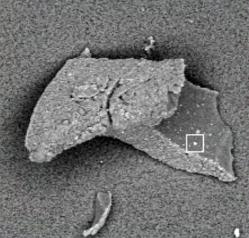
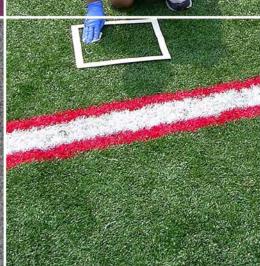


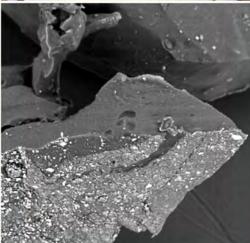
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A Scoping-Level Field Monitoring Study of Synthetic Turf Fields and Playgrounds



Office of Research and Development

National Exposure Research Laboratory

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Prepared by the National Exposure Research Laboratory Office of Research and Development U.S. Environmental Protection Agency

with contributions from the Agency's Tire Crumb Science Workgroup

Disclaimer

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Abstract

Recycled tire material, or "tire crumb," is used as a component in many recreational fields, including synthetic turf fields and playgrounds. The use of tire crumbs in these applications provides several benefits, including reduced sports injury. The public recently has raised concerns regarding potential human health and environmental risks associated with the presence of and potential exposures to tire crumb constituents in recreational fields, especially with regard to children's exposures.

In early 2008, U.S. Environment Protection Agency (EPA) Region 8 requested that the Agency consider this issue. A cross-EPA workgroup inventoried and considered the limited available scientific information: some laboratory studies of tire material content, off-gassing, and leaching characteristics and a few European studies describing the extent and availability of tire crumb constituents for potential human exposure. The workgroup recommended that research be conducted to generate additional field monitoring data for potential U.S. environmental conditions and potential exposures.

A limited-scale study was conducted during the 2008 summer and fall seasons to
(1) gain experience conducting multiroute field monitoring of recreational surfaces that contain tire crumb by evaluating readily available methods for measuring environmental concentrations of tire crumb constituents; and

(2) generate limited field monitoring data that will be used by EPA to help the Agency determine possible next steps to address questions from the public regarding the safety of tire crumb infill in recreational fields.

The field sites were selected based on availability and proximity to facilities of EPA's National Exposure Research Laboratory; thus, the results reported here may not be representative of environmental concentrations found at other sites. Because validated methods for sampling synthetic turf fields or playgrounds did not exist, methods used for other microenvironmental sampling were used. The full study protocol was implemented at two synthetic turf fields and one playground. At each field and the playground, air sampling was conducted to collect integrated particulate matter (PM₁₀) and grab volatile organic chemical (VOC) samples at two to three locations on each turf field and playground and also at an upwind background location. The air samples were collected at a height of 1 m in close proximity to, but without interfering with, planned recreational activities. The VOC samples were collected around 2:00 p.m. Wipe samples were collected at the three turf field sampling locations, along with readily available tire crumb infill and turf blade samples. Tire crumb material was collected from the playground. The full protocol was implemented at one of the synthetic turf fields on a second consecutive day providing repeat sampling data. Selected samples were collected at a few additional synthetic turf fields and one playground.

Standard laboratory analysis methods were employed to analyze the environmental samples for the targeted analytes. The PM_{10} samples were analyzed for PM mass, metals, and particle morphology. The VOC samples were analyzed for 56 volatile organic analytes. The wipe and material samples were analyzed for total extractable concentrations of several metals and bioaccessible lead.

Key findings are summarized below.

- (1) The study protocol and many of the methods were found to be reliable and could be implemented in the field. Several limitations are noted below.
 - a. Collecting integrated air samples provided a high burden in terms of time and equipment.
 - b. Semivolatile organic compounds were not measured.
 - c. At any single site, there can be substantial variability in the materials used and the concentrations of contaminants measured. More work is needed to determine where to collect samples and how many samples to collect to fully characterize a given site.

- d. It was difficult to obtain access and permission to sample at playgrounds and synthetic turf fields. More work is needed to increase public and private owner participation if additional monitoring studies are conducted.
- (2) Methods used to measure air concentrations of PM₁₀ and metals were found to be reliable.
 - a. Concentrations of PM₁₀ and metals (including lead) measured in air above the turf fields were similar to background concentrations.
 - b. Concentrations of PM₁₀ and metals at the playground site with high play activity were higher than background levels.
 - c. All PM₁₀ air concentrations were well below the National Ambient Air Quality Standards (NAAQS) for PM₁₀ (150 μg/m³). All air concentrations for lead were well below the NAAQS for lead (150 ng/m³).
- (3) Methods used to measure VOCs in air were found to be reliable.
 - a. All VOCs were measured at extremely low concentrations that are typical of ambient air concentrations.
 - b. One VOC associated with tire crumb materials (methyl isobutyl ketone) was detected in the samples collected on one synthetic turf field but was not detected in the corresponding background sample.
- (4) Methods used to measure extractable metals from turf field blades, tire crumb materials, and turf field wipe samples were found to be reliable. However, the aggressive acid extraction procedure likely will overestimate the concentration of metals that are readily available for human uptake. Since understanding uptake is a key component in understanding risk, methods to determine bioavailable metal concentrations still are needed.
 - a. Total extractable metal concentrations from the infill, turf blade samples and tire crumb material were variable in the samples collected at a given site and between sites.
 - b. The average extractable lead concentrations for turf blade, tire crumb infill, and tire crumb rubber were low. Although there are no standards for lead in recycled tire material or synthetic turf, average concentrations were well below the EPA standard for lead in soil (400 ppm).
 - c. Likewise the average extractable lead concentrations for turf field wipe samples were low. Although there are no directly comparable standards, average concentrations were well below the EPA standard for lead in residential floor dust (40 µg/ft²).
- (5) On average, concentrations of components monitored in this study were below levels of concern; however, given the very limited nature of this study (i.e., limited number of components monitored, samples sites, and samples taken at each site) and the wide diversity of tire crumb material, it is not possible to reach any more comprehensive conclusions without the consideration of additional data.

