ca, Na | Al | |
| P44 | Na/Ca/Hg/Zr | Ca, Hg, Na, Zr | | | |
| P45 | Ca/Na/W | Ca, Na, W | E2, E3 | | |
| P46 | Ca/W | Ca, W | | | |
| P47 | Ba/S/Bi | Ba, Bi, S | Ba, S | | |
| P48 | Ba/S/Na | Ba, Na, S | Ba, S ; Na, S | | |
| P49 | Ca/Ba/S | Ba, Ca, S | Ba, Ca | | |
| P50 | Ca/S (no Pb, Bi) | Ca, S | Ca, S | | Bi, Pb |
| P51 | S/Ca/Bi/Hg | Bi, Ca, Hg, S | Ca, S | | |
| P52 | S/Ca/Bi/Au | Au, Bi, Ca, S | Ca, S | | |
| P53 | S/Ca/Bi | Bi, Ca, S | Ca, S | | Au, Hg |
| P54 | S/Ca/Pb/Hg | Ca, Hg, Pb, S | Ca, S | | |
| P55 | S/Ca/Pb/Au | Au, Ca, Hg, Pb | Ca, S | | |
| P56 | S/Ca/Pb | Ca, Pb, S | Ca, S | | Au, Bi, Hg |
| P57 | Na/Ca/S | Ca, Na, S | Ca, Na | | |
| P58(1/2) | Na/S/Ca/Bi | Bi, Ca, Na, S | E1, E2 | | |
| P58(2/2) | Na/S/Ca/Bi | Bi, Ca, Na, S | Na, S | | |
| P59 | Na/S/Hg | Hg, Na, S | Na, S | Bi, Ca | |
| P60 | Fe | Fe | | | |
| P61 | Fe/Al | Al, Fe | Al, Fe | | |
| P62 | Fe/Cr/Na | Cr, Fe, Na | Cr, Fe ; Cr, Na | | |
| P63 | Fe/Na | Fe, Na | Fe, Na | | |
| P64 | Fe/Al/Si | Al, Fe, Si | Al, Fe | | |
| P65 | Cl/Na/Ca | Ca, Cl, Na | Ca, Na ; Cl, Na | | |
| P66 | Cu/Cl/Ni | Cl, Cu, Ni | Cl, Ni ; Cu, Cl | | |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb | Ca, Hg, Mg, Na, Pb, W, Zr | | | |
| P68 | Ti/(Al,La,Na) | Al, La, Na, Ti | Ti, E2 | | |

[13] E1, … E4 = Four elements with highest normalized x-ray counts in descending order

F. Occurrence of Target Particle Types for Carpets, Carpet Fibers and Process Blanks

The incidences of Target Particle Types (TPTs) in each of the samples and process blanks are given in *Appendix K. Incidence of Target Particle Types*. Examples of the data format are given in Tables 17 and 18. Ranges in incidence in these tables, and throughout this report and its appendices, are highlighted as follows: dark gray (0 to 5), gray (6 to10), yellow (11 to 50), orange (51 to 200) and red (> 200).

Process Blanks showed non-negligible levels for some TPTs. This occurrence is "particle noise" associated with the methodology that must be considered when interpreting the levels of occurrence of TPTs in this project. Most of the TPTs occur very rarely in the Blanks. Table 19 shows those TPTs with an incidence of more than five particles in any process blank or with an estimated Limit of Detection (LOD) greater than five.[14] Recurring high levels are present for seven of the TPTs (P2, P5, P15, P16, P24, P26, and P30). It is reasonable to omit these from further consideration.[15] Only one or two occurrences of high levels are present for the other 17 TPTs, and their significance is best judged in the context of the levels observed in the samples themselves. Table 20 shows the number of samples with levels greater than the LOD and greater than twice the LOD for these 17 TPTs. Sixteen of the TPTs have many occurrences in the samples above the LOD, ranging from 15 (12.5% of samples) to 66 (55% of samples). It is reasonable to consider these going forward. One of the TPTs (P1) shows only five samples with occurrences above the LOD, and only one of these samples has a level above twice the LOD. It is reasonable to omit this Type from further consideration.

[14] Mean plus 3 standard deviations using the one-tailed t-statistic.

[15] Although there are levels present in some of the samples above the LOD for these TPTs, the LOD are based on statistical assumptions and practices that are only strictly applicable for well-established methods and processes. This is not the case for the present project, and it would be reckless to assume that levels of these particles in the samples represent a reliable "signal."

Table 17. Example of Data in Appendix K for Sets of ten Fibers from One Area[16]
Target Particle Types in Residential Carpets - Sets of ten Fibers from One Area

| Target Particle Type | | Carpet A | | | | Carpet BH | | | | Carpet PH | | | | Broader Set of Carpets | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Type | Description | Area 1 | Area 2 | Area 3 | Mean | Area 1 | Area 2 | Area 3 | Mean | Area 1 | Area 2 | Area 3 | Mean | R1 | R2 | R3 | R4 | R5 | R6 | R7 |
| P47 | Ba/S/Bi | 4 | 0 | 0 | 1 | 2 | 4 | 1 | 2 | 14 | 96 | 62 | 57 | 5 | 3 | 3 | 1 | 8 | 7 | 4 |
| P48 | Ba/S/Na | 2 | 2 | 3 | 2 | 1 | 4 | 0 | 2 | 7 | 87 | 59 | 51 | 2 | 1 | 0 | 0 | 0 | 0 | 1 |
| P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 18 | 5 | 15 | 13 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P50 | Ca/S (no Pb, Bi) | 57 | 3 | 15 | 25 | 2 | 3 | 16 | 7 | 0 | 0 | 4 | 1 | 1 | 4 | 7 | 26 | 66 | 34 | 2 |
| P51 | S/Ca/Bi/Hg | 130 | 29 | 51 | 70 | 6 | 6 | 65 | 26 | 1 | 3 | 4 | 3 | 24 | 20 | 43 | 113 | 314 | 132 | 25 |
| P52 | S/Ca/Bi/Au | 64 | 15 | 29 | 36 | 4 | 5 | 42 | 17 | 1 | 0 | 3 | 1 | 22 | 5 | 16 | 90 | 198 | 79 | 10 |
| P53 | S/Ca/Bi | 35 | 6 | 8 | 16 | 3 | 2 | 19 | 8 | 1 | 4 | 4 | 3 | 6 | 15 | 21 | 56 | 73 | 43 | 9 |
| P54 | S/Ca/Pb/Hg | 38 | 5 | 13 | 19 | 2 | 8 | 30 | 13 | 1 | 3 | 2 | 2 | 4 | 5 | 10 | 29 | 78 | 28 | 6 |
| P55 | S/Ca/Pb/Au | 17 | 4 | 9 | 10 | 0 | 4 | 17 | 7 | 0 | 0 | 1 | 0 | 1 | 0 | 2 | 12 | 35 | 12 | 0 |
| P56 | S/Ca/Pb | 25 | 5 | 6 | 12 | 1 | 2 | 2 | 2 | 0 | 5 | 1 | 2 | 0 | 0 | 5 | 8 | 21 | 7 | 1 |
| P57 | Na/Ca/S | 0 | 115 | 26 | 47 | 0 | 0 | 1 | 0 | 1 | 0 | 4 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P58 | Na/S/Ca/Bi | 175 | 383 | 119 | 226 | 129 | 37 | 25 | 64 | 13 | 9 | 33 | 18 | 83 | 5 | 855 | 15 | 96 | 88 | 345 |
| P59 | Na/S/Hg | 19 | 167 | 18 | 68 | 9 | 19 | 10 | 13 | 1 | 4 | 27 | 11 | 8 | 0 | 19 | 0 | 2 | 1 | 4 |
| P60 | Fe | 4 | 0 | 12 | 5 | 8 | 8 | 220 | 79 | 0 | 0 | 1 | 0 | 2 | 5 | 6 | 8 | 11 | 27 | 5 |
| P61 | Fe/Al | 6 | 0 | 5 | 4 | 2 | 8 | 28 | 13 | 0 | 1 | 1 | 1 | 5 | 5 | 10 | 14 | 16 | 22 | 9 |
| P62 | Fe/Cr/Na | 6 | 2 | 6 | 5 | 16 | 30 | 2 | 16 | 1 | 1 | 3 | 2 | 0 | 2 | 0 | 4 | 0 | 1 | 0 |
| P63 | Fe/Na | 35 | 2 | 27 | 21 | 26 | 36 | 401 | 154 | 1 | 4 | 8 | 4 | 7 | 7 | 13 | 6 | 16 | 30 | 6 |
| P64 | Fe/Al/Si | 5 | 3 | 24 | 11 | 10 | 29 | 6 | 15 | 0 | 1 | 7 | 3 | 2 | 15 | 11 | 24 | 28 | 25 | 18 |
| P65 | Cl/Na/Ca | 0 | 0 | 0 | 0 | 44 | 0 | 0 | 15 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 1 | 6 | 9 | 5 | 497 | 373 | 174 | 348 | 1 | 2 | 5 | 3 | 0 | 1 | 2 | 2 | 49 | 28 | 140 |
| P68 | Ti/(Al,La,Na) | 61 | 5 | 31 | 32 | 26 | 39 | 579 | 215 | 20 | 9 | 15 | 15 | 5 | 11 | 11 | 27 | 38 | 25 | 18 |
| | Total Particles | 3001 | 4000 | 4000 | 3667 | 2948 | 4000 | 4000 | 3649 | 1363 | 4000 | 4000 | 3121 | 642 | 4000 | 4000 | 3206 | 4000 | 4000 | 4000 |
| | % as Target Types | 0.57 | 0.50 | 0.61 | 0.56 | 0.52 | 0.50 | 0.54 | 0.52 | 0.51 | 0.62 | 0.55 | 0.561 | 0.56 | 0.52 | 0.64 | 0.69 | 0.58 | 0.63 | 0.46 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

[16] See *Appendix K. Incidence of Target Particle Types*: Table K1 (3 of 3).

Table 18. Example of Data in Appendix K for Sets of ten and Individual Fibers in One Carpet[17]
Target Particle Types in Residential Carpet PH - Sets of ten and Individual Fibers

| Target Particle Type | | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Type | Description | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| P26 | Ca | 202 | 9 | 9 | 20 | 463 | 21 | 30 | 487 | 325 | 26 | 51 | 82 |
| P27 | Ca/Al | 29 | 1 | 2 | 0 | 251 | 3 | 12 | 295 | 163 | 2 | 22 | 21 |
| P28 | Ca/Bi | 14 | 0 | 0 | 1 | 71 | 1 | 1 | 71 | 34 | 2 | 5 | 16 |
| P29 | Ca/Br | 19 | 0 | 0 | 2 | 80 | 4 | 6 | 124 | 70 | 1 | 4 | 13 |
| P30 | Ca/Fe/S | 1 | 1 | 0 | 2 | 2 | 0 | 2 | 55 | 49 | 56 | 52 | 92 |
| P31 | Ca/Mg Higher Ca | 15 | 0 | 0 | 1 | 79 | 0 | 0 | 118 | 55 | 0 | 2 | 8 |
| P32 | Ca/Mg Lower Ca | 21 | 1 | 1 | 0 | 60 | 1 | 2 | 63 | 67 | 1 | 3 | 10 |
| P33 | Ca/Mg/Al | 0 | 0 | 0 | 0 | 8 | 0 | 0 | 21 | 16 | 0 | 0 | 2 |
| P34 | Ca/Mg/Na | 2 | 0 | 0 | 0 | 17 | 0 | 0 | 7 | 28 | 1 | 0 | 2 |
| P35 | Ca/Na Lower Ca | 39 | 0 | 0 | 4 | 107 | 1 | 0 | 85 | 103 | 5 | 8 | 11 |
| P36 | Ca/Na Medium Ca | 40 | 0 | 1 | 3 | 185 | 2 | 10 | 208 | 152 | 2 | 6 | 18 |
| P37 | Ca/Na Higher Ca | 81 | 0 | 3 | 7 | 383 | 10 | 13 | 463 | 286 | 9 | 22 | 31 |
| P38 | Na/Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 1 |
| P39 | Ca/Na/Al Higher Ca | 20 | 0 | 0 | 0 | 150 | 1 | 4 | 139 | 66 | 6 | 7 | 15 |
| P40 | Ca/Na/Al Lower Ca | 2 | 0 | 0 | 0 | 3 | 1 | 0 | 1 | 3 | 1 | 0 | 2 |
| P41 | Na/Ca/Al | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P42 | Ca/Na/Br | 12 | 0 | 0 | 1 | 63 | 1 | 4 | 63 | 39 | 1 | 1 | 10 |
| P43 | Ca/Na/(Hg,W) | 1 | 0 | 0 | 0 | 6 | 0 | 0 | 2 | 8 | 0 | 0 | 3 |
| P44 | Na/Ca/Hg/Zr | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 0 | 0 | 0 |
| P45 | Ca/Na/W | 25 | 1 | 0 | 0 | 108 | 2 | 4 | 94 | 85 | 3 | 4 | 8 |
| P46 | Ca/W | 31 | 1 | 4 | 2 | 175 | 3 | 4 | 207 | 114 | 7 | 15 | 15 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

[17] See *Appendix K. Incidence of Target Particle Types*: Table K6 (2 of 3).