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Executive Summary

Background

Recycled tire material, or "tire crumb", is used in many applications, including as a component in synthetic turf fields and playground installations. The use of tire crumbs in these applications provides several benefits, including but not limited to reduced impact injuries; reduced or eliminated use of water, fertilizer, and pesticides needed to maintain grass fields; reduced need for disposal of used tires in landfills; and increased availability of fields for recreation. The public recently has raised concerns regarding potential human health and environmental risks associated with the presence of and potential exposures to tire crumb constituents in recreational fields, especially with regard to children's exposures.

In early 2008, U.S. Environmental Protection Agency (EPA) Region 8 requested that the Agency consider this issue, and a cross-EPA workgroup was formed. The workgroup inventoried and considered the limited available scientific information: laboratory studies of tire material content, off-gassing, and leaching characteristics. Also, a few European studies reported data describing the extent and availability of tire crumb constituents for potential human exposure through various routes and pathways (inhalation, ingestion, and dermal contact).

In the late spring of 2008, a smaller EPA Tire Crumb Science Workgroup (science workgroup) subsequently was formed and charged to consider the quality of the current science and make recommendations regarding the need for future research. Because minimal environmental or exposure data for U.S. populations were available, a limited scoping study was proposed and designed to evaluate a protocol and methods for generating consistently collected U.S. environmental data for select tire crumb constituents.

This report provides the EPA scoping study results. The EPA scoping study results, along with results from other studies conducted by Federal, State, and local organizations, such as the Consumer Product Safety Commission (CPSC); the Agency for Toxic Substances and Disease Registry; States including New Jersey, Connecticut, California, and New York; and New York City, will be considered by EPA to identify possible next steps to address questions from the public regarding the safety of tire crumb infill in ball fields and playgrounds.

Scoping Study Objectives

The EPA science workgroup proposed a limited scoping-level study during 2008 that included the following elements.

- Evaluate, through real-world measurements, the application of readily available sampling and analysis methods for characterizing environmental concentrations of selected tire crumb contaminants in and around playgrounds and synthetic turf fields.
- Evaluate the overall study protocol (monitoring, analytical, and quality control [QC] procedures) for generating the quantity and quality of environmental measurement data needed to characterize the contribution of the tire crumb constituents to environmental concentrations.
- Collect a limited environmental dataset to help understand and assess methods for characterizing potential route- and pathway-specific exposures (inhalation, ingestion, and dermal) based on selected sentinel species.
- Generate a limited set of consistently collected field measurement data from a very few
 playgrounds and synthetic turf fields that, along with other study data, may be used to
 develop insights regarding the importance of the various exposure routes and pathways and
 to inform decisions regarding possible next steps to address questions from the public
 regarding the safety of tire crumb infill in ball fields and playgrounds.

Study Approach

The proposed final study design included the collection and analysis of selected air, wipe, and material samples at one playground and one synthetic turf field site in the EPA regions where the four National Exposure Research Laboratory (NERL) facilities are located. This design (a total of eight sites) was based on the availability of NERL technical support. During a single daytime period at each site, air samples were to be collected at up to three "on field" or "on playground" sampling locations within the site boundaries in areas as close to anticipated human activity as possible without interfering with routine activities. Air samples also were to be collected at site background upwind sampling locations to characterize local ambient background levels. A comparison of "on playground" or "on field" data with the background data would be used to characterize the environmental availability of tire crumb constituents. Surface wipe samples were to be collected at the "on field" air sampling locations, but not at the background sampling location. Tire crumb and synthetic turf blade samples were to be collected at multiple sampling locations, but these were not always the same locations as the air sampling locations. The following samples were planned for collection and analysis.

- Grab air samples during the hottest daytime period (~2:00 p.m.) to assess organic vapor concentrations (56 volatile organic compounds [VOCs])
- Integrated air particulate matter (PM₁₀) samples to assess particle mass concentrations and concentrations of selected metals (including lead [Pb], chromium [Cr], zinc [Zn], and others)
- Integrated air PM₁₀ samples to characterize ambient particles based on morphology (sizes and structure) using scanning electron microscopy (SEM) and, if possible, to estimate the relative contribution of tire crumb particles to the overall particle mass
- Wet surface wipe samples to assess environmental concentrations of metals (e.g., Pb, Cr, Zn, and others) associated with turf field materials (tire crumb rubber and turf blades)
- Turf field tire crumb infill granules, turf blades, and playground tire crumb material to assess concentrations of metals (e.g., Pb, Cr, Zn, and others) associated with these materials
- Field and laboratory QC samples to document the quality of the study data. Duplicate samples for each measure described above were collected where appropriate. Routine field and laboratory QC samples (e.g., blanks, spikes) also were analyzed.

Study Limitations

This limited scoping-level study was designed to evaluate the methods for generating quality environmental data for selected tire crumb constituents and for understanding potential exposure routes and pathways. The study was planned based on readily available resources (personnel, equipment, media, etc.) and in consideration of the workgroup's desired study time period (the summer and early fall of 2008). This time period was recommended, as the projected high ambient temperatures should result in conditions promoting the greatest potential for the environmental release of tire-related constituents.

This study and the resulting data have many limitations. The study was not designed to provide representative U.S. environmental measurement data for all tire crumb constituents or applications. Nor was the study designed to inform conclusions regarding differences in U.S. environmental concentrations or potential exposures to turf field and playground tire crumb constituents based on geographical location, type, manufacturing materials, age, use, or conditions. The study also was not designed to compare potential exposures to turf field and playground tire crumb constituents with those at natural turf fields or playgrounds constructed with other types of surfaces. The study collected limited environmental data to help understand and assess methods for characterizing potential route- and pathway-specific exposures (inhalation, ingestion, and dermal) based on selected sentinel species. No personal exposure data or related information were collected. Validated sampling and analysis methods for characterizing recreational fields were not available, so existing methods used in similar studies

were applied. The study did not evaluate methods for all the reported tire crumb constituents. Semivolatile organic compounds (SVOCs [e.g., benzothiazole, aniline, polycyclic aromatic hydrocarbons (PAHs)]) reported in some studies were not sampled or analyzed because of resource limitations.

Sample Collection Results Sampling Sites

The full study protocol was implemented at only two synthetic turf field sites (F1 and F4) and one playground site (P1), fewer than the planned four turf field and four playground sites. Difficulties in identifying and arranging site access, logistical limitations, and personnel requirements to operate the extensive array of equipment and sites were the key factors impacting the number of sites monitored.

Unplanned sampling also occurred and is reported herein. The full protocol was conducted at F1 on a second consecutive day providing repeat measures. A reduced set of samples (without integrated air particle monitoring) was completed at a third synthetic turf field site consisting of two collocated fields (F2 and F3). Some samples were collected for two additional turf fields (F5 and F6) collocated with F4. Two F4 "on field" sampling locations were very near a busy commuter road and parking deck.

When a site consisted of multiple fields, one field was designated as the primary location for implementing the protocol. In total, samples were collected for six different synthetic turf fields.

Gaining access to playgrounds was very difficult and became even more difficult with increased media attention. The full sampling protocol was completed at only one playground (P1) and at only two "on playground" sampling locations because of space limitations. Tire crumb material molded to mimic wood bark was obtained from a second playground site (P2).

Sample Collection and Analysis Methods

Air VOCs. Grab air VOC samples (6-L Summa-polished stainless steel canisters) were collected at each sampling location at a 1-m inlet height during the hottest time of day (~2:00 p.m.). The standard EPA Method TO-15 gas chromatography/mass spectrometry (GC/MS) analytical method provided ambient-level concentration measurements for 56 VOC analytes.

Air Particulate Matter. Two integrated air PM_{10} samples (one for particle mass and metals analysis and another for scanning electron microscopy [SEM] analysis) were collected at each sampling location at a 1-m inlet height over collection periods ranging from 5.8 to 7.8 h. This resulted in individual sample air volumes ranging from approximately 7.0 to 9.2 m³ (3.5 to 5.0 m³ for SEM samples). PM_{10} mass was determined gravimetrically; metal concentrations by X-ray fluorescence; and assessment of particle size and morphology and attempts to identify the tire crumb component contribution by SEM.

Synthetic Turf Field Surface Wipes. No known validated methods exist for characterizing environmental concentrations of metals on synthetic turf surfaces comprised of both turf blades and tire crumb rubber. A standard wet-wipe method (American Society for Testing and Materials [ASTM] E1728-03) used routinely to measure residential surface dust Pb levels was used for this study. Advantages of this method were the availability of standard wipe material and the existing, well-characterized, sampling and analytical methodologies. Samples were collected at each "on field" turf field sampling location. Wipe samples were not collected at the "on playground" or background sampling locations. Each surface wipe, tire crumb, and turf blade sample (described below) was extracted first using the EPA In Vitro Relative Bioaccessibility Assessment Method 9200.1-86. (Note: In vitro methods measure the bioaccessibility [e.g., solubility] of metals during a simulated gastric extraction process to assess the percentage of a metal in a material that may become available for absorption in the gastro-

intestinal [GI] tract.) The same material from each sample then was extracted using EPA Method 3050B. A total extractable concentration of Pb, Cr, Zn, arsenic (As), aluminum (Al), barium (Ba), cadmium (Cd), copper (Cu), iron (Fe), manganese (Mn), and nickel (Ni) was determined by an analysis of the combined bioaccessibility and Method 3050B extracts. Extracts were analyzed by inductively coupled plasma (ICP)/MS using EPA Method 6020A. The percent bioaccessible Pb was calculated from the relative amount in the bioaccessible extract as compared with the total extractable amount.

Synthetic Turf Field Tire Crumb Infill. Tire rubber infill was collected randomly at the synthetic turf fields. In this study, it was decided to collect infill material that was readily available at the surface rather than dislodging material trapped deep within the turf blades. This decision was based partly on avoiding potential damage to field components but primarily because the material on the surface was more available for potential human contact. Infill material was not available uniformly across the field surface.

Synthetic Turf Field Blades. Blades were randomly collected at the synthetic turf fields. Collecting blades of each color present at the field was attempted. Turf blade collection relied on the availability of loose blades found on the field surface in lieu of a destructive (i.e., cutting) method. Collection and analysis decisions were complicated by the limited availability of loose blades and a later determination that a minimum of 0.7 g of material was required for analysis.

Playground Tire Crumb Rubber. Tire crumb samples were obtained from two playground sites. It was not clear how many pieces needed to be collected nor at what depth (surface/subsurface) for site characterization, as the crumb shifts with mechanical action. A further challenge is that relatively small amounts (1 g or less) are required for analysis; large amounts may overwhelm the digestion and analytical systems. Intact tire crumb rubber pieces were larger than 1 g. A decision was made not to cut samples, as this would expose unweathered surfaces and possibly impact the bioaccessible Pb estimate.

Conclusions

The key study findings are summarized below. The narrative and appendixes that follow this Executive Summary provide additional details regarding the study, along with all of the measurement and laboratory data. This descriptive report focuses on the study design and methodologies; assessing the methodology for characterizing environmental concentrations of tire crumb constituents in future studies; describing the quality of the scoping study data; and providing recommendations for consideration in the design of any future research, if needed.