Table 19. Target Particle Types with More than Five Particles in a Process Blank or an Estimated LOD Greater than Five Particles

| Target Particle Type | | Sample Designation (Process Blanks) | | | | | | | | | | | | LOD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Type | Description | B0 | B1 | B2 | B3 | B4 | B5 | B6 | B7 | B8 | B9 | B10 | B11 | |
| P1 | Al | 8 | 2 | 2 | 1 | 1 | 2 | 1 | 2 | 1 | 0 | 0 | 0 | 8.1 |
| P2 | Si | 2 | 5 | 5 | 12 | 3 | 2 | 1 | 23 | 13 | 1 | 2 | 0 | 26.3 |
| P3 | Si/Al Lower Si | 0 | 12 | 2 | 2 | 0 | 0 | 0 | 2 | 0 | 0 | 2 | 0 | 11.8 |
| P5 | Si/Al Higher Si | 1 | 3 | 0 | 10 | 4 | 1 | 0 | 27 | 14 | 0 | 5 | 0 | 29.8 |
| P6 | Al/Si/Ca | 0 | 3 | 0 | 4 | 1 | 0 | 0 | 4 | 1 | 0 | 1 | 2 | 6.0 |
| P7 | Si/Al/Ca | 0 | 0 | 1 | 2 | 0 | 0 | 0 | 3 | 0 | 0 | 0 | 5 | 5.8 |
| P11 | Si/Al/Mg Higher Mg | 0 | 0 | 0 | 6 | 0 | 0 | 0 | 6 | 1 | 0 | 0 | 0 | 8.0 |
| P14 | Si/Al/Na Lower Si | 3 | 2 | 0 | 5 | 0 | 0 | 0 | 5 | 0 | 0 | 0 | 0 | 7.3 |
| P15 | Si/Al/Na Higher Si | 7 | 2 | 1 | 18 | 0 | 0 | 0 | 41 | 38 | 0 | 1 | 2 | 54.4 |
| P16 | Al/Si | 2 | 7 | 1 | 35 | 4 | 2 | 1 | 50 | 9 | 11 | 1 | 0 | 57.6 |
| P17 | Al/Si/W | 1 | 4 | 1 | 0 | 0 | 0 | 0 | 3 | 2 | 1 | 0 | 0 | 5.0 |
| P19 | Si/Al/Sr | 0 | 6 | 0 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 2 | 1 | 6.1 |
| P23 | Si/Mg | 0 | 0 | 4 | 1 | 2 | 4 | 0 | 0 | 1 | 0 | 0 | 0 | 5.6 |
| P24 | Si/Sr | 2 | 4 | 4 | 22 | 5 | 5 | 1 | 33 | 17 | 1 | 1 | 1 | 39.1 |
| P25 | Si/W | 0 | 1 | 0 | 0 | 1 | 0 | 0 | 9 | 7 | 1 | 0 | 0 | 10.8 |
| P26 | Ca | 3 | 57 | 7 | 5 | 12 | 4 | 2 | 10 | 5 | 1 | 0 | 3 | 55.6 |
| P27 | Ca/Al | 0 | 9 | 3 | 3 | 11 | 3 | 1 | 1 | 1 | 0 | 0 | 0 | 13.6 |
| P30 | Ca/Fe/S | 15 | 14 | 0 | 16 | 75 | 64 | 15 | 3 | 0 | 0 | 1 | 0 | 93.7 |
| P37 | Ca/Na Higher Ca | 0 | 11 | 0 | 1 | 1 | 5 | 0 | 3 | 1 | 1 | 0 | 0 | 11.6 |
| P46 | Ca/W | 0 | 8 | 1 | 1 | 2 | 2 | 0 | 1 | 4 | 0 | 0 | 0 | 8.6 |
| P60 | Fe | 0 | 0 | 1 | 1 | 5 | 1 | 0 | 4 | 2 | 0 | 0 | 0 | 6.3 |
| P61 | Fe/Al | 0 | 0 | 0 | 1 | 0 | 5 | 0 | 1 | 2 | 0 | 0 | 1 | 5.2 |
| P62 | Fe/Cr/Na | 0 | 1 | 2 | 2 | 1 | 14 | 8 | 4 | 1 | 0 | 0 | 5 | 15.7 |
| P63 | Fe/Na | 1 | 2 | 3 | 1 | 5 | 1 | 1 | 2 | 1 | 0 | 0 | 0 | 5.7 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

Table 20. Number of Sample Occurrences Above LOD for 27 Target Particle Types with One or Two High Levels of Occurrence in Process Blanks

| Target Particle Type | | | Number of Samples | |
|---|---|---|---|---|
| Type | Description | LOD | > LOD | >2 LOD |
| P1 | Al | 8.1 | 5 | 1 |
| P62 | Fe/Cr/Na | 15.7 | 15 | 9 |
| P23 | Si/Mg | 5.6 | 25 | 8 |
| P61 | Fe/Al | 5.2 | 27 | 14 |
| P11 | Si/Al/Mg Higher Mg | 8.0 | 28 | 15 |
| P60 | Fe | 6.3 | 28 | 8 |
| P25 | Si/W | 10.8 | 39 | 19 |
| P46 | Ca/W | 8.6 | 42 | 25 |
| P27 | Ca/Al | 13.6 | 43 | 27 |
| P37 | Ca/Na Higher Ca | 11.6 | 46 | 26 |
| P6 | Al/Si/Ca | 6.0 | 47 | 33 |
| P19 | Si/Al/Sr | 6.1 | 50 | 34 |
| P63 | Fe/Na | 5.7 | 53 | 26 |
| P14 | Si/Al/Na Lower Si | 7.3 | 55 | 39 |
| P17 | Al/Si/W | 5.0 | 56 | 44 |
| P3 | Si/Al Lower Si | 11.8 | 58 | 37 |
| P7 | Si/Al/Ca | 5.8 | 66 | 40 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

G. Variation Among Different Areas of the Same Carpet

In this section and those that follow, some Target Particle Types (TPTs) have been omitted and others consolidated. Omitted types follow the rationale given in the previous section, with one additional omission: type P41, which had no occurrences. Five TPT consolidations were made for two or more closely related classifications that were found to occur with similar relative proportions in the samples. Table 21 shows these consolidations. The resulting 47 TPTs are conveniently considered in five chemical groups, as shown in Table 22: siliceous (P3 to P25), calcareous (P27 to P46), sulfurous (P47 to P59, ferrous (P60 to P64) and other (P65 to P68).

Table 21. Consolidations of Closely Related Target Particle Type Classifications that Occur with Similar Relative Proportions in the Samples[18]

| Target Particle Types | | | |
|---|---|---|---|
| Consolidated | | Original | |
| Type | Description | Type | Description |
| P6* | Si/Al/Ca | | |
| | | P6 | Si/Al/Ca |
| | | P7 | Al/Si/Ca |
| P8* | Si/Al/Fe | | |
| | | P8 | Al/Si/Fe Higher Fe |
| | | P9 | Al/Si/Fe Lower Fe |
| | | P10 | Si/Al/Fe Lower Fe |
| P31* | Ca/Mg | | |
| | | P31 | Ca/Mg Higher Ca |
| | | P32 | Ca/Mg Lower Ca |
| P35* | Ca/Na | | |
| | | P35 | Ca/Na Lower Ca |
| | | P36 | Ca/Na Medium Ca |
| | | P37 | Ca/Na Higher Ca |
| P50* | Ca/S | | |
| | | P50 | Ca/S (no Pb, Bi) |
| | | P51 | S/Ca/Bi/Hg |
| | | P52 | S/Ca/Bi/Au |
| | | P53 | S/Ca/Bi |
| | | P54 | S/Ca/Pb/Hg |
| | | P55 | S/Ca/Pb/Au |
| | | P56 | S/Ca/Pb |

[18] Unconsolidated data can be found in *Appendix K. Incidence of Target Particle Types.*

Table 23 shows results for three areas of Carpet A, which illustrate trends seen in the carpet area results. There are both notable similarities and notable differences. A linear, proportional distribution of a uniform fine particle population can be quickly dismissed. Under such a model we would expect each of the particle types to occur in proportions of 3:4:4 (given the respective total particle numbers of 3001, 4000 and 4000). Consideration of TPT P3 is sufficient: the levels of occurrence for Areas 1 and 2 are comparable (though off in proportion), but Area 3 has more than three times the particles. TPTs P4 and P20 show alternative trends which are also divergent from linear proportions based on the total particle count.

Table 22. Five Convenient Chemical Groupings of the Target Particle Types

| **Siliceous Group** | |
|---|---|
| Type | Description |
| P3 | Si/Al Lower Si |
| P4 | Si/Al Higher Si |
| P6* | Si/Al/Ca |
| P8* | Si/Al/Fe |
| P11 | Si/Al/Mg Higher Mg |
| P12 | Si/Al/Mg Lower Mg |
| P13 | Al/Si/Na |
| P14 | Si/Al/Na Lower Si |
| P17 | Al/Si/W |
| P18 | Si/Al/S |
| P19 | Si/Al/Sr |
| P20 | Si/Al/Ca/Na |
| P21 | Ca/Si/Mg |
| P22 | Si/Ca/Mg |
| P23 | Si/Mg |
| P25 | Si/W |

| **Calcareous Group** | |
|---|---|
| Type | Description |
| P27 | Ca/Al |
| P28 | Ca/Bi |
| P29 | Ca/Br |
| P31* | Ca/Mg |
| P33 | Ca/Mg/Al |
| P34 | Ca/Mg/Na |
| P35* | Ca/Na |
| P38 | Na/Ca |
| P39 | Ca/Na/Al Higher Ca |
| P40 | Ca/Na/Al Lower Ca |
| P42 | Ca/Na/Br |
| P43 | Ca/Na/(Hg,W) |
| P44 | Na/Ca/Hg/Zr |
| P45 | Ca/Na/W |
| P46 | Ca/W |

| **Sulfurous Group** | |
|---|---|
| Type | Description |
| P47 | Ba/S/Bi |
| P48 | Ba/S/Na |
| P49 | Ca/Ba/S |
| P50* | Ca/S |
| P57 | Na/Ca/S |
| P58 | Na/S/Ca/Bi |
| P59 | Na/S/Hg |
| **Ferrous Group** | |
| P60 | Fe |
| P61 | Fe/Al |
| P62 | Fe/Cr/Na |
| P63 | Fe/Na |
| P64 | Fe/Al/Si |
| **Other Group** | |
| P65 | Cl/Na/Ca |
| P66 | Cu/Cl/Ni |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb |
| P68 | Ti/(Al,La,Na) |

Table 23. Carpet A Target Particle Type Occurrences: ten Fibers from Each of Three Areas

| Carpet A | Area 1 | Area 2 | Area 3 |
|---|---|---|---|
| Total Particles | 3001 | 4000 | 4000 |

| | Carpet A: Siliceous | Area 1 | Area 2 | Area 3 |
|---|---|---|---|---|
| P3 | Si/Al Lower Si | 44 | 34 | 148 |
| P4 | Si/Al Medium Si | 15 | 82 | 48 |
| P6* | Si/Al/Ca | 32 | 17 | 71 |
| P8* | Si/Al/Fe | 28 | 8 | 114 |
| P11 | Si/Al/Mg Higher Mg | 11 | 2 | 51 |
| P12 | Si/Al/Mg Lower Mg | 23 | 8 | 61 |
| P13 | Al/Si/Na | 40 | 28 | 100 |
| P14 | Si/Al/Na Lower Si | 65 | 25 | 121 |
| P17 | Al/Si/W | 30 | 3 | 47 |
| P18 | Si/Al/S | 1 | 0 | 11 |
| P19 | Si/Al/Sr | 44 | 30 | 74 |
| P20 | Si/Al/Ca/Na | 15 | 4 | 39 |
| P21 | Ca/Si/Mg | 1 | 13 | 3 |
| P22 | Si/Ca/Mg | 15 | 10 | 64 |
| P23 | Si/Mg | 20 | 12 | 10 |
| P25 | Si/W | 11 | 3 | 26 |

| | Carpet A: Calcareous | Area 1 | Area 2 | Area 3 |
|---|---|---|---|---|
| P27 | Ca/Al | 46 | 27 | 55 |
| P28 | Ca/Bi | 7 | 4 | 7 |
| P29 | Ca/Br | 5 | 2 | 9 |
| P31* | Ca/Mg | 51 | 260 | 108 |
| P33 | Ca/Mg/Al | 20 | 52 | 29 |
| P34 | Ca/Mg/Na | 13 | 111 | 15 |
| P35* | Ca/Na | 145 | 56 | 129 |
| P38 | Na/Ca | 0 | 91 | 2 |
| P39 | Ca/Na/Al Higher Ca | 31 | 8 | 27 |
| P40 | Ca/Na/Al Lower Ca | 2 | 143 | 20 |
| P42 | Ca/Na/Br | 6 | 1 | 2 |
| P43 | Ca/Na/(Hg,W) | 1 | 36 | 6 |
| P44 | Na/Ca/Hg/Zr | 0 | 8 | 1 |
| P45 | Ca/Na/W | 22 | 5 | 9 |
| P46 | Ca/W | 28 | 11 | 15 |

| | Carpet A: Sulfurous | Area 1 | Area 2 | Area 3 |
|---|---|---|---|---|
| P47 | Ba/S/Bi | 4 | 0 | 0 |
| P48 | Ba/S/Na | 2 | 2 | 3 |
| P49 | Ca/Ba/S | 0 | 0 | 0 |
| P50* | Ca/S | 366 | 67 | 131 |
| P57 | Na/Ca/S | 0 | 115 | 26 |
| P58 | Na/S/Ca/Bi | 175 | 383 | 119 |
| P59 | Na/S/Hg | 19 | 167 | 18 |

| | Carpet A: Ferrous | Area 1 | Area 2 | Area 3 |
|---|---|---|---|---|
| P60 | Fe | 4 | 0 | 12 |
| P61 | Fe/Al | 6 | 0 | 5 |
| P62 | Fe/Cr/Na | 6 | 2 | 6 |
| P63 | Fe/Na | 35 | 2 | 27 |
| P64 | Fe/Al/Si | 5 | 3 | 24 |

| | Carpet A: Other | Area 1 | Area 2 | Area 3 |
|---|---|---|---|---|
| P65 | Cl/Na/Ca | 0 | 0 | 0 |
| P66 | Cu/Cl/Ni | 0 | 0 | 0 |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 1 | 6 | 9 |
| P68 | Ti/(Al,La,Na) | 61 | 5 | 31 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

Another possibility is that the relative proportions of the TPTs in each of the areas will be consistent. This is found for some types and not for others. Among the siliceous TPTs in Carpet A, for example, P6*, P13, P14 and P19 share a proportional relationship that is persuasive: Area 1 ranges from 40 to 60% of Area 3, while Area 2 ranges from 20 to 40%. A quick glance around the table shows that this is not a consistent trend: proportions vary widely among the three carpet areas.

Qualitative similarities among the areas are more abundant, notably the very low quantities of 11 of the TPTs (calcareous: P28, P29, P42, P44; sulfurous: P47, P48, P49; ferrous: P61, P62; other: P65, P66, P67). These, combined with the proportionality of the four siliceous TPTs (P6*, P13, P14, P19) are points of similarity.

Differences among the three carpet areas are also present, but they show trends. For many TPTs where Area 2 has its highest levels of occurrence, Areas 1 and 3 are low (calcareous: P34, P38, P40, P43; sulfurous: P57, 59). For TPTs with high incidence in Area 2, Areas 1 and 3 have levels ranging up to half that of Area 2 low (calcareous: P31*, P33; sulfurous: P58). Though not as striking, there are TPTs where Areas 1 and 3 have higher levels, with very low levels in Area 2 (siliceous: P8*, P11, P12, P17, P20, P25; calcareous: P35; ferrous: P63; other: P68). Overall, Area 1 and Area 3 are much closer, both qualitatively and with similar proportionality.