In general, the study protocol is expected to reliably yield data for assessing environmental concentrations of selected tire crumb constituents and understanding potential exposure routes and pathways. However, when considering future study designs and implementation, the research needs to carefully consider issues associated with identifying and gaining site access, the cost benefit of obtaining the data versus the resource burden, and the implementation of other methods for generating data to address specific research hypotheses. Future studies will need a carefully developed and implemented communications plan to promote the value of the research and gain access to the required facilities.

- (1) The study protocol and many of the methods were found to be reliable and could be implemented in the field. Several limitations are noted as follows.
 - Collecting integrated air samples provided a high burden in terms of time and equipment.
 - SVOCs were not measured.
 - At any single site, there can be substantial variability in the materials used and the
 concentrations of contaminants measured. More work is needed to determine where to
 collect samples and how many samples to collect to fully characterize a given site.

- It was difficult to obtain access and permission to sample at playgrounds and recreational fields. More work is needed to increase public and private owner participation if these studies are to be implemented.
- (2) Methods used to measure air concentrations of PM₁₀ and metals were found to be reliable.
 - Concentrations of PM₁₀ and metals (including Pb) measured in air above the turf fields were similar to background concentrations.
 - Concentrations of PM₁₀ and metals at the playground site with high play activity were higher than background levels.
 - All PM₁₀ air concentrations were well below the National Ambient Air Quality Standards (NAAQS) for PM₁₀ (150 μg/m³). All air concentrations for Pb were well below the NAAQS for Pb (150 ng/m³).
- (3) Methods used to measure VOCs in air were found to be reliable.
 - All VOCs were measured at extremely low concentrations that are typical of ambient air concentrations.
 - One VOC associated with tire crumb materials (methyl isobutyl ketone) was detected in the samples collected on one synthetic turf field but was not detected in the corresponding background sample.
- (4) Methods used to measure extractable metals from turf field blades, tire crumb materials, and turf field wipe samples were found to be reliable. However, the aggressive acid extraction procedure likely will overestimate the concentration of metals that are readily available for human uptake. Because understanding uptake is a key component in understanding risk, methods to determine bioavailable metal concentrations are still needed.
 - Total extractable metal concentrations from the infill, turf blade samples, and tire crumb material were variable both between sites and at the same sites.
 - The average extractable lead concentrations for turf blade, tire crumb infill, and tire crumb rubber were low. Although there are no standards for Pb in recycled tire material or synthetic turf, average concentrations were well below the EPA standard for lead in soil (400 ppm).
 - Likewise the average extractable Pb concentrations for turf field wipe samples were low. Although there are no directly comparable standards, average concentrations were well below the EPA standard for lead in residential floor dust (40 µg/ft²).
- (5) On average, concentrations of components monitored in this study were below levels of concern; however, given the very limited nature of this study (i.e., limited number of components monitored, samples sites, and samples taken at each site) and the wide diversity of tire crumb material, it is not possible to reach any more comprehensive conclusions without the consideration of additional data.

1. Introduction

1.1 Background

Tire crumb or crumb rubber is produced from scrap tires or from the tire retreading process. During the recycling process, steel and usually fiber are removed, and the remaining rubber material is processed either by mechanical means or through freeze cracking into "chips" or into various sizes of rubber mesh with a granular consistency. Tire crumb is used in several commercial applications, including road construction, sidewalks, automobile parts, and in a number of athletic and recreational applications. Recreational uses include ground cover (chips) under playground equipment, landscaping mulch (chips), running track material (granular or molded), and filler material used with many synthetic turf sports and playing fields (granular).

The use of tire crumb materials for playground and turf fields provides numerous benefits. First, it cushions falls, reducing sports injuries when compared with other playground or athletic surfaces. Second, synthetic turf is a low-maintenance alternative to natural grass, as there is no or reduced need for water, fertilizers, or pesticides. Because turf fields are installed with below-ground drainage systems, there is reduced waiting time after storms, which promotes their use. Third, reusing expended tires reduces their potential as disease vectors (e.g., water hosting mosquitoes) and reduces the burden on landfills.

There have been increased reports in the media of parents becoming alarmed when their children returned home with tire crumb particles or fragments adhering to their socks and clothing picked up while playing on tire-crumb-surfaced playgrounds and turf fields. The U.S. Environmental Protection Agency (EPA) Region 8 asked several EPA program offices to help understand the extent of crumb rubber recreational uses, fill critical data gaps, and assess the available data to determine if there was any unreasonable exposure or risk, particularly to children. In response to this request, an Agency-wide workgroup was formed to assess the existing information and determine whether the Agency needed to collect additional information. The workgroup included representatives from the various program, policy, scientific, and communications staff, including the Office of Children's Health Protection and Environmental Education (co-lead), the Office of Pollution Prevention and Toxic Substances, the Office of Solid Waste and Emergency Response, the Office of Research and Development (ORD; co-lead). and several EPA regional offices. The workgroup requested that a smaller science workgroup familiar with planning and conducting environmental field studies be formed to consider the quality of the current science and make recommendations regarding the need for future research, if any is needed.

This scoping study was proposed, designed, and recommended by the science workgroup as a means for evaluating readily available methods and to generate consistently collected U.S. data that could be used to help inform decisions regarding possible next steps to address questions from the public regarding the safety of tire crumb infill used in ball fields and playgrounds. This study was not intended to address the very large number of variables that might impact environmental concentrations or potential exposures (e.g., manufacturers, materials, installation practices, spatial/temporal differences, age, use). The limited study data were intended to complement data collected or planned for collection by other State and Federal agencies. Although this study included collection and analysis of environmental samples that may be associated with several synthetic turf components, the focus of EPA's work is developing and evaluating methods for characterizing tire crumb constituents. Analysis of the other components was included to better understand the relative portion of any observed tire crumb constituent environmental levels measured in the various samples. This study may complement research performed by the Consumer Product Safety Commission (CPSC); the States of California, New Jersey, New York, and Connecticut; and New York City regarding synthetic turf, but is distinct from the other studies in that the focus is on the tire crumb material. Prior to preparing for a scoping study, a search of the scientific literature revealed limited environmental or exposure measurement data associated with the use of tire crumb rubber for U.S. recreational fields. Only a few peer reviewed laboratory or environmental studies were reported, with many of these studies conducted in Europe.

Although the results were limited, the search identified a number of compounds and metals that *may* be found in tires, although not all of these compounds and metals are contained in every tire nor are they contained in the same concentration in any tire at any given time. These compounds and metals include those that follow.

Acetone Polycyclic aromatic Manganese Aniline hydrocarbons Mercury Styrene-butadiene Nickel Benzene Benzothiazole Toluene Sulfur Chloroethane Trichloroethylene Zinc Halogenated flame Arsenic Pigments retardants Barium Nylon Isoprene Cadmium Polyester Methyl ethyl ketone Rayon Chromium Methyl isobutyl ketone Latex Cobalt

Naphthalene Copper Phenol Lead

1.2 Exposure Science Questions

A series of general science questions was considered before the study protocol was developed; they include the following ones.

- Can existing collection and analysis approaches and methods be used to assess environmental concentrations of tire crumb rubber constituents at synthetic turf fields and playgrounds?
- How well do such methods perform under real-world conditions?
- Do the methods produce data of sufficient quality to characterize potential exposure routes and pathways?
- Do the methods produce data of sufficient quality to characterize the contribution of constituents to various sources?
- Are the data and information produced through this research, when included with data from other studies, useful for developing hypotheses and informing the design of future research, if needed?
- What new methods are needed to fully characterize tire crumb environmental concentrations and to understand potential exposure routes and pathways?

1.3 Project Objectives

The science workgroup planned a very limited scoping-level field measurement study during the 2008 summer/fall season to

- evaluate, through real-world measurements, the application of readily available sampling and analysis methods for characterizing environmental concentrations of selected tire crumb contaminants in and around playgrounds and synthetic turf fields;
- evaluate the overall study protocol (monitoring, analytical, and QC procedures) for generating
 the quantity and quality of environmental measurement data needed to characterize the
 contribution of the tire crumb constituents to environmental concentrations;
- generate a limited set of consistently collected field measurement data from a few playgrounds and synthetic turf fields that, along with other study data, may be used to

- develop insights regarding the importance of the various exposure routes and pathways and inform the decision regarding future research, if any is needed; and
- understand the factors influencing the development and implementation of future study protocols.

1.4 Study Limitations

This study was designed as a limited scoping-level methods evaluation study. It was planned based on readily available resources (personnel, equipment, media, etc.) and in consideration of the workgroup's desired study time period, the 2008 summer and early fall months when high ambient temperatures should result in conditions promoting the greatest potential for release of tire-related constituents. The study collected limited environmental data to help understand and assess methods for characterizing potential route and pathway-specific exposures (inhalation, ingestion, and dermal) based on selected sentinel species. This study and the resulting data have many limitations, which are described below.

- The study was not designed to provide representative U.S. environmental measurement data for all tire crumb constituents or applications, nor to make conclusions regarding differences in environmental concentrations or potential U.S. exposures to field and playground tire crumb constituents based on geographical location, type of recreational field, manufacturing materials, age, use, or conditions. Resource constraints prohibited the survey, coordination, and random selection of U.S. playgrounds and turf fields and the use of the study data in supporting statistical analysis or making statistical inferences. The study results can be used only to describe the playgrounds and turf fields monitored.
- The number of samples collected at each site was relatively small and will not necessarily support the spatial characterization of the species concentrations across the monitored area.
- Sampling was planned to be conducted only on one day. Therefore, temporal characterization of the targeted environmental contaminants will not be supported.
- No personal exposure data or related information were collected.
- No scripted activities were planned or conducted. The study results were dependent on normal activity levels by the individuals using the playground or turf field. However, the limited data collected in this study likely will not be useful in characterizing differences associated with these factors.
- The study did not evaluate methods for all the reported tire crumb constituents. Sampling and analysis of semivolatile organic compounds (SVOCs; e.g., benzothiazole, aniline, polycyclic aromatic hydrocarbons [PAHs]), reported in many studies, were not performed because of resource limitations.
- Validated sampling approaches and analysis methods were not available for real-world
 playground and synthetic turf field conditions. Currently accepted methods for measurement
 and analysis of the targeted species in indoor and outdoor microenvironments and in soils
 were used, with modifications required in some cases.
- QC/QA activities were implemented to document the quality of the sampling and analysis measurements; however, suitable QA/QC materials and standards were not available for some of the types of samples.

2. Conclusions and Recommendations

The following narrative provides key highlights regarding the approach and methods that were applied and assessed in this scoping study. The study results are being provided to the workgroup for assessment and interpretation.