Table 24 shows results for siliceous TPTs from three different areas of three of the carpets. Carpet BH shows a consistent (though unequal, and non-proportional) relationship among the particle occurrences. Almost all siliceous TPTs in Carpet BH Area 2 show two to several-fold higher occurrences, with comparable levels for Areas 1 and 3. There is also good qualitative agreement among low occurrences across all three areas. Carpet PH shows very low occurrences of siliceous TPTs for two areas, contrasting markedly with the third area. Areas 1 and 2 of Carpet PV are remarkably similar for all 16 siliceous TPTs, while Area 3 shows much lower occurrences (along with a lower overall particle recovery).

Table 25 shows results for calcareous TPTs from three different areas of three of the carpets.[19] Carpet BH again shows a consistent (though unequal, and non-proportional) relationship among the particle occurrences. For the four most frequently occurring TPTs (P27, P31*, P33, P35*) Areas 1 and 2 have comparable levels (within about 30%), while Area 3 is consistently some 30 to 60% lower. Notably, this differs from the trend seen in the siliceous TPTs (higher Area 2 occurrences, with comparable occurrences for Areas 1 and 3). Four other TPTs, however, do show the siliceous TPT trend (P38, P39, P40, P45). Carpet PH, which showed nearly no siliceous TPTs for Areas 1 and 2, shows consistent relative proportions in calcareous TPT occurrences with Area 2 > Area 3 >> Area 1 for the nine most frequently occurring calcareous TPTs. Carpet BV shows negligibly low occurrences for most calcareous TPTs, and concurrence among the three areas for the four TPTs occurring at higher (though still low) levels (P27, P31*, P35* and P46).

[19] Two of these, BH and PH are the same carpets as shown in Table 24, one (BV), is not. These are examples of the data; full data are present in *Appendix K. Incidence of Target Particle Types*.

Table 24. Siliceous Particle Types from Different Areas of Carpets BH, PH and PV

| | | Carpet BH | | | Carpet PH | | | Carpet PV | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Area | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 |
| | Total Particles | 2948 | 4000 | 4000 | 1363 | 4000 | 4000 | 4000 | 4000 | 1005 |
| P3 | Si/Al Lower Si | 29 | 86 | 22 | 8 | 1 | 16 | 172 | 142 | 22 |
| P4 | Si/Al Medium Si | 28 | 47 | 18 | 0 | 0 | 0 | 28 | 14 | 5 |
| P6* | Si/Al/Ca | 13 | 26 | 12 | 6 | 3 | 11 | 77 | 132 | 32 |
| P8* | Si/Al/Fe | 11 | 35 | 10 | 0 | 0 | 20 | 125 | 170 | 32 |
| P11 | Si/Al/Mg Higher Mg | 4 | 10 | 5 | 0 | 1 | 7 | 26 | 21 | 12 |
| P12 | Si/Al/Mg Lower Mg | 16 | 27 | 6 | 1 | 1 | 6 | 63 | 69 | 9 |
| P13 | Al/Si/Na | 9 | 44 | 4 | 2 | 4 | 13 | 163 | 73 | 12 |
| P14 | Si/Al/Na Lower Si | 15 | 62 | 7 | 1 | 3 | 18 | 199 | 98 | 19 |
| P17 | Al/Si/W | 18 | 93 | 15 | 11 | 6 | 46 | 233 | 224 | 33 |
| P18 | Si/Al/S | 2 | 2 | 3 | 0 | 0 | 1 | 6 | 11 | 5 |
| P19 | Si/Al/Sr | 17 | 35 | 11 | 1 | 2 | 11 | 118 | 100 | 13 |
| P20 | Si/Al/Ca/Na | 1 | 7 | 1 | 5 | 2 | 7 | 20 | 13 | 1 |
| P21 | Ca/Si/Mg | 5 | 16 | 8 | 0 | 0 | 3 | 1 | 5 | 14 |
| P22 | Si/Ca/Mg | 8 | 29 | 4 | 1 | 0 | 9 | 34 | 54 | 12 |
| P23 | Si/Mg | 0 | 6 | 0 | 4 | 0 | 0 | 2 | 0 | 4 |
| P25 | Si/W | 9 | 32 | 6 | 3 | 4 | 28 | 64 | 35 | 7 |

Table 25. Calcareous Particle Types from Different Areas of Carpets BH, PH and BV

| | | Carpet BH | | | Carpet PH | | | Carpet BV | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Area | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 |
| | Total Particles | 2948 | 4000 | 4000 | 1363 | 4000 | 4000 | 1233 | 3613 | 1431 |
| P27 | Ca/Al | 51 | 47 | 29 | 29 | 251 | 163 | 23 | 19 | 23 |
| P28 | Ca/Bi | 11 | 8 | 9 | 14 | 71 | 34 | 4 | 6 | 1 |
| P29 | Ca/Br | 10 | 15 | 6 | 19 | 80 | 70 | 6 | 6 | 2 |
| P31* | Ca/Mg | 126 | 124 | 85 | 36 | 139 | 122 | 22 | 13 | 15 |
| P33 | Ca/Mg/Al | 33 | 45 | 18 | 0 | 8 | 16 | 2 | 5 | 1 |
| P34 | Ca/Mg/Na | 9 | 10 | 11 | 2 | 17 | 28 | 0 | 3 | 1 |
| P35* | Ca/Na | 73 | 100 | 34 | 160 | 675 | 541 | 26 | 27 | 9 |
| P38 | Na/Ca | 2 | 17 | 4 | 0 | 0 | 0 | 0 | 0 | 0 |
| P39 | Ca/Na/Al Higher Ca | 7 | 22 | 4 | 20 | 150 | 66 | 3 | 3 | 2 |
| P40 | Ca/Na/Al Lower Ca | 7 | 29 | 5 | 2 | 3 | 3 | 0 | 0 | 1 |
| P42 | Ca/Na/Br | 7 | 6 | 1 | 12 | 63 | 39 | 1 | 1 | 0 |
| P43 | Ca/Na/(Hg,W) | 0 | 4 | 2 | 1 | 6 | 8 | 0 | 0 | 0 |
| P44 | Na/Ca/Hg/Zr | 3 | 15 | 11 | 1 | 0 | 2 | 1 | 0 | 0 |
| P45 | Ca/Na/W | 8 | 26 | 4 | 25 | 108 | 85 | 1 | 2 | 1 |
| P46 | Ca/W | 33 | 37 | 28 | 31 | 175 | 114 | 11 | 10 | 7 |

Table 26 shows results for sulfurous, ferrous and other TPTs from three different areas of three of the carpets. Carpet BH is again included. These TPT occurrences show none of the trend seen for the siliceous particles (highest occurrence in Area 2, with comparable levels for Areas 1 and 3), and only one TPT (P67) shows the trend seen for calcareous particles (comparable Area 1 and 2, with lower Area 3). There is also a new "trend": much higher levels in Area 3 (for the TPTs with the four highest levels: P50*, P60, P63 and P68). As with the siliceous and calcareous TPTs, Carpet BH shows good qualitative agreement for low occurrences across all three areas. Carpet PL results are noteworthy for the high levels of one TPT (P50*), whose relative magnitude across the three areas follows the order, though not the proportion, of the total particle counts. Some other TPTs in these groups occur at lower levels in both Areas 2 and 3 (P58, P60, P63, P64 and P68). Carpet F, which has low total particle counts overall, shows very low occurrences for all of the TPTs, with the exception of Area 2, which shows one TPT at a high level (P66) and one at a low level (P63).

The trends illustrated in these examples continue throughout the dataset in Appendix K. For some carpets, and some TPTs, occurrences are highly localized: present in only one of three carpet areas sampled, or present in grossly different amounts among the three areas.[20] This occurred regularly. Among the 47 TPTs used, and the nine carpets, 55 clear instances were observed (13.0%). For many other TPTs, in these same carpets, comparable occurrences were much more frequently observed, some of which showed a regular ranking following the total number of particles. Comparable occurrences across the three carpet three areas were observed in 176 clear instances (41.6%).[21] Comparable absences also occurred frequently, with some 141 instances (33.3%).[22] Overall, comparable results across the three carpet areas occurred approximately 75% of the time.

[20] Grossly different is taken here as a difference of greater than an order of magnitude, after normalizing the occurrence frequencies to a total particle count of 4000 (the maximum number analyzed as set by computer-assisted SEM/EDS parameters). See *Appendix K. Incidence of Target Particle Types*, Table K3A for the normalized frequencies.

[21] Comparable occurrences (in frequencies per 4000) were taken as those where the minimum of the three area values and the maximum differed by less than a factor of 5.

[22] Comparable absences (in frequencies per 4000) were taken as those where all three areas had frequencies of 10 or less.

Table 26. Sulfurous, Ferrous and Other Particle Types from Different Areas of Carpets BH, PL and F.

| | | | Carpet BH | | | Carpet PL | | | Carpet F | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Area | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 | Area 1 | Area 2 | Area 3 |
| | | Total Particles | 2948 | 4000 | 4000 | 4000 | 2169 | 3662 | 445 | 1179 | 359 |
| Sulfurous | P47 | Ba/S/Bi | 2 | 4 | 1 | 0 | 0 | 2 | 1 | 2 | 2 |
| | P48 | Ba/S/Na | 1 | 4 | 0 | 0 | 1 | 0 | 0 | 0 | 1 |
| | P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P50* | Ca/S | 18 | 30 | 191 | 2797 | 310 | 931 | 3 | 5 | 4 |
| | P57 | Na/Ca/S | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P58 | Na/S/Ca/Bi | 129 | 37 | 25 | 8 | 22 | 18 | 0 | 1 | 2 |
| | P59 | Na/S/Hg | 9 | 19 | 10 | 0 | 2 | 0 | 0 | 0 | 0 |
| Ferrous | P60 | Fe | 8 | 8 | 220 | 1 | 11 | 13 | 1 | 10 | 8 |
| | P61 | Fe/Al | 2 | 8 | 28 | 0 | 8 | 8 | 3 | 8 | 3 |
| | P62 | Fe/Cr/Na | 16 | 30 | 2 | 5 | 8 | 1 | 2 | 2 | 1 |
| | P63 | Fe/Na | 26 | 36 | 401 | 4 | 14 | 23 | 6 | 16 | 4 |
| | P64 | Fe/Al/Si | 10 | 29 | 6 | 2 | 10 | 18 | 0 | 3 | 0 |
| Other | P65 | Cl/Na/Ca | 44 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 136 | 0 |
| | P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 497 | 373 | 174 | 2 | 1 | 3 | 3 | 2 | 1 |
| | P68 | Ti/(Al,La,Na) | 26 | 39 | 579 | 3 | 15 | 12 | 2 | 10 | 3 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

H. Variation Within Single Carpet Areas

Within-carpet variation was tested using TPT occurrences for three individual carpet fibers from each of three carpet areas in nine carpets. Tables 27 to 29 illustrate trends in the results, using selected TPT occurrences for six of the carpets.[23]

Table 27 shows selected results for residential Carpet A and residential Carpet BH, featuring calcareous, sulfurous, ferrous and other TPTs. For Carpet A some individual fibers from Areas 1 and 3 show very low particle totals, with values for total particles of 67, 85, 161 and 164. Area 2 makes a striking contrast with ten-fold higher values. For Area 2 almost all TPTs found in the 10-Set are seen well above the LOD in the single fibers, and most of the levels in the single fibers follow the ranking of the total particle values: > Fiber 3 > Fiber 2 > Fiber 1. Two of the Carpet A fibers, with particle numbers in the 300-500 range, show occurrences near the LOD that closely follow the 10-Sets: Fiber 3 in Area 1 and Fiber 2 in Area 3.

For Carpet BH in Table 27, Area 3 shows one fiber (Fiber 1) with abundant total particles (4000, the maximum measured) and two others with 504 and 234 total particles. Four of the TPTs for this area (P60, P61, P63 and P67) show a reasonable proportionality following these total particle numbers. In contrast, another particle type (P68) shows exceptionally high levels in both the Area 3 10-Set and in Fiber 1, but has no occurrence in Fibers 2 and 3. Similarly, in Areas 1 and 2, TPT P67 shows high levels in the 10-Sets, while individual fibers show low or insignificant levels. For TPT P62 four of the individual fibers (with low overall particle numbers) show higher occurrences for TPTs than do their corresponding 10-Sets (Area 1, Fibers 2, 3; Area 4, Fibers 1, 3).

Table 28 shows selected results for residential Carpet PH and workplace Carpet PV, featuring calcareous and sulfurous TPTs. For Carpet PH (as with Carpet A in Table 27) some individual fibers from Areas 1 and 2 show very low particle totals. The two fibers with particle totals below 100 (Area 1, Fibers 1, 2) show insignificant levels of the TPTs. With the somewhat higher particle totals, occurrences are consistently seen in the individual fibers for those TPTs showing the highest levels in the Area 10-Sets.[24] With the still higher individual fiber particle total seen in Area 3, Fiber 3, levels are seen for the five highest TPTs occurring in the Area 10-Set. For the highest individual fiber total particle level (4000, seen in Area 2, Fiber 3) the TPT occurrences closely follow those seen in the 10-Set.

For Carpet PL there is a single TPT (P50*) that occurs at high to very high levels in all three Area 10-Sets. This is seen in all individual fibers whose total particle numbers are 375 or above.

Table 29 shows selected results for vehicular Carpet PV and workplace Carpet W, featuring siliceous TPTs. Carpet PV, Area 2 shows high total particle levels for each of the three individual fibers, and the levels of the TPTs seen are very close to those in the Area 10-Set. For Area 3, the total particle levels are lower for both the 10-Set (1005) and the individual fibers. Fiber 3, with only 330 total particles, shows insignificant levels of TPT occurrence, while Fibers 1 and 2, with 2 to 3 times this number, show levels in reasonably close agreement with the

[23] *Appendix K. Incidence of Target Particle Types* contains the full set of data.

[24] Specifically, (1) Particle Type P35* levels for Area 1, Fiber 3; Area 2, Fibers 1, 2; Area 3, Fibers 1, 2, and (2) Particle Type P27 levels for Area 2, Fiber 2 and Area 3, Fiber 2.

10-Set. Area 1 shows a 10-Set with the maximum total particle count and individual fibers in the 400 to 800 range. TPT occurrences are very low for the individual fibers, although the particle type occurring at the highest level in the 10-Set (P17) also shows the highest occurrence in the three individual fibers. Fiber 3 shows similar occurrences for TPTs P3, P12 and P25, but lacks other occurrences that would be expected if the effect were proportional (e.g. higher levels for P8*, P13 and P14).

Each of the Areas for Carpet W show lower TPT occurrences for individual fibers, ranging from 150 to 535. Area 3 also shows low values for the fiber 10-Set. Even so, Fiber 1 shows levels above the LOD for TPT P6*, the type with the highest level of occurrence in the Area 3 10-Set. Fiber 2 from Area 1 shows the same, as do Fibers 2 and 3 from Area 2. These fibers from Area 2 also show levels above the LOD for TPT P3, the type with the second highest level of occurrence in the Area 2 10-Set.

Fourteen individual fibers had total particle counts greater than 1000. The TPT occurrences for these fibers are shown in Tables 30 and 31, together with the results for the corresponding carpet areas. With few exceptions, the TPT occurrences from these fibers closely follow those from their originating area. The patterns of TPTs are highly characteristic and show both qualitative similarity and similarities in rank and proportion.. Ten of the fibers show a very close correspondence. Three show differences in one TPT[25] and one shows a number of differences in the concentrations of calcareous TPTs.[26]

[25] High levels of P62 for Carpet BV, Area 1, Fiber 2 and for Carpet PV, Area 2, Fiber 3. High levels of P67 for Carpet BV, Area 2, which are not seen in the fibers.
[26] Higher levels of approximately half of the calcareous particles are seen in Carpet PH, Area 3 compared to Carpet PH, Area 3, Fiber 3.

Table 27. Within-Carpet Area Results for Selected Target Particle Types from Residential Carpets A and BH

| | | Carpet A | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 3001 | 164 | 161 | 490 | 4000 | 872 | 1110 | 1782 | 4000 | 67 | 306 | 85 |
| Calcareous | P31* | Ca/Mg | 51 | 1 | 0 | 1 | 260 | 62 | 64 | 80 | 108 | 0 | 10 | 1 |
| | P33 | Ca/Mg/Al | 20 | 1 | 0 | 2 | 52 | 27 | 22 | 22 | 29 | 0 | 2 | 2 |
| | P34 | Ca/Mg/Na | 13 | 1 | 0 | 1 | 111 | 8 | 33 | 48 | 15 | 0 | 2 | 0 |
| | P35* | Ca/Na | 145 | 4 | 2 | 6 | 56 | 12 | 27 | 27 | 129 | 2 | 10 | 3 |
| Sulfurous | P50* | Ca/S | 366 | 9 | 1 | 12 | 67 | 28 | 24 | 130 | 131 | 1 | 7 | 0 |
| | P57 | Na/Ca/S | 0 | 0 | 0 | 0 | 115 | 15 | 37 | 40 | 26 | 0 | 3 | 2 |
| | P58 | Na/S/Ca/Bi | 175 | 5 | 1 | 11 | 383 | 37 | 70 | 119 | 119 | 1 | 16 | 3 |
| | P59 | Na/S/Hg | 19 | 0 | 0 | 2 | 167 | 12 | 18 | 60 | 18 | 1 | 9 | 0 |

| | | Carpet BH | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 2948 | 244 | 345 | 239 | 4000 | 421 | 312 | 691 | 4000 | 4000 | 504 | 234 |
| Ferrous | P60 | Fe | 8 | 0 | 2 | 1 | 8 | 1 | 0 | 4 | 220 | 165 | 30 | 7 |
| | P61 | Fe/Al | 2 | 1 | 0 | 3 | 8 | 4 | 1 | 2 | 28 | 21 | 5 | 0 |
| | P62 | Fe/Cr/Na | 16 | 13 | 76 | 47 | 30 | 42 | 9 | 87 | 2 | 7 | 8 | 23 |
| | P63 | Fe/Na | 26 | 2 | 3 | 1 | 36 | 6 | 1 | 8 | 401 | 233 | 22 | 16 |
| | P64 | Fe/Al/Si | 10 | 0 | 0 | 0 | 29 | 2 | 1 | 2 | 6 | 6 | 0 | 1 |
| Other | P65 | Cl/Na/Ca | 44 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 497 | 10 | 25 | 3 | 373 | 2 | 7 | 2 | 174 | 94 | 28 | 4 |
| | P68 | Ti/(Al,La,Na) | 26 | 3 | 2 | 1 | 39 | 4 | 0 | 0 | 579 | 1020 | 0 | 0 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

Table 28. Within-Carpet Area Results for Selected Target Particle Types from Residential Carpet PH and Workplace Carpet PL

| | | Carpet PH | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 1363 | 70 | 59 | 205 | 4000 | 137 | 147 | 4000 | 4000 | 504 | 711 | 1568 |
| Calcareous | P27 | Ca/Al | 29 | 1 | 2 | 0 | 251 | 3 | 12 | 295 | 163 | 2 | 22 | 21 |
| | P28 | Ca/Bi | 14 | 0 | 0 | 1 | 71 | 1 | 1 | 71 | 34 | 2 | 5 | 16 |
| | P29 | Ca/Br | 19 | 0 | 0 | 2 | 80 | 4 | 6 | 124 | 70 | 1 | 4 | 13 |
| | P31* | Ca/Mg | 36 | 1 | 1 | 1 | 139 | 1 | 2 | 181 | 122 | 1 | 5 | 18 |
| | P33 | Ca/Mg/Al | 0 | 0 | 0 | 0 | 8 | 0 | 0 | 21 | 16 | 0 | 0 | 2 |
| | P34 | Ca/Mg/Na | 2 | 0 | 0 | 0 | 17 | 0 | 0 | 7 | 28 | 1 | 0 | 2 |
| | P35* | Ca/Na | 160 | 0 | 4 | 14 | 675 | 13 | 23 | 756 | 541 | 16 | 36 | 60 |
| | | Carpet PL | Area 1 | | | | Area 2 | | | | Area 3 | | | |
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 4000 | 1492 | 1742 | 848 | 2169 | 505 | 494 | 518 | 3662 | 375 | 692 | 454 |
| Sulfurous | P47 | Ba/S/Bi | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 0 | 0 | 0 |
| | P48 | Ba/S/Na | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 1 | 0 | 1 | 1 | 0 |
| | P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P50* | Ca/S | 2797 | 746 | 633 | 243 | 310 | 22 | 31 | 15 | 931 | 50 | 25 | 79 |
| | P57 | Na/Ca/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P58 | Na/S/Ca/Bi | 8 | 6 | 3 | 0 | 22 | 1 | 2 | 1 | 18 | 1 | 4 | 0 |
| | P59 | Na/S/Hg | 0 | 1 | 0 | 0 | 2 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

Table 29. Within-Carpet Area Results for Selected Target Particle Types from Vehicular Carpet PV and Workplace Carpet W

| | | Carpet PV | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 4000 | 809 | 438 | 604 | 4000 | 4000 | 4000 | 3779 | 1005 | 674 | 950 | 330 |
| Siliceous | P3 | Si/Al Lower Si | 172 | 3 | 3 | 16 | 142 | 93 | 119 | 122 | 22 | 27 | 17 | 5 |
| | P4 | Si/Al Medium Si | 28 | 1 | 0 | 2 | 14 | 13 | 19 | 18 | 5 | 5 | 1 | 0 |
| | P6* | Si/Al/Ca | 77 | 4 | 6 | 7 | 132 | 97 | 101 | 96 | 32 | 11 | 7 | 7 |
| | P8* | Si/Al/Fe | 125 | 4 | 1 | 7 | 170 | 71 | 155 | 131 | 32 | 31 | 19 | 1 |
| | P11 | Si/Al/Mg Higher Mg | 26 | 3 | 0 | 1 | 21 | 21 | 24 | 21 | 12 | 3 | 3 | 0 |
| | P12 | Si/Al/Mg Lower Mg | 63 | 2 | 2 | 11 | 69 | 40 | 41 | 61 | 9 | 5 | 14 | 1 |
| | P13 | Al/Si/Na | 163 | 5 | 3 | 5 | 73 | 98 | 65 | 89 | 12 | 16 | 10 | 4 |
| | P14 | Si/Al/Na Lower Si | 199 | 9 | 8 | 10 | 98 | 111 | 119 | 94 | 19 | 18 | 13 | 3 |
| | P17 | Al/Si/W | 233 | 11 | 8 | 25 | 224 | 168 | 137 | 154 | 33 | 26 | 22 | 4 |

| | | Carpet W | Area 1 | | | | Area 2 | | | | Area 3 | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 |
| | | Total Particles | 2896 | 514 | 464 | 535 | 4000 | 419 | 367 | 280 | 710 | 320 | 258 | 152 |
| Siliceous | P3 | Si/Al Lower Si | 67 | 4 | 11 | 4 | 203 | 8 | 15 | 4 | 25 | 8 | 6 | 3 |
| | P4 | Si/Al Medium Si | 9 | 1 | 4 | 2 | 19 | 1 | 1 | 0 | 3 | 1 | 2 | 0 |
| | P6* | Si/Al/Ca | 143 | 7 | 15 | 9 | 291 | 49 | 41 | 8 | 71 | 8 | 2 | 4 |
| | P8* | Si/Al/Fe | 35 | 4 | 3 | 2 | 125 | 5 | 6 | 0 | 11 | 3 | 0 | 0 |
| | P11 | Si/Al/Mg Higher Mg | 15 | 0 | 5 | 2 | 35 | 1 | 4 | 1 | 2 | 0 | 1 | 0 |
| | P12 | Si/Al/Mg Lower Mg | 20 | 0 | 2 | 0 | 80 | 4 | 2 | 1 | 11 | 0 | 1 | 0 |
| | P13 | Al/Si/Na | 91 | 5 | 9 | 2 | 7 | 0 | 1 | 0 | 1 | 0 | 0 | 0 |
| | P14 | Si/Al/Na Lower Si | 117 | 7 | 12 | 11 | 62 | 3 | 0 | 4 | 8 | 2 | 1 | 0 |
| | P17 | Al/Si/W | 34 | 1 | 4 | 2 | 60 | 2 | 4 | 0 | 4 | 1 | 1 | 1 |

| dark gray | 0 to 5 | gray | 6 to 10 | yellow | 11 to 50 | gold | 51 to 200 | red | > 200 |
|---|---|---|---|---|---|---|---|---|---|

Table 30. Within-Carpet Area Results: Fibers with More Than 1000 Particles (1 of 2)

| | | Carpet A | | | Carpet BH | | Carpet PH | | Carpet PH | | Carpet BV | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Target Particle Type | | Area 2 | | | Area 3 | | Area 2 | | Area 3 | | Area 1 | |
| Type | Description | 10-Set | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | 10-Set | Fiber 3 | 10-Set | Fiber 3 | 10-Set | Fiber 2 |
| P3 | Si/Al Lower Si | 34 | 18 | 28 | 22 | 14 | 1 | 1 | 16 | 27 | 65 | 14 |
| P4 | Si/Al Medium Si | 82 | 33 | 26 | 18 | 18 | 0 | 2 | 0 | 2 | 13 | 4 |
| P6* | Si/Al/Ca | 17 | 10 | 6 | 12 | 7 | 3 | 0 | 11 | 17 | 51 | 21 |
| P8* | Si/Al/Fe | 8 | 7 | 12 | 10 | 11 | 0 | 1 | 20 | 11 | 15 | 7 |
| P11 | Si/Al/Mg Higher Mg | 2 | 3 | 5 | 5 | 1 | 1 | 0 | 7 | 2 | 6 | 5 |
| P12 | Si/Al/Mg Lower Mg | 8 | 4 | 0 | 6 | 5 | 1 | 0 | 6 | 4 | 11 | 10 |
| P13 | Al/Si/Na | 28 | 14 | 13 | 4 | 17 | 4 | 2 | 13 | 13 | 5 | 4 |
| P14 | Si/Al/Na Lower Si | 25 | 8 | 21 | 7 | 20 | 3 | 3 | 18 | 16 | 24 | 14 |
| P17 | Al/Si/W | 3 | 2 | 1 | 15 | 23 | 6 | 10 | 46 | 27 | 37 | 20 |
| P18 | Si/Al/S | 0 | 3 | 0 | 3 | 2 | 0 | 0 | 1 | 3 | 26 | 6 |
| P19 | Si/Al/Sr | 30 | 12 | 21 | 11 | 11 | 2 | 1 | 11 | 8 | 23 | 10 |
| P20 | Si/Al/Ca/Na | 4 | 5 | 8 | 1 | 4 | 2 | 0 | 7 | 2 | 2 | 2 |
| P21 | Ca/Si/Mg | 13 | 3 | 4 | 8 | 5 | 0 | 3 | 3 | 2 | 0 | 1 |
| P22 | Si/Ca/Mg | 10 | 3 | 1 | 4 | 9 | 0 | 1 | 9 | 5 | 16 | 6 |
| P23 | Si/Mg | 12 | 1 | 2 | 0 | 0 | 0 | 0 | 0 | 6 | 3 | 1 |
| P25 | Si/W | 3 | 2 | 2 | 6 | 8 | 4 | 1 | 28 | 10 | 14 | 13 |
| P27 | Ca/Al | 27 | 7 | 12 | 29 | 22 | 251 | 295 | 163 | 21 | 23 | 7 |
| P28 | Ca/Bi | 4 | 0 | 0 | 9 | 5 | 71 | 71 | 34 | 16 | 4 | 3 |
| P29 | Ca/Br | 2 | 1 | 1 | 6 | 3 | 80 | 124 | 70 | 13 | 6 | 4 |
| P31* | Ca/Mg | 260 | 64 | 80 | 85 | 53 | 139 | 181 | 122 | 18 | 22 | 8 |
| P33 | Ca/Mg/Al | 52 | 22 | 22 | 18 | 22 | 8 | 21 | 16 | 2 | 2 | 3 |
| P34 | Ca/Mg/Na | 111 | 33 | 48 | 11 | 3 | 17 | 7 | 28 | 2 | 0 | 0 |
| P35* | Ca/Na | 56 | 27 | 27 | 34 | 29 | 675 | 756 | 541 | 60 | 26 | 18 |
| P38 | Na/Ca | 91 | 11 | 34 | 4 | 2 | 0 | 0 | 0 | 1 | 0 | 0 |
| P39 | Ca/Na/Al Higher Ca | 8 | 1 | 3 | 4 | 4 | 150 | 139 | 66 | 15 | 3 | 1 |
| P40 | Ca/Na/Al Lower Ca | 143 | 23 | 59 | 5 | 6 | 3 | 1 | 3 | 2 | 0 | 0 |
| P42 | Ca/Na/Br | 1 | 0 | 0 | 1 | 1 | 63 | 63 | 39 | 10 | 1 | 1 |
| P43 | Ca/Na/(Hg,W) | 36 | 11 | 21 | 2 | 1 | 6 | 2 | 8 | 3 | 0 | 0 |
| P44 | Na/Ca/Hg/Zr | 8 | 0 | 0 | 11 | 4 | 0 | 0 | 2 | 0 | 1 | 0 |
| P45 | Ca/Na/W | 5 | 2 | 0 | 4 | 6 | 108 | 94 | 85 | 8 | 1 | 1 |
| P46 | Ca/W | 11 | 5 | 6 | 28 | 10 | 175 | 207 | 114 | 15 | 11 | 12 |
| P47 | Ba/S/Bi | 0 | 0 | 0 | 1 | 1 | 96 | 66 | 62 | 3 | 1 | 1 |
| P48 | Ba/S/Na | 2 | 0 | 0 | 0 | 0 | 87 | 45 | 59 | 5 | 0 | 0 |
| P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 5 | 5 | 15 | 2 | 0 | 0 |
| P50* | Ca/S | 67 | 24 | 130 | 191 | 94 | 15 | 0 | 19 | 18 | 13 | 6 |
| P57 | Na/Ca/S | 115 | 37 | 40 | 1 | 0 | 0 | 1 | 4 | 0 | 0 | 0 |
| P58 | Na/S/Ca/Bi | 383 | 70 | 119 | 25 | 10 | 9 | 2 | 33 | 14 | 1 | 1 |
| P59 | Na/S/Hg | 167 | 18 | 60 | 10 | 9 | 4 | 0 | 27 | 0 | 0 | 0 |
| P60 | Fe | 0 | 0 | 0 | 220 | 165 | 0 | 0 | 1 | 2 | 2 | 7 |
| P61 | Fe/Al | 0 | 0 | 1 | 28 | 21 | 1 | 0 | 1 | 1 | 3 | 2 |
| P62 | Fe/Cr/Na | 2 | 0 | 2 | 2 | 7 | 1 | 2 | 3 | 2 | 2 | 171 |
| P63 | Fe/Na | 2 | 13 | 2 | 401 | 233 | 4 | 1 | 8 | 7 | 3 | 0 |
| P64 | Fe/Al/Si | 3 | 1 | 0 | 6 | 6 | 1 | 0 | 7 | 3 | 7 | 3 |
| P65 | Cl/Na/Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 6 | 1 | 3 | 174 | 94 | 2 | 1 | 5 | 1 | 3 | 0 |
| P68 | Ti/(Al,La,Na) | 5 | 1 | 1 | 579 | 1020 | 9 | 2 | 15 | 47 | 3 | 4 |
| | Total Particles | 4000 | 1110 | 1782 | 4000 | 4000 | 4000 | 4000 | 4000 | 1568 | 1233 | 1107 |