2.1 Implementation of the Study Protocol

- A small scoping-level study protocol was fully implemented at two synthetic turf fields and one
 playground. The protocol was successfully implemented at one of these fields on a second
 day, providing a set of unplanned consecutive day data. Additional data were collected from
 four turf fields and for tire crumb from a second playground.
- The study's success reflects the excellent collaborations and contributions of scientists and staff across many program offices, regions, States, and ORD.
- The protocol, and a majority of the corresponding methods employed in this study, generated
 quantitative data that can be used to characterize the contribution of tire crumb constituents to
 the environmental concentrations measured at the synthetic turf field, playground, and
 background sampling locations.
- Although none of the methods have been validated for this specific application, most methods
 were able to provide measurement data of known quality and at concentrations adequate for
 assessing potential tire crumb constituents.
- Air particle collection required considerable time, equipment, and expertise.
- Other collection procedures (air volatile organic compounds [VOCs]), wipe, and material collection) required much less time, equipment, and expertise.
- In general, the study protocol can be implemented and will yield data for assessing environmental concentrations and potential exposures for tire crumb constituents for various routes and pathways. However, when considering the design and implementation of future studies, the research needs to carefully consider
 - issues associated with identifying and gaining site access,
 - the value of the data being generated versus the resource burden, and
 - the implementation of other methods for generating data to address specific research hypotheses.
- Any future study will need a corresponding carefully developed and implemented communications plan to help promote the value of the research and gain access to the required facilities.

2.2 Air Sampling and Analysis

- The air sample collection and analysis methods provided data suitable (both quality and concentration levels) for assessing environmental levels of particles, metals, and VOCs in air.
- Air particulate matter sampling employing relatively large (carry-on-size suitcase), battery-operated pumps and size selective inlets yielded sufficient particle mass for measuring selected metals at commonly reported ambient air levels. This sampling approach required significant resources (equipment and experienced field staff) and long setup and sampling durations (8 to 10 h).
- Collecting air VOCs via grab sampling during the hottest daytime period (conditions when the
 greatest emissions from tire crumb material were anticipated) was simple and required little
 time (~1 h).
- The air VOCs methods generated concentration data for many compounds. Slightly elevated MIBK levels were found at one turf field. The reproducibility in the data approximates what previously has been reported in other field measurement studies. The use of an integrated

- sampling pump method likely would increase the number of species with reportable data but would not necessarily generate substantially different data for characterizing tire crumb source contributions, environmental concentrations, or potential exposures.
- The air particulate matter (PM) mass and metals methods yielded reproducible results, with the turf field concentrations approximating the background levels. Concentrations on one playground site were somewhat higher than the background concentration.
- Tire crumb related fibers were not observed in the air samples analyzed by scanning electron microscopy (SEM). The SEM data were not sufficient for source apportionment or attribution of the data to any tire crumb constituent because of the variability of compositional or morphological characteristics of particles associated with the tire crumb material collected in this study.

2.3 Surface Wipe, Tire Crumb, and Turf Blade Sampling and Analysis

- No evaluated method was available for assessing dermal and indirect ingestion from tire crumb constituents in turf field or playground surfaces. A standard surface wet wipe sample collection method for residential lead (Pb) measurement was used at the synthetic turf fields. This method performed reasonably well for assessing extractable metals and required modest skills and time (~1 h).
- Collection of tire crumb infill and turf blade material at synthetic turf fields and tire crumb at
 playgrounds was straightforward, requiring minimal skills and resources (~1 h). Convenience
 samples were collected in this study based on the materials being readily available on the
 surface. There is evidence that the material is not homogeneous with regard to some
 constituents (Pb for example). Future site characterization studies should be considered to
 evaluate the issue of sample heterogeneity and the impact on data interpretation.
- Wipe, tire crumb, and turf blade samples were extracted using EPA Method 9200.1-86 for in vitro Pb bioaccessibility and EPA Method 3050B for total extractable Pb (and other metals). Both extraction techniques were combined with EPA inductively coupled plasma (ICP)/MS Method 6020A. These methods require extensive skill and resources. Multiple analyses of sample extracts with varying dilutions were required to capture the range of elements and concentrations within appropriate calibration parameters.
- The in vitro Pb bioaccessibility method was judged not appropriate for the surface wipe samples. Because the in vitro method has been validated only for soil samples, additional validation studies would be required to fully demonstrate the relevance of the method for tire crumb and turf blade materials.
- Although the methods appeared to perform reasonably well, a number of sample handling, size, and heterogeneity issues were discovered that may affect method performance and data interpretation.
- There is a lack of appropriate QC/QA materials and spiking methods. QA/QC materials and procedures need further development for the methods as applied to these materials.
- The wipe, tire crumb, and turf blade data identified a potentially significant variability in source contribution based on turf field blade color and type, along with the tire crumb fraction being analyzed. Additional research is needed to understand the factors influencing the reported variability before future studies are designed and conducted. Understanding the variability is important in developing improved approaches for site characterization.

2.4 Conclusions with Regard to the Exposure Science Questions

• Can existing collection and analysis approaches and methods be used to assess environmental concentrations of tire crumb rubber constituents at synthetic turf fields and playgrounds? Yes, existing air sampling and analysis methods can be used. Existing methods

- for analysis of metals in synthetic turf field and playground components can be successfully applied, but may require additional validation assessments.
- How well do such methods perform under real-world conditions? The air sampling and analysis methods evaluated performed well. Methods for analysis of metals in synthetic turf field and playground components showed good precision, but the assessment of recovery for some metals was difficult because of the nonhomogeneity of the bulk materials.
- Do the methods produce data of sufficient quality to characterize potential exposure routes and pathways? In most cases, the methods appeared to produce data of sufficient quality with regard to sensitivity, precision, and accuracy. Additional validation efforts may be needed to interpret measurement results, particularly with regard to bioaccessibility of metals in synthetic turf field and playground components.
- Do the methods produce data of sufficient quality to characterize the contribution of constituents to various sources? Some of the methods generated data of sufficient quantity and quality. Research is needed to better understand relative source contributions, in particular for the wipe and air particle samples.
- Are the data and information produced useful, when included with data from other studies, for developing hypotheses and informing the design of future research, if needed? The assessment of approaches and methods tested in this scoping study, in combination with research recently completed and ongoing by other organizations, will be very useful for developing hypotheses and informing the design of future research, if needed.
- What new methods are needed to fully characterize tire crumb environmental concentrations
 and to understanding potential exposure routes and pathways? Testing and application of
 personal sampling methods would provide a more complete understanding of how
 environmental concentrations translate into potential exposures. Methods for collection and
 analysis of SVOCs were not tested in this scoping study but would be needed for a full
 characterization.