Table 31. Within-Carpet Area Results: Fibers with More Than 1000 Particles (2 of 2)

| | | Carpet BV | | | | Carpet PV | | | | Carpet PL | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Target Particle Type | | Area 2 | | | | Area 2 | | | | Area 1 | | |
| Type | Description | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 | Fiber 3 | 10-Set | Fiber 1 | Fiber 2 |
| P3 | Si/Al Lower Si | 70 | 22 | 15 | 24 | 142 | 93 | 119 | 122 | 19 | 7 | 15 |
| P4 | Si/Al Medium Si | 26 | 16 | 7 | 12 | 14 | 13 | 19 | 18 | 5 | 2 | 8 |
| P6* | Si/Al/Ca | 100 | 24 | 51 | 43 | 132 | 97 | 101 | 96 | 33 | 7 | 42 |
| P8* | Si/Al/Fe | 35 | 9 | 13 | 7 | 170 | 71 | 155 | 131 | 10 | 1 | 3 |
| P11 | Si/Al/Mg Higher Mg | 56 | 59 | 63 | 54 | 21 | 21 | 24 | 21 | 7 | 3 | 6 |
| P12 | Si/Al/Mg Lower Mg | 24 | 6 | 3 | 14 | 69 | 40 | 41 | 61 | 5 | 2 | 9 |
| P13 | Al/Si/Na | 21 | 23 | 8 | 7 | 73 | 98 | 65 | 89 | 6 | 6 | 7 |
| P14 | Si/Al/Na Lower Si | 48 | 21 | 13 | 15 | 98 | 111 | 119 | 94 | 15 | 4 | 15 |
| P17 | Al/Si/W | 53 | 10 | 22 | 22 | 224 | 168 | 137 | 154 | 16 | 4 | 17 |
| P18 | Si/Al/S | 13 | 1 | 0 | 3 | 11 | 6 | 12 | 6 | 4 | 2 | 1 |
| P19 | Si/Al/Sr | 35 | 13 | 11 | 11 | 100 | 72 | 69 | 80 | 8 | 3 | 9 |
| P20 | Si/Al/Ca/Na | 6 | 1 | 7 | 0 | 13 | 19 | 15 | 18 | 1 | 1 | 0 |
| P21 | Ca/Si/Mg | 3 | 3 | 4 | 2 | 5 | 4 | 5 | 4 | 3 | 0 | 1 |
| P22 | Si/Ca/Mg | 16 | 1 | 6 | 8 | 54 | 33 | 47 | 45 | 12 | 0 | 1 |
| P23 | Si/Mg | 5 | 1 | 0 | 3 | 0 | 4 | 3 | 18 | 3 | 3 | 3 |
| P25 | Si/W | 69 | 26 | 33 | 46 | 35 | 48 | 34 | 22 | 14 | 26 | 31 |
| P27 | Ca/Al | 19 | 8 | 12 | 8 | 63 | 40 | 51 | 49 | 23 | 8 | 20 |
| P28 | Ca/Bi | 6 | 3 | 2 | 3 | 8 | 9 | 9 | 11 | 1 | 2 | 2 |
| P29 | Ca/Br | 6 | 0 | 0 | 1 | 7 | 15 | 13 | 8 | 8 | 2 | 7 |
| P31* | Ca/Mg | 13 | 5 | 3 | 6 | 44 | 28 | 37 | 27 | 63 | 10 | 15 |
| P33 | Ca/Mg/Al | 5 | 1 | 0 | 0 | 21 | 15 | 15 | 13 | 9 | 5 | 3 |
| P34 | Ca/Mg/Na | 3 | 0 | 1 | 1 | 4 | 9 | 4 | 7 | 2 | 0 | 1 |
| P35* | Ca/Na | 27 | 6 | 8 | 9 | 57 | 68 | 71 | 43 | 23 | 14 | 24 |
| P38 | Na/Ca | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 |
| P39 | Ca/Na/Al Higher Ca | 3 | 0 | 1 | 0 | 21 | 31 | 20 | 14 | 4 | 0 | 4 |
| P40 | Ca/Na/Al Lower Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 0 |
| P42 | Ca/Na/Br | 1 | 0 | 0 | 0 | 4 | 5 | 4 | 4 | 3 | 0 | 0 |
| P43 | Ca/Na/(Hg,W) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P44 | Na/Ca/Hg/Zr | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P45 | Ca/Na/W | 2 | 1 | 1 | 0 | 6 | 21 | 11 | 8 | 0 | 0 | 1 |
| P46 | Ca/W | 10 | 4 | 5 | 3 | 19 | 23 | 22 | 21 | 14 | 4 | 10 |
| P47 | Ba/S/Bi | 0 | 0 | 1 | 0 | 4 | 2 | 4 | 2 | 0 | 0 | 0 |
| P48 | Ba/S/Na | 0 | 0 | 1 | 0 | 4 | 3 | 5 | 0 | 0 | 0 | 0 |
| P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P50* | Ca/S | 24 | 11 | 11 | 8 | 92 | 76 | 58 | 85 | 2797 | 746 | 633 |
| P57 | Na/Ca/S | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 |
| P58 | Na/S/Ca/Bi | 1 | 0 | 2 | 0 | 5 | 24 | 7 | 13 | 8 | 6 | 3 |
| P59 | Na/S/Hg | 0 | 0 | 0 | 0 | 0 | 9 | 0 | 1 | 0 | 1 | 0 |
| P60 | Fe | 24 | 5 | 7 | 7 | 6 | 5 | 8 | 11 | 1 | 3 | 5 |
| P61 | Fe/Al | 24 | 4 | 12 | 7 | 16 | 4 | 18 | 14 | 0 | 3 | 2 |
| P62 | Fe/Cr/Na | 7 | 7 | 12 | 8 | 1 | 7 | 12 | 106 | 5 | 5 | 5 |
| P63 | Fe/Na | 52 | 6 | 12 | 17 | 21 | 17 | 38 | 19 | 4 | 10 | 9 |
| P64 | Fe/Al/Si | 10 | 3 | 5 | 3 | 106 | 46 | 89 | 57 | 2 | 2 | 3 |
| P65 | Cl/Na/Ca | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 51 | 1 | 1 | 5 | 0 | 5 | 3 | 2 | 2 | 0 | 0 |
| P68 | Ti/(Al,La,Na) | 10 | 2 | 0 | 3 | 19 | 19 | 35 | 25 | 3 | 2 | 0 |
| | Total Particles | 3613 | 1500 | 1757 | 2121 | 4000 | 4000 | 4000 | 3779 | 4000 | 1492 | 1742 |

I. Variation Among Different Carpets

The frequencies of occurrence of TPTs in a sampling of 21 carpets are shown in Table 32. Frequencies have been calculated per 4000 particles, the maximum number analyzed as set by computer-assisted SEM/EDS parameters. Average values of the three separate carpet areas are given for the carpets used for study of within-carpet variation.[27]

Some TPTs occur at high levels in only one to a few of the carpets. Two distinct examples are P57 (Carpet A) and P66 (Carpet F). Additional examples are P23, P34, P38, P40, P42 and P45. Three TPTs occur at low levels, but in only one or two carpets: P44 (Carpet BH), P49 (Carpet PH) and P65 (Carpet BH).

Some TPTs occur at moderate to high levels in several carpets, but are at very low levels in most. Examples are P39, P47, P48 and P67.

Some TPTs occur over a wide range of levels across the set of carpets. Notable examples are P31*, P35* and P50*. Other TPTs show a narrower range of levels, present in moderate to high levels in most of the carpets. Examples are P3 and P19.

Many of the carpets show a characteristic profile of TPTs distinguishing them from most others in this set of 21 carpets, based either on qualitative differences or large differences in the frequencies of specific TPTs.

Carpet PH stands out with high occurrences of calcareous TPTs containing sodium, and very low levels of siliceous and ferrous TPTs. There are very high levels of P35* and the highest observed levels of four other sodium containing calcareous TPTs: P39, P42, P45 and P46. Carpet PH is also one of the few carpets with occurrences of the two sulfurous TPTs, P47 and P48.

Carpet R5 is distinguished by high levels of TPTs P23, P50* and P58.

Carpet R3 has very high levels of TPTs P31* and P58, along with very low levels of P20 to P23. It is also one of the few carpets with co-occurrences of TPTs P39, P42 and P45.

Carpet F is the only one with very high levels of TPT P66 and is further characterized by only low levels of P50* and moderate levels of P23. The only other carpet with levels of TPT P66 is Carpet V1, which is further characterized by moderate levels of the three sulfurous TPTs P47, P50* and P58.

Carpet BH has very high levels of both TPT P67 and P68 as well as the highest levels in two of the ferrous TPTs, P60 and P63.

Carpet A is distinguished by its sulfurous and calcareous particles. It is the only carpet with occurrences of TPT P57, one of the few carpets with high levels of both TPTs P50* and P58, and

[27] Specific occurrence data for all Carpet Areas and unconsolidated Target Particle Types are present in *Appendix K. Incidence of Target Particle Types*.

has the highest occurrence of P59. Carpet A also has the highest levels of occurrence for the rarely occurring calcareous TPTs P40 and P43.

Apart from Carpets BH and R5 (already distinguished by other TPTs), there are only two other carpets among the 21 with high levels of TPT P67: Carpets R7 and BV. These two are further distinguished from each other by their levels of P58, which are very high for Carpet R7 and negligible for Carpet BV.

Carpets R2 and W are the only ones with very low occurrences of TPTs P57 through P63. They are distinguishable from each other in their levels of occurrence of TPT 50* and differences in a number of TPTs where one carpet has very low or negligible occurrences and the other has low levels: P11, P13, P18, P20, P21, P28 and P29.

Carpet R1 has very high levels of TPTs P50* and P58, along with moderate levels of P59, very low levels of P47, and no occurrences of P67. Carpet R6 also has high levels of P50*, but moderate levels of P58, and, in contrast with Carpet R1, low levels of P67 and insignificant levels of P59.

Of the eight carpets not specifically mentioned above, additional discrimination is clearly possible using, for example, the broad range of frequencies seen for TPT 50*, and the ranges seen among several of the siliceous TPTs including P3, P13, P17 and P20.

Table 32. Frequency of Occurrence (per 4000 Particles) of Target Particle Types in 21 Carpets[28]

| | | | Residential | | | | | | | | | | Vehicular | | | | | | | | Workplace | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Carpet | R1 | R2 | R3 | R4 | R5 | R6 | R7 | A | BH | PH | V1 | V2 | V3 | V4 | V5 | BV | F | PV | MT | PL | W |
| | | Total Particles | 642 | 4000 | 4000 | 3206 | 4000 | 4000 | 4000 | 3667 | 3649 | 3121 | 3422 | 1803 | 3485 | 1030 | 518 | 2092 | 661 | 3002 | 2771 | 3277 | 2535 |
| Siliceous | P3 | Si/Al Lower Si | 150 | 192 | 80 | 302 | 72 | 147 | 138 | 82 | 50 | 11 | 144 | 211 | 216 | 163 | 154 | 126 | 129 | 149 | 105 | 68 | 155 |
| | P4 | Si/Al Medium Si | 12 | 15 | 29 | 42 | 14 | 36 | 80 | 53 | 34 | 0 | 19 | 35 | 55 | 31 | 31 | 31 | 8 | 21 | 18 | 12 | 16 |
| | P6* | Si/Al/Ca | 87 | 161 | 36 | 116 | 62 | 96 | 67 | 44 | 19 | 9 | 141 | 120 | 168 | 62 | 154 | 134 | 63 | 107 | 195 | 64 | 266 |
| | P8* | Si/Al/Fe | 25 | 49 | 31 | 76 | 42 | 56 | 56 | 55 | 20 | 9 | 46 | 106 | 67 | 62 | 100 | 41 | 34 | 145 | 48 | 24 | 90 |
| | P11 | Si/Al/Mg Higher Mg | 6 | 7 | 8 | 15 | 27 | 20 | 5 | 23 | 7 | 3 | 9 | 38 | 16 | 12 | 23 | 48 | 28 | 26 | 14 | 12 | 27 |
| | P12 | Si/Al/Mg Lower Mg | 25 | 16 | 15 | 39 | 25 | 29 | 29 | 33 | 18 | 3 | 30 | 95 | 62 | 35 | 46 | 32 | 18 | 63 | 30 | 15 | 58 |
| | P13 | Al/Si/Na | 0 | 7 | 6 | 130 | 75 | 24 | 61 | 61 | 21 | 8 | 14 | 16 | 38 | 0 | 8 | 21 | 26 | 110 | 66 | 14 | 52 |
| | P14 | Si/Al/Na Lower Si | 19 | 55 | 27 | 77 | 27 | 70 | 31 | 77 | 31 | 9 | 30 | 67 | 84 | 31 | 23 | 59 | 44 | 140 | 110 | 36 | 98 |
| | P17 | Al/Si/W | 19 | 34 | 27 | 64 | 32 | 38 | 39 | 29 | 46 | 27 | 35 | 67 | 79 | 39 | 54 | 79 | 2 | 218 | 119 | 44 | 52 |
| | P18 | Si/Al/S | 12 | 2 | 17 | 25 | 11 | 12 | 30 | 4 | 3 | 0 | 22 | 13 | 10 | 16 | 39 | 29 | 4 | 10 | 19 | 7 | 14 |
| | P19 | Si/Al/Sr | 50 | 98 | 78 | 143 | 55 | 76 | 80 | 54 | 23 | 6 | 98 | 111 | 135 | 117 | 108 | 48 | 36 | 103 | 62 | 25 | 101 |
| | P20 | Si/Al/Ca/Na | 6 | 5 | 5 | 2 | 6 | 7 | 8 | 21 | 3 | 6 | 5 | 2 | 23 | 0 | 0 | 5 | 0 | 15 | 34 | 3 | 28 |
| | P21 | Ca/Si/Mg | 0 | 27 | 8 | 2 | 3 | 2 | 13 | 6 | 11 | 1 | 1 | 4 | 0 | 0 | 8 | 2 | 8 | 9 | 3 | 4 | 3 |
| | P22 | Si/Ca/Mg | 12 | 43 | 5 | 30 | 31 | 46 | 14 | 32 | 15 | 4 | 42 | 35 | 45 | 39 | 31 | 31 | 16 | 44 | 50 | 20 | 61 |
| | P23 | Si/Mg | 6 | 3 | 1 | 9 | 187 | 10 | 1 | 15 | 2 | 2 | 4 | 2 | 7 | 4 | 23 | 6 | 46 | 3 | 4 | 4 | 6 |
| | P25 | Si/W | 12 | 20 | 9 | 22 | 8 | 19 | 8 | 15 | 17 | 15 | 14 | 24 | 15 | 16 | 31 | 69 | 12 | 47 | 75 | 31 | 16 |
| Calcareous | P27 | Ca/Al | 44 | 151 | 85 | 55 | 61 | 135 | 36 | 47 | 46 | 189 | 76 | 171 | 67 | 82 | 100 | 41 | 38 | 53 | 77 | 65 | 53 |
| | P28 | Ca/Bi | 19 | 29 | 20 | 7 | 11 | 30 | 10 | 7 | 10 | 51 | 22 | 35 | 10 | 12 | 8 | 7 | 6 | 7 | 19 | 11 | 7 |
| | P29 | Ca/Br | 19 | 36 | 21 | 10 | 9 | 22 | 4 | 6 | 11 | 72 | 33 | 31 | 17 | 19 | 8 | 9 | 12 | 10 | 22 | 16 | 7 |
| | P31* | Ca/Mg | 69 | 101 | 252 | 80 | 61 | 112 | 69 | 152 | 122 | 127 | 35 | 78 | 38 | 101 | 69 | 32 | 159 | 32 | 89 | 71 | 40 |
| | P33 | Ca/Mg/Al | 31 | 32 | 87 | 22 | 11 | 24 | 29 | 37 | 35 | 10 | 11 | 11 | 26 | 23 | 15 | 5 | 22 | 16 | 29 | 15 | 13 |
| | P34 | Ca/Mg/Na | 0 | 2 | 13 | 0 | 0 | 0 | 1 | 51 | 11 | 20 | 0 | 2 | 3 | 8 | 0 | 3 | 2 | 4 | 9 | 4 | 1 |
| | P35* | Ca/Na | 12 | 110 | 195 | 30 | 32 | 81 | 26 | 120 | 76 | 588 | 56 | 106 | 62 | 70 | 62 | 40 | 52 | 60 | 81 | 77 | 47 |
| | P38 | Na/Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 34 | 8 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P39 | Ca/Na/Al Higher Ca | 6 | 22 | 49 | 4 | 3 | 4 | 10 | 24 | 12 | 101 | 5 | 9 | 17 | 4 | 8 | 5 | 4 | 26 | 10 | 11 | 15 |
| | P40 | Ca/Na/Al Lower Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 60 | 15 | 3 | 0 | 0 | 5 | 0 | 0 | 1 | 0 | 0 | 0 | 1 | 1 |
| | P42 | Ca/Na/Br | 0 | 2 | 14 | 0 | 1 | 0 | 4 | 3 | 5 | 49 | 1 | 7 | 3 | 4 | 0 | 1 | 0 | 6 | 4 | 4 | 5 |
| | P43 | Ca/Na/(Hg,W) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 16 | 2 | 6 | 1 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P44 | Na/Ca/Hg/Zr | 0 | 0 | 0 | 0 | 2 | 1 | 0 | 3 | 11 | 1 | 0 | 0 | 0 | 0 | 0 | 1 | 2 | 0 | 0 | 0 | 1 |
| | P45 | Ca/Na/W | 0 | 10 | 33 | 4 | 1 | 2 | 4 | 13 | 14 | 93 | 5 | 4 | 13 | 0 | 0 | 3 | 0 | 14 | 5 | 9 | 5 |
| | P46 | Ca/W | 6 | 78 | 47 | 24 | 31 | 67 | 22 | 20 | 36 | 137 | 41 | 87 | 32 | 47 | 85 | 18 | 26 | 18 | 45 | 33 | 23 |
| Sulfurous | P47 | Ba/S/Bi | 31 | 3 | 3 | 1 | 8 | 7 | 4 | 1 | 3 | 73 | 159 | 7 | 16 | 19 | 0 | 3 | 10 | 3 | 4 | 1 | 3 |
| | P48 | Ba/S/Na | 12 | 1 | 0 | 0 | 0 | 0 | 1 | 3 | 2 | 65 | 15 | 7 | 10 | 0 | 0 | 1 | 2 | 3 | 1 | 0 | 1 |
| | P49 | Ca/Ba/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 16 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | P50* | Ca/S | 361 | 49 | 104 | 417 | 785 | 335 | 53 | 205 | 87 | 16 | 132 | 100 | 8 | 482 | 193 | 31 | 24 | 46 | 21 | 1643 | 297 |
| | P57 | Na/Ca/S | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 51 | 0 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 |
| | P58 | Na/S/Ca/Bi | 517 | 5 | 855 | 19 | 96 | 88 | 345 | 246 | 70 | 23 | 89 | 4 | 6 | 4 | 8 | 1 | 6 | 2 | 6 | 20 | 7 |
| | P59 | Na/S/Hg | 50 | 0 | 19 | 0 | 2 | 1 | 4 | 74 | 14 | 14 | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4 | 1 | 2 |
| Ferrous | P60 | Fe | 12 | 5 | 6 | 10 | 11 | 27 | 5 | 6 | 86 | 0 | 14 | 7 | 11 | 19 | 15 | 21 | 38 | 6 | 18 | 10 | 5 |
| | P61 | Fe/Al | 31 | 5 | 10 | 17 | 16 | 22 | 9 | 4 | 14 | 1 | 15 | 22 | 17 | 23 | 8 | 18 | 28 | 12 | 9 | 7 | 9 |
| | P62 | Fe/Cr/Na | 0 | 2 | 0 | 5 | 0 | 1 | 0 | 5 | 18 | 2 | 1 | 13 | 6 | 12 | 8 | 8 | 10 | 31 | 2 | 6 | 0 |
| | P63 | Fe/Na | 44 | 7 | 13 | 7 | 16 | 30 | 6 | 23 | 169 | 6 | 21 | 18 | 33 | 16 | 8 | 36 | 52 | 16 | 41 | 17 | 7 |
| | P64 | Fe/Al/Si | 12 | 15 | 11 | 30 | 28 | 25 | 18 | 12 | 16 | 3 | 21 | 40 | 24 | 12 | 46 | 17 | 6 | 79 | 22 | 12 | 25 |
| Other | P65 | Cl/Na/Ca | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 16 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 1 |
| | P66 | Cu/Cl/Ni | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 15 | 0 | 0 | 0 | 0 | 0 | 278 | 0 | 0 | 0 | 0 |
| | P67 | Hg,Mg,Na,W,Zr/Ca/Pb | 0 | 1 | 2 | 2 | 49 | 28 | 140 | 6 | 381 | 3 | 4 | 13 | 9 | 4 | 0 | 116 | 12 | 1 | 2 | 2 | 3 |
| | P68 | Ti/(Al,La,Na) | 31 | 11 | 11 | 34 | 38 | 25 | 18 | 35 | 235 | 19 | 28 | 24 | 38 | 78 | 46 | 11 | 30 | 23 | 23 | 12 | 25 |

dark gray 0 to 5 | gray 6 to 10 | yellow 11 to 50 | gold 51 to 200 | red > 200

[28] Frequencies are given per 4000 particles, the maximum number analyzed as set by computer-assisted SEM/EDS parameters. Average values of 3 separate carpet areas are given for Carpets A, BH, BV, F, MT, PH, PL, PV and W. Types with an asterisk are consolidated categories, as detailed in Table 21. Specific occurrence data for all Carpet Areas and unconsolidated Target Particle Types are present in *Appendix K. Incidence of Target Particle Types.*

## IV. Conclusions

Our research has demonstrated that hundreds to thousands of very small particles (VSP) cling to the surfaces of individual carpet fibers. We have developed methods to remove these particles, analyze them, and use them to link the fibers to the carpet from which they came.

This project focused on one specific trace evidence application (carpet fibers) and one specific instrumental particle analysis method (computer-assisted SEM/EDS) to explore and test a fundamentally different approach to trace evidence analysis. In doing so, a set of reasonable assumptions and choices were made that can themselves be tested and refined for this specific application.

As proof of principle our findings are a highly significant breakthrough for the future of trace evidence analysis. They demonstrate a way to remove fundamental limitations of class associations and provide the impetus and direction for fundamental change in the way forensic trace evidence is conceptualized, analyzed and used in the criminal justice system. Further research is encouraged to allow the independent, quantitative testing of common origin using populations of VSP.

### A. Discussion of Findings

All of the findings in this project are based on the analysis of carpets covering a limited range of characteristics (primarily trilobal, nylon fibers) and present in a limited set of environments. Accordingly, the conclusions made from these findings would apply only to these types of carpets. They serve as a model and as an initial study. As with any research, the initial efforts define a starting point. Further work on other carpet types can then be considered in reference to these data and the findings can then be either generalized or more definitively qualified.

Nine specific findings are discussed in this section:

1. Very small particles (VSP) present on the surfaces of individual carpet fibers can be recovered nearly completely and prepared for computer-assisted SEM/EDS analysis by extraction with reagent ethanol and filtration onto polycarbonate filters.
2. Hundreds to thousands of VSP routinely occur on the surface of individual carpet fibers.
3. VSP on individual carpet fibers, when classified using criteria developed for environmental applications, cannot be considered as an unbiased statistical sampling of a VSP population on the carpet itself.
4. Environmental particle groupings have significant weaknesses for investigation of VSP variation and were found to be unsuitable.
5. Clearly defined, mutually exclusive Target Particle Types (TPTs), based on the commonly occurring elemental profile groupings within samples, were found to be useful for study of the within- and between-carpet variations in VSP occurrence.
6. Among different areas of the same carpet, most TPTs showed comparable occurrence, or comparable absence. Some TPTs were localized.
7. Different carpets vary widely in the TPTs and quantities of VSP adhering to their fiber surfaces.

8. When sufficient particles are recovered, individual fibers show highly characteristic patterns of TPTs that closely correspond to those from their originating carpet area.
9. Using a set of TPTs, VSP adhering to the surface of individual carpet fibers can be recovered, analyzed by computer-assisted SEM/EDS and used to associate these fibers with the carpet and carpet area from which they came.

1. Very small particles (VSP) present on the surfaces of individual carpet fibers can be recovered almost completely and prepared for computer-assisted SEM/EDS analysis by extraction with reagent ethanol and filtration onto polycarbonate filters.

Based on examination of fibers treated using a variety of solvents and agitation methods, the combination found to be most suitable for quantitative removal of very small particles (VSP) from commonly occurring carpet fibers was a ten-minute sonication in pure ethanol. Among those methods tested, this one resulted in the cleanest observed fibers without leaving a residue. The choice of ethanol as a solvent may not be optimum for recovery of VSP from all fiber types and it is not optimum for the recovery of all particle types. Fibers, both naturally occurring and man-made, have a range of chemical compositions, textures and surface treatments that will necessarily create a range of surface conditions with which any chosen solvent will interact. Any chosen solvent will also dissolve some portion of the particles present on the fiber's surface. Ethanol will dissolve many organic substances, and VSP made up of such organic material will be irretrievably lost when ethanol is used. Some particles can also be significantly altered or lost due to agitation using sonication methods. Examples are particles that are fragile, friable, or loosely aggregated. One specific type of alteration is the disruption of a larger particle into many smaller ones. This could change both quantitative and qualitative results for a method based on the numbers of VSP with a specific chemical element profile. For this reason sonication was limited to a duration achieving effective particle removal.