3. Scoping Study Approach

3.1 Scoping Study Goals

The primary goal of this scoping study was to evaluate readily available methods and approaches for characterizing environmentally available concentrations of selected contaminants at synthetic turf fields and playgrounds that include tire crumb material. There are currently no known validated sampling and analysis methods for these types of installations and materials. Integrated and/or grab air, wipe, and material sample collection and analysis methods (Table 1) were selected based on professional evaluation. Where available, standard methods used routinely to characterize the targeted environmental contaminants in other microenvironments were selected. However, because of time and resource constraints, none of these methods were evaluated for the intended study application. A list of the sample collection and analysis methods used or developed for this study is provided in Appendix A. The detailed methods were included in the approved study QA project plan.

A number of constraints influenced the decisions on proposed methodologies. These included limited resources (e.g., time, people); an anticipated lack of readily available electrical power at the sites; uncertainty in sample collection times because of site availability or activity issues; the need for equipment that can be shipped to multiple sites across the country; and the need for rugged, simple methods that could be implemented consistently by minimally trained technical staff at several sites.

3.2 Organizations

The scoping study approach was developed based on the cross-Agency collaborative effort outlined below.

- National Exposure Research Laboratory (NERL): Prepare and ship sample collection equipment and media (less VOCs [see below]). Provide technical support for measurements. Analyze air filter media for mass, metals, and morphology. Analyze tire crumb material, turf blade material, and surface wipes for metals.
- EPA Regions 4, 5, and 9: Identify, assess, and coordinate access to the study sites. Communicate study to the public.
- EPA Region 1: Prepare VOC sampling media (canisters) and conduct TO-15 analyses.
- Workgroup: Assessment and interpretation of the study data provided in this report in context with other research data and Agency compliance guidelines following receipt of this report.

3.3 Selection of Target Analytes

Target analytes in this study were selected based on a combination of three factors: (1) chemicals that have been associated with tire material (see Section 1.1); (2) chemicals that have been reported in other measurement studies at synthetic turf fields or playgrounds or are of interest for these types of facilities; and (3) chemicals that could be analyzed using the methods and resources that were readily available for this study. Methyl isobutyl ketone (MIBK) was selected as a potential marker for emissions of volatile tire components into the air. PM_{10} was selected because it may occur from physical degradation of tire crumb material and its potential for activity-related suspension into the air. PM_{10} particles are of interest because they may be inhaled and also swallowed following trapping by mucus membranes. The metals Pb and chromium (Cr) were of interest both because of their potential presence in tire material, and also because they have been shown to be associated with pigments used in some types of synthetic turf blades.

The metal zinc (Zn) was of interest as a potential marker for tire crumb material. Other metals were of secondary interest because of their potential association with tire material or because they can provide additional particle source information. In some cases, additional VOC or metal

Table 1. Summary of Sample Collection and Analysis Methods

Table 1. Summary of Sample Collection and Analysis Methods						
Sample Type	Sites	Sampling Method	Analytical Method	Target Analytes		
Air Particulate Matter (PM ₁₀ — particles with	Synthetic turf fields	ORD/NERL Research Protocol	ORD/NERL Research Protocol	PM ₁₀ mass		
aerodynamic size <10 µm)	Playgrounds	SKC pump, gel-cell battery, Harvard 10-µm impactor, 37-mm Teflon filter, 20-L/min flow rate 1-m sampling height, three sites on/near playground/field, one site for background	Gravimetric analysis			
Air Metals in PM ₁₀	Synthetic turf fields	Same sample as collected for PM ₁₀ mass.	ORD/NERL Research Protocol	Primary: Pb, Cr, Zn		
	Playgrounds		XRF (X-ray fluorescence)	Secondary: Ca, Cl, Cu, Fe, K, Mn, S, Si, Ti		
Air Particles/	Synthetic	ORD/NERL Research	ORD/NERL Research	Particle		
Fibers for Scanning	turf fields	Protocol	Protocol	morphology		
Electron Microscopy (SEM)	Playgrounds	SKC pump, gel-cell battery, Harvard 10-µm impactor, polycarbonate filter, 10 L/min Same sites and height as PM ₁₀	SEM (scanning electron microscopy)	Particle size distribution Attempt to characterize tire crumb composition signature		
Air Volatile Organic Compounds (VOCs)	Synthetic turf fields	Method TO-15 6-L Summa canisters	TO-15 GC/MS (gas chromatography/mass spectrometry)	Primary: Methyl-isobutyl- ketone		
	Playgrounds	Grab sample collected at approx. 2:00 pm, or hottest time of day when access to the field is possible. 1-m sampling height, three sites "on playground/field", one site for background		Secondary: 55 other alkane, aromatic, oxygenated, and halogenated compounds		

Table 1. Summary of Sample Collection and Analysis Methods (cont'd.)