A successful method was developed for recovery of the VSP removed from individual carpet fibers and preparation of the VSP for computer-assisted SEM/EDS analysis. This method uses filtration through a small (5 mm x 5 mm) area of a polycarbonate membrane filter with 0.4 μm pore size, followed by mounting on carbon tape and then carbon-coating. The use of a filter with pore size 0.4 μm defines the lower size limit for particle recovery by this method, since all particles passing through the filter are not recovered. Although additional, still finer VSP could be recovered by using a smaller pore size, the computer-assisted analysis is more efficient for the larger particles, as is the filtration process. Furthermore, the larger particles also include the size ranges of interest for evaluation of the impact of airborne pollutants on the environment and on public health. By focusing on this size range, forensic applications can benefit from heavily funded research and existing environmental monitoring programs that are serving these fields of study. None-the-less, alteration of the procedures to include finer particles would increase the numbers of VSP recovered, and could result in the detection of additional particle varieties or particles with special transport characteristics that occur only in this smaller size range.

The purpose of developing the method was to remove, as quantitatively as possible, VSP adhering to the surface of commonly occurring carpet fibers, so that the types and numbers of these particles could be characterized by computer-assisted SEM/EDS. For this purpose the

method was well suited. SEM/EDS, as routinely applied in forensic science laboratories, is most applicable to particles in the size range that is recovered, and to inorganic particles, few of which will dissolve in ethanol. The method is effective, employs non-toxic materials, and is easy to apply. Practitioner review of the method established that it could be easily incorporated as a preliminary washing step prior to polarized light microscopy. With the development of this method, the first of the project objectives was achieved: to develop methods, compatible with existing fiber analysis protocols, and using currently available crime laboratory resources, to quantitatively remove and analyze the VSP adhering to carpet fibers.

2. Hundreds to thousands of VSP routinely occur on the surface of individual carpet fibers.

VSP isolated from individual fibers and analyzed by computer-assisted SEM/EDS varied in number from less than a hundred to greater than 4000 (the maximum number examined). After allowing for particles occurring in process blanks, there is an average of over 500 ethanol-insoluble VSP on the surface of a single carpet fiber. The confirmed presence of VSP in this quantity on carpet fibers enables research on the best means to analyze and interpret them, unlocking their extraordinary potential to enhance probative value and independently test hypotheses of common origin, as outlined in the guiding principles for this research effort.[29]

Almost certainly, the numbers of VSP present and recoverable from fiber surfaces will be related to the total amount of surface area on the fiber, as determined by its length, its width, and its cross-section. We did not test this relationship, but it is an important aspect that would affect the generalization of the findings. The fibers in this study were from 10 to 15 mm long, on the order of 70 micrometers in diameter, and almost all of trilobal cross-section. If shorter fibers were to be encountered in casework, for example, the available surface area would be less, and accordingly the numbers of particles present would be expected to be less. Our suspicion is that case-specific factors (e.g. how soiled the carpet is) would be the dominant consideration, irrespective of the fiber length.

The level of particle recovery in this study was more than sufficient for testing the specific program hypothesis and to achieve the related objectives to characterize within- and between-carpet variations in VSP profiles.

3. VSP on individual carpet fibers, when classified using criteria developed for environmental applications, cannot be considered as an unbiased statistical sampling of a VSP population on the carpet itself.

The hypothesis that the measured Environmental Particle Profiles on individual fibers represent an unbiased statistical sampling of the Environmental Particle Profiles on sets of ten fibers was strongly rejected. The specific program goal, to exploit existing computer assisted SEM/EDS methods to test whether the resulting fine particle profiles are useful to quantitatively associate shed fibers with a source carpet, was met.

[29] See page 12.

4. Environmental Particle Groupings have significant weaknesses for investigation of VSP variation and were found to be unsuitable.

The principal weaknesses of the Environmental Particle Groupings are that many particle compositions have ambiguous group classifications and are based on only a small percentage of the particle's composition.[30] The groupings, based on percentages of the total x-ray counts for a suite of 28 elements, are typically based on percentages of 10 and 20%. This means that a particle having, say, 25% of four different elements, could easily fit criteria for more than one class. As an extreme example, a particle with the following percentages

| Si | Al | Ca | Fe | S |
|---|---|---|---|---|
| 0.45 | 0.22 | 0.12 | 0.12 | 0.12 |

would fit the criteria for 5 out of the ten particle classes. Another weakness is that for most of the classes, the majority of the elemental composition is ignored. Only one particle class (Ca-rich, defined as Ca>=60) is based on more than 50% of the x-ray counts. The remaining classes are based on from 10 to 40% of the counts.

The classification ambiguities in the environmental classification scheme are resolved by sequentially applying the classification criteria. Once a particle fits a class it is removed from consideration in the remaining classes. This approach is a reasonable one when targeting specific types of particles, with a defined priority, in a given environmental problem. There is a specifically designed flexibility in the order of application of the criteria, and in the definition of the criteria themselves. In practice, an initial classification scheme is used essentially as a presumptive or screening test, to identify candidate particles that might meet a targeted particle type of environmental concern. Images and x-ray spectra of these individual particles, or a sampling of them, can then be examined as "confirmatory testing." Ignoring the majority of some particles' composition is also reasonable for the environmental task; if particles don't meet the presumptive screening criteria, they are conclusively not of interest and are disregarded.

Ultimately, once specific VSP of interest and significance are defined, a sequential analytical approach, such as described above may be extremely useful. Indeed, this is the approach taken for the analysis of gunshot residue (GSR) samples. The purpose of the current project, however, is the systematic investigation of VSP profiles on trace evidence and evaluation of their practical utility. The ambiguously defined, overlapping classes that are based on less than half of the elemental composition were found to be a poor means to investigate this variation.

5. Clearly defined, mutually exclusive Target Particle Types (TPTs), based on the commonly occurring elemental profile groupings within samples, were found to be useful for study of the within- and between-carpet variations in VSP occurrence.

[30] See Table 7 on page 22 for the specific environmental particle classification criteria.

The deficiencies of the Environmental Particle Groupings were overcome by defining Elemental Profile Groupings based on the four highest x-ray counts of the 28 elements detected by the computer-assisted SEM/EDS procedure. Groupings occurring at the highest levels in 27 carpet area samples were used to define 68 mutually exclusive TPTs which were then used to study within- and between-carpet variation.

Of the 68 TPTs, eight were found to have high occurrences in the process blanks, leading to high Limits of Detection (LODs) relative to the levels of occurrence found in the samples. These levels of occurrence of particles in the process blanks were not anticipated. Possible sources for the particles are: reagents (ethanol), glass/plasticware, settling airborne dusts, and the polycarbonate filters. A number of preventative measures were taken to minimize contamination. The ethanol was pre-filtered. Glassware and plasticware were washed with the pre-filtered ethanol and the sample preparations were conducted on a clean bench. The filters themselves were used directly and not pre-washed, but they are marketed to be particle-free. In this study no direct attempts were made to determine the source of the particles in the process blanks. Overall, there was a limited range of TPTs occurring in the them, and after estimating LODs, a sufficient number of TPTs remained to meet program goals.

The selection of TPTs was based on their high levels of occurrence among a subset of the program samples. This was a reasonable choice as a starting point for investigation, but this choice is likely to be superseded after further work by a selection based on forensic performance characteristics, once the variability and sources of VSP become better understood. For example, VSP that are the most discriminating particle types, or those with the most consistent levels of occurrence, or those with the lowest LODs, may not be those that occur at the highest levels. A likely ultimate analytical strategy will be to use TPTs whose origin is known and whose distribution in the population is well understood, thereby providing a sound foundation for estimation of their variability and the determination of the significance of correspondence.

A TPT as used in this project is a functional grouping, made for its utility in this initial study of VSP variation. Some of the TPTs were closely related to one another chemically and occurred in similar relative proportions in the samples. This led to the consolidation of a number of TPTs. Even so, the extent of correlation among the TPTs has not been determined, and this is a clear, necessary step toward the understanding of their forensic performance characteristics and their adoption in a formal methodology.

The origin of the TPTs as defined in this project is uncertain. Many different sources could, and likely do, contribute to particles meeting many of the broadly defined TPT categories. Nor need the TPTs be of a single type. A commonly occurring combination of particles, as an aggregate, or as a smaller particle adhering to the surface of another, could create a separate TPT as defined in this study.

Possible sources for the particle types occurring on carpets include both indoor and outdoor dusts, originating from the building itself, from activities occurring within it, from materials that were intentionally or unintentionally brought in, or from those transported by air from the surrounding environment. Indeed, the carpet composition itself may be the source of some of the TPTs, through the wear of its backing materials, for example. This variety in the sources of VSP

was anticipated and is part of the solution, rather than part of the problem. A central motivation for this project was to take advantage of these complex origins of VSP and to use the co-occurrence of many particle types to exploit those of modest frequency, leading to a highly probative test of common origin. However, an important part of the solution is also to a better understanding of the sources for the specific TPTs that are used for VSP characterization. At this stage of development, forensic performance characteristics of specific TPTs are poorly understood, and the initial criterion for selection, leading to their testing and evaluation in this project, was based on their levels of occurrence.

6. Among different areas of the same carpet, most TPTs showed comparable occurrence, or comparable absence. Some TPTs were localized.

Qualitatively, within-carpet variation for the TPTs was usually very low (~ 75% of the time), but occasionally ranged to very high for some TPTs and some areas (~ 13% of the time). This indicates an underlying, roughly even distribution of most particle types, together with occasional localized particle types. The findings do not support a single homogenized VSP distribution on fibers from a carpet, but they conclusively demonstrate that there are many particle types that occur with a sufficiently even distribution to be a useful for characterization.

The finding of some highly localized variability is not an unreasonable or unexpected result, given the likelihood of local carpet exposures to soiling or staining. Individual fibers can be exposed to a liquid, for example, and wicking along the fiber could well result in high levels of occurrence for those particles initially entrained in the liquid, but left behind once the liquid dries. Nearby carpet areas, with fibers that were not involved in the wicking process would lack these particles.

The effect of localized variation in carpets is both advantageous and challenging. Localized variation can provide additional spatial specificity to associations, but it also requires more comprehensive sampling. The sampling requirements would be analogous to soil cases, where localized variations occur along with more broadly occurring characteristics. At present, it is unknown how much sampling will be sufficient to effectively document a carpet's within-item variation. One reasonable initial approach is to focus only on that (majority) of particles that are shared widely in a sampling of several areas of the reference carpet, or those that represent a specific stained area.

The need to take multiple reference carpet samples, and to perform multiple analyses to establish within-carpet variation, raises questions of practicality. What is the balance between the level of effort required and the possibility of added probative value? Clearly more must be known regarding both aspects before this can be evaluated, but two things can be anticipated. Firstly, there will be increased probative value as research reveals the best means to systematically exploit the VSP profiles that are present, and secondly, there will be increased efficiency as semi-automated and computer assisted methodologies develop. Both of these will increase the practicality of the approach.

7. Different carpets vary widely in the TPTs and quantities of VSP adhering to their fiber surfaces.

The TPTs used to assess VSP variation among different carpets differed in their occurrence and discrimination potential across the set of 21 carpets studied in this project. Eight of the TPTs occurred at high levels in only one to a few of the 21 carpets studied, and another four TPTs occurred at moderate to high levels in several carpets, but at very low levels in most. Three other TPTs occurred over a wide range of levels across the full set of carpets. These 15 TPTs, in particular, are highly discriminating among the set of carpets.

Considering the profiles of VSP observed in each of the carpets, 12 of the 21 carpets are easily distinguished from one another based on qualitative differences and large differences in the frequencies of specific TPTs. The remaining eight carpets show a broad range of frequencies among five specific TPTs.

These findings provide a compelling demonstration of the potential of VSP to discriminate among areas from different carpets. The actual discrimination among carpets, as opposed to an area of a carpet, will be dependent on more complete studies of within-carpet variation and more carefully planned surveys of carpets. In this project our objective was to conduct a broader qualitative survey of carpet VSP profiles to explore between-item variation and to put the results of our investigations into a reasonable context. This objective has been met.

8. When sufficient particles are recovered, individual fibers show highly characteristic patterns of TPTs that closely correspond to those from their originating carpet area.

Very low particle totals were found on some individual fibers, but there was an average of over 700 per fiber. With few exceptions, when totals were over 1000, the TPT occurrences from individual fibers closely followed those from their originating area. The patterns were highly characteristic and showed both qualitative similarity, and similarities in rank and proportion. With lower totals (e.g. 300 to 800 particles), many fibers still showed reasonable similarities with their originating area.

TPTs allowed meaningful comparisons of the occurrences of VSP on individual carpet fibers. This directly supports a major project goal: to exploit existing computer assisted SEM/EDS methods to test whether VSP profiles are useful to quantitatively associate shed fibers with a source carpet. For the present methods, a strictly quantitative relationship among the full set of TPTs was not observed. However, the frequent occurrence of similarities in rank and proportionality, in separate experiments using multiple carpets and multiple areas within each carpet, establishes the proof of principle: VSP profiles on shed fibers can be measured, and they regularly correspond to reference samples taken from the area of the source carpet from which they came.

9. Using a set of TPTs, VSP adhering to the surface of individual carpet fibers can be recovered, analyzed by computer-assisted SEM/EDS and used to associate these fibers with the carpet and carpet area from which they came.

This finding is supported by the overall program results, including:

- Development and use of a practical method for the recovery of VSP from individual carpet fibers
- Demonstration of the regular occurrence of VSP on individual carpet fibers, in quantity and character sufficient to associate them with their carpet area of origin
- Development of a method suitable for the study of VSP variation, using a computer-assisted SEM/EDS analysis method based on TPTs
- Establishing that within-carpet variations show a roughly even distribution for most TPTs
- Establishing that between-carpet variations show a wide range in types and quantities of VSP, as demonstrated by TPT profiles

B. Discussion of the Underlying Technology and Scope

The exploitation of VSP to test the association of carpet fibers is a new approach, and one that lacks the comfort of actual particle identification and actual tracing of the source of the multitude individual particles. This differs from the exisiting paradigm where identification is a pre-condition to the use of particles, and where specificity in the measured characteristics provides the route to forensic utility. As discussed in the Introduction, the existing paradigm leads to fundamental limitations in our ability to interpret trace evidence. The impetus for the present research is to augment what is now being done, using VSP that we are currently ignoring, and using methods that will allow the combination of multiple characteristics that have measureable, testable frequencies of occurrence. These need to be valid characteristics, but they do not need to be particle identifications.

In particular, initial fundamental investigations such as this one must simplify the problem, in order to best learn what to study and improve. Within this context, moving forward requires trade-offs. Important aspects are the specificity of the particle characterizations, the reliability of their detection, the efficiency of the process, and the measureable, testable frequencies of occurrence. It is certain, for example, that better understanding of significance would follow from better identification of the VSPs, and that manual SEM/EDS particle imaging and EDS spectroscopy of individual particles would better and describe different particle types. Ultimately, if VSP are successfully exploited for associations, it is expected that other methods of VSP characterization, both more efficient and more specific will be developed.

At the present time, however, unambiguous identification of individual particles, attempting to determine their sources, and modeling evidential value based on characteristics of these sources is not the focus. TPTs are functional classifications consisting of groups VSP that share common characteristics as measured by a well-defined, practical methodology. SEM/EDS methodology is being used as a means to characterize thousands of particle X-ray count profiles and, by binning these profiles, to use populations of these particles in a useful way to test associations between carpet fibers and possible source carpets.

C. Implications for Policy and Practice

Five specific implications for policy and practice are discussed in this section:

1. The usefulness of VSP to remove fundamental limitations to the probative value of carpet fiber evidence has been demonstrated, providing the impetus and direction for fundamental change in the way that forensic trace evidence is conceptualized, analyzed and used in the criminal justice system.
2. The results of this research are likely extendable, with minor modifications, to other trace evidence types, and are expected to contribute significantly for those types of trace evidence that have long been considered of low evidential value.
3. An entirely new approach to trace evidence is enabled: comparing different types of trace evidence with one another by way of their adhering VSP.
4. An additional, high priority use for existing crime laboratory SEM/EDS analytical capabilities and related practitioner skills can now be anticipated, guiding the allocation of laboratory resources.
5. A need can be anticipated for policies and practices for evidence collection and processing of crime scenes that are sensitive to requirements for the preservation and analysis of VSP.

1. The usefulness of VSP to remove fundamental limitations to the probative value of carpet fiber evidence has been demonstrated, providing the impetus and direction for fundamental change in the way that forensic trace evidence is conceptualized, analyzed and used in the criminal justice system.

This project has increased our fundamental understanding of the next (finer) dimension of particles, and the potential use of these particles in one specific trace evidence application has been established. The implications for policy and practice are profound. The fundamental limitation to the probative value of trace evidence, noted in the NRC report,[5] stems from conventional trace evidence characteristics being determined by their manufacture. As mass-produced commodities, probative value is limited to class associations, with qualitative, expert assessments of evidential value. The analysis of VSP, riding "piggy back" on the surface of other trace evidence particles, can remove this fundamental limitation, by adding independent, quantitative, objectively measured fine particle profiles that have accumulated from the environment during use in a specific location. This will lead to much more sensitive testing of associations, and more certain and convincing associative evidence.

At the outset of this project, the existence of useful VSP profiles on the surface of carpet fibers had yet to be established. This has now been conclusively demonstrated. There is now a clearly achievable impact of these methods that will remove the fundamental limitations discussed above, increase the probative value of trace evidence and provide an independent quantitative means to test hypotheses of common origin. The occurrence, with modest frequency, of specific TPTs will ultimately be estimated, and with such modestly occurring events upper bounds on frequencies and correlations can be reliably estimated. It is the co-occurrence of multiple events

of modest frequency that is the foundation for all highly probative types of physical evidence, including DNA and fingerprint identifications.
To unlock this potential, further research will be required that (1) determines which TPTs have the best forensic performance characteristics, (2) rigorously documents within- and between-carpet variability for these TPTs, (3) optimizes the analytical protocol, (4) determines how susceptible VSP profiles are (post shedding) to contamination and loss, and (5) develops and validates quantitative methods for the testing of common origin using VSP.[31]

2. The results of this research are likely extendable, with minor modifications, to other trace evidence types, and are expected to contribute significantly for those types of trace evidence that have been considered of low evidential value.

The results of this research have implications for other types of trace evidence: for example, there are VSP adhering to other types of fibers, hair, glass, and paint. Some types of evidence, now of extremely low value for trace evidence associations (such as undyed cotton fibers) may become highly useful for association based on the VSP that adheres to them. The morphological examination of hair, for example, vulnerable to criticism based on subjectivities and variations in methodology, can be supplemented by comparisons of adhering VSP. In extension of the research to these areas, the methodologies and interpretations will be similar. That is, separate studies of fine particle populations for each type of trace evidence are unlikely to be necessary, since what is being compared are the particles from the environments. This is a noteworthy advantage over the use of new types of trace evidence that would each require their own separate study of population frequencies.

3. An entirely new approach to trace evidence is enabled: comparing different types of trace evidence with one another by way of their adhering VSP.

Any trace evidence type can act as the specific carrier of a more general “VSP signal.” Any fiber from a residence, for example, could carry VSP corresponding to any other fiber. They need not be fibers from the same carpet. Extending this somewhat further, VSP on a knife or tool left at a crime scene could, for example, be linked with VSP from a suspect’s residence, or VSP on the edges of tape on the inside of an IED could be linked with that found on the edges of an entirely different kind of tape in a suspect’s workshop.

4. An additional, high priority use for existing crime laboratory SEM/EDS analytical capabilities and related practitioner skills can now be anticipated, guiding the allocation of laboratory resources.

The computer-assisted SEM/EDS methods used for analysis of VSP on carpet fibers were specifically developed with a view toward utilization of existing crime laboratory equipment and related practitioner skills. Some laboratories utilize nearly identical methods in support of GSR analysis. Others use closely related equipment and skills as a part of materials analysis and

[31] The specific implications for this further research are discussed in the following section.

characterization. The anticipation of an additional high priority application will impact policy and practice as it will provide additional impetus and objectives for the retention and further development of capabilities in these areas. As an alternative, there are possibilities for commercial outsourcing of these types of analyses, which are already in place as applied to GSR and environmental dust analyses.

5. A need can be anticipated for policies and practices for evidence collection and processing of crime scenes that are sensitive to requirements for the preservation and analysis of VSP.

As methods for the exploitation of VSP come closer to implementation, a need for appropriate evidence collection and processing methods can be anticipated. These methods will include collection of appropriate control samples and information that would help to interpret the presence of VSP that may be present. The requirements for actual collection practices will only be clear after additional research, but at this point a need for such methods can be anticipated.

## D. Implications for Further Research

The establishment of useful VSP profiles on the surface of individual carpet fibers provides both the impetus and direction for follow-on research. This research can be broadly divided as (1) that focused on developing the specific application of computer-assisted SEM/EDS analysis of VSP on carpet fibers, and (2) that focused on expanding the VSP approach to other applications.

For the specific carpet fiber application, proof of principle has been established with the development of practical methodologies, establishment of workable within-item variability and the measurement of corresponding profiles on individual fibers. The focus for this application now shifts to direct follow-on steps, the most important of which are: (1) determining which TPTs have the best forensic performance characteristics for carpets, (2) rigorously measuring within and between variability for these TPTs, (3) optimizing the analytical protocol, (4) determining how susceptible shed fibers are to contamination and loss of VSP profiles, and (5) developing and validating quantitative methods for the testing the origin of carpet fibers using VSP.

For expansion of the VSP approach, additional alternative directions are (6) application to other trace evidence types, (7) application of other instrumental analysis methods, (8) the use of VSP to associate an environment with an object and (9) more general development and validation of quantitative methods for the use and interpretation of VSP.

### 1. Determining Which TPTs have the Best Forensic Performance Characteristics for Carpets

TPTs must be studied to determine which have the best forensic performance characteristics for the linking carpet fibers to carpets. Performance characteristics take into account the combination of occurrence within samples, correlations among TPTs, discrimination among samples, levels of detection, and reliabilities of detection.

Multivariate statistics are essential for investigation of correlations and discrimination. Principle components analysis, or one of the more modern approaches to discriminant analysis, needs to be employed.

Another very important aspect for effective assessment of forensic performance characteristics is having a good understanding of the actual identity and (generic) source of the particles. Some sources may be inherently highly correlated, either with one another, or with a particular brand or composition of carpet. The experience of the broader scientific community in the analysis of respirable particles of environmental, public health and occupational health significance is directly applicable. This experience needs to be incorporated, but built upon and augmented to include additional types of particles that occur routinely in human dwellings or vehicles and that are not of interest to these disciplines.

2. Rigorously Measuring Within and Between Variability for these TPTs

Variability, both within and between carpets, must be better understood. Investigation of within-carpet variability will require testing of carpet samples taken comprehensively from entire rooms, entire buildings and entire areas of vehicles. Shed fibers, as opposed to cut fibers from fabric, will need to be included in these tests. The length of such fibers is an important parameter to include among those studied.

Investigation of between-carpet variability will require testing of carpets from different geographical areas, and testing multiple locations in the same geographical area. As with research on performance characteristics, knowledge of the actual source of the particles, when determinable, will lead to greater understanding of the factors contributing to their variability.

3. Optimization of the Analytical Protocol

Parameters for the computer-assisted SEM/EDS method employed in this project were chosen as those found to be reasonable for characterization of environmentally significant particle types. These parameters need to be reconsidered and tested, now that the application itself is better defined. This research is linked with the determination of which TPTs have the best performance characteristics for carpets and the two research tasks must run in parallel.

For example, parameters for analysis in this project included particles in a broad size range (0.4 to 50 μm). A narrower size range may be better for VSP profile comparisons in carpets. Easily airborne, respirable particles, with a particle size up to about 10 μm, may well be more evenly distributed on carpets than larger particles. If so, omission of larger particles may eliminate some of the highly localized TPT occurrences that are seen. Other aspects of the method could have similar effects, such as changing the range of elements analyzed, the maximum number of particles analyzed, the detection thresholds, and the particular algorithms that are used as tests for specific TPTs.

Overall, SEM/EDS methods related improvements that should be considered as part of further research are:

- Inclusion of manual SEM/EDS particle imaging and viewing of SEM/EDS spectra for identifying and discussing the TPTs
- Inclusion of the analysis of known materials or known examples of TPTs a quality control procedure
- Conducting manual SEM analyses on samples and comparison with the computer-assisted data as a quality control procedure for possible peak misidentifications
- Extension of the particle size range (and thereby the particle numbers) by using a backing filter to collect smaller particles and employing a higher magnification in the SEM

4. Determining How Susceptible Shed Fibers are to Contamination and Loss of VSP Profiles

Forensic applications will be based on the transfer of carpet fibers from a source carpet to an object or person. During or post transfer, VSP profiles on carpet fibers are subject to contamination (acquisition of additional VSP) or losses (loss of some of the originally adhering VSP). These changes may occur very slowly, and at low levels, or may occur quickly. There may be exchange with those VSP on a receiving substrate. Losses, should they occur, may be proportional (all VSP affected evenly) or disproportional (some types of VSP markedly more subject to retention or loss). The levels of occurrence of any such effects are unknown. If they occur, they need not greatly affect the methodology in actual application, but they must be understood in any case. Additionally, the consequences of fiber recovery methods (such as taping and scraping) need to be evaluated.

5. Development and Validation of Quantitative Methods for the Testing the Origin of Carpet Fibers using VSP

A rigorous quantitative means for interpretation of VSP profiles will be the ultimate outcome of this research program, building on each of the four contributions mentioned above

- TPTs with high forensic performance parameters for carpets
- rigorously measured of within- and between-carpet VSP variability
- an optimized analytical protocol
- known limits on the effects of contamination and loss

With these inputs a well-defined quantitative interpretive model for carpet fiber evidence must be developed and validated using appropriate experimental trials and statistical tests.

6. Application to Additional Trace Evidence Types

As noted under Implications for Policy and Practice, the results of this research are likely extendable, with minor modifications, to other trace evidence types, and are expected to contribute significantly for those types of trace evidence that have been considered of low evidential value. The two types specifically mentioned were undyed cotton fibers, currently

considered of nearly no probative value for association, and hairs, currently vulnerable to criticism based on subjectivities and variations in methodology. The ability of VSP to test associations for these two evidence types would have a highly significant, immediate impact.

## 7. Application of Other Instrumental Analysis Methods

SEM/EDS is not the only candidate for the analysis of VSP. Particle profiles can be analyzed by a growing number of high-throughput methods based on light or electron microscopy, Raman microscopy, FTIR microspectroscopy, Raman microspectroscopy or even methods of molecular biology as applied to non-human DNA. Additional techniques will be forthcoming and each should be evaluated for a possible niche in the forensic analysis of VSP.

## 8. Use of VSP to Associate an Environment with an Object

Again, as noted under Implications for Policy and Practice, an entirely new approach to trace evidence is possible using VSP: comparing different types of trace evidence with one another. Any trace evidence type can act as the specific carrier of a more general "VSP signal." These methods are essentially comparing an environment (via its VSP profile) to items that could have originated from that environment. *Any* item or piece of trace evidence originating from a crime scene can potentially be linked to that crime scene using the crime scene's VSP profile.

## 9. General Development and Validation of Quantitative Methods for the Use and Interpretation of VSP

Parallel to the specific development and validation of quantitative methods for carpet fiber applications, research providing inputs to more general applications will include better understanding of forensic performance parameters of a broader set of TPTs, generalizations (as possible) of their within- and between-item variability, correlations among them, and the limits of contamination and loss during contact and transfer.

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## VI. Dissemination of Research Findings

1. Presentations Resulting from this Award (as of 1/30/12)

Bowen, A. “A Procedure for Recovering Fine Particles from Carpet Fibers,” Inter/Micro 2011, Chicago, IL, July 11, 2011.

Stoney, DA, Bowen, A and Stoney, PL. “Time to Rethink Dusts,” NIJ/FBI 2011Trace Evidence Symposium, Kansas City, MO, August 11, 2011.

Stoney, DA, Bowen, A and Stoney, PL. “Use of Computer Controlled Scanning Electron Microscopy (CCSEM) Methods for the Analysis of Small Particles Adhering to Carpet Fiber Surfaces,” San Diego Police Department Crime Laboratory, Trace Evidence Section, San Diego, CA, December 5, 2012.

Stoney, DA, Bowen, A and Stoney, PL. “Use of Computer Controlled Scanning Electron Microscopy (CCSEM) Methods for the Analysis of Small Particles Adhering to Carpet Fiber Surfaces,” American Academy of Forensic Sciences, 40th Annual Meeting, Atlanta, GA, February 25, 2012. (Upcoming)

2. Publications Resulting from this Award (as of 1/30/12)

A New Method for the Removal and Analysis of Small Particles Adhering to Carpet Fiber Surfaces, (Accepted with Minor Revisions, publication pending as a Technical Note).

3. Training Module

A training module was developed and has been delivered as an attachment to this report: *Appendix L. Training Module*.