Table 1. Summary of Sample Collection and Analysis Methods (cont'd.)					
Sample Type	Sites	Sampling Method	Analytical Method	Target Analytes	
Surface Wipe	Synthetic	ASTM E1792 wipes	EPA Method 3050B, acid	Primary:	
	turf fields	(Ghost Wipes) and	digestion with	Pb, Cr, Zn	
		ASTM E1728 dust	determination by EPA		
		collection method.	Method 6020A (ICP/MS	Secondary:	
		This is a standard wet	inductively coupled	Al, As, Ba, Cd,	
		wipe method.	plasma mass spectrometry)	Cu, Fe, Mn, Ni	
		Collect wipes from		Pb	
		three 1-ft ² sites on turf	RBALP in vitro extraction		
		fields.	(EPA Method 9200.1-86) for bioaccessible lead,		
		Wipes placed in	and determination using		
		precleaned 50-mL	ICP/MS by EPA Method		
		polyethylene	6020A		
		container.			
		Collect a second			
		sample wipe next to			
		each original			
		sampling location for			
		archival or possible			
		metals bioavailability			
T' - O I	0 . 11 . 11 .	analysis	EDA Marilla LOOGOD and L	D :	
Tire Crumb	Synthetic	ORD/NERL Research	EPA Method 3050B, acid	Primary:	
Material	turf fields	Protocol	digestion, and	Pb, Cr, Zn	
(crumbs from playgrounds	Playgrounds	Collect samples of	determination by EPA Method 6020A (ICP/MS)	Cocondom	
and infill	Playgrounds	crumb material from	Wethod 6020A (ICP/WS)	Secondary: Al, As, Ba, Cd,	
material from		three sampling	RBALP in vitro extraction	Cu, Fe, Mn, Ni	
synthetic turf		locations on each	(EPA Method 9200.1-86)	Cu, re, will, M	
fields)		playground or field.	for bioaccessible lead,	Pb	
ileius)		playground of field.	and determination using		
		Add material to clean	ICP/MS by EPA Method		
		HDPE bottle.	6020A		
		Collect a second set			
		of samples for			
		archival for possible			
		metals bioavailability			
		analysis or SVOC			
		analysis.			

Table 1. Summary of Sample Collection and Analysis Methods (cont'd.)

Sample Type	Sites	Sampling Method	Analytical Method	Target Analytes
Turf Blades	Synthetic	ORD/NERL Research	EPA Method 3050B, acid	Primary:
	turf fields	Protocol	digestion, and	Pb, Cr, Zn
			determination by EPA	
		Collect loose blades	Method 6020A (ICP/MS)	Secondary:
		from field surface in		Al, As, Ba, Cd,
		several sampling	RBALP in vitro extraction	Cu, Fe, Mn, Ni
		locations on the field,	(EPA Method 9200.1-86)	
		collect samples of	for bioaccessible Pb, and	Pb
		different colors where	determination using	
		possible, place blades in a clean	ICP/MS by EPA Method 6020A	
		50-mL polyethylene	6020A	
		container.		
		Container.		
		Where there is		
		sufficient material,		
		archive material for		
		possible metals		
		bioavailability		
		analysis.		

analytes were included because they could be measured as part of the routine analysis of analytes of higher interest. As noted earlier, semivolatile chemicals, such as benzothiazole and PAHs, were of interest but were not measured in this scoping study because of the lack of readily available resources.

3.4 Proposed Sampling Sites and Sampling Locations

A study goal was to collect real-world environmental samples in four geographical areas across the United States (located near the four NERL laboratory locations) in late summer and fall of 2008:

- Athens, GA (EPA Region 4),
- Research Triangle Park, NC (EPA Region 4),
- Cincinnati, OH (EPA Region 5), and
- Las Vegas, NV (EPA Region 9).

The recommended approach relied on available NERL technical staff to implement the sampling protocol and the use of the laboratories' facilities as staging areas. In each geographical region, two sampling sites were to be defined: (1) a playground with crumb rubber material and (2) a synthetic turf field with tire crumb rubber infill. The proposed design would result in sampling at four playground sites and four synthetic turf field sites. Based on availability and access, alternate approaches were to be considered regarding the number of sites to be monitored in an area.

At a given sampling site (turf field or playground), four sampling locations were to be selected: three "on field" sampling locations and a background sampling location. The proposed "on field" sampling location configuration was an isosceles triangle, with one sampling location near the center of the playground or field and the other two at approximately equally distanced downwind positions. Actual deployment configuration was dependent on the site layout, planned activities, and wind direction. The background sampling location was intended to be within 100 m of the field or playground, over a natural grass surface when possible, and not in close proximity to likely pollutant sources. At each of the four sampling locations, all the following environmental samples and measures were to be collected (nominal, except where noted):

- integrated air PM₁₀ sample for particle mass and metals composition,
- integrated air PM₁₀ sample for particle morphology (size and shape),
- grab air sample for VOCs,
- wet wipe for metals composition (not at the background sampling location), and
- materials (tire crumb and turf fibers).

All air sampling was to be conducted with the inlet at a height of 1 m. Additional duplicate samples and measures were collected at one sampling location at each site. Standard meteorological measures (temperature, relative humidity [RH], and wind speed and direction) were taken periodically at each site sampling location. Activity levels (e.g., number of activities, number of individuals, type of activity) also were recorded. A 1-m² square plastic child barrier was set up around each sampling location to prevent the participants from running into or falling on the sampling equipment.

3.5 Sampling Considerations

There is no standard approach for determining the number of sample collection locations or the timing of sample collection at any one playground or synthetic turf field site. Key factors that were considered included

- potential variability of materials and chemical concentrations within a site;
- potential variability of activities at a site over time;
- meteorological conditions, particularly moisture, temperature, and wind speed and direction;
 and
- contribution of ambient background levels or nearby source contribution of the targeted chemicals to onsite measurements.

Each key factor is briefly described below, along with the proposed approach taken to minimize or characterize the impact on the resulting data.

3.5.1 Material Variability

Factor: Materials and chemical concentrations could vary within a site resulting in variation of targeted species across space and time at the playground or turf field. However, few data were available regarding the variability in contaminant concentrations within a playground or synthetic turf field site. Also, there was little information available to guide optimum locations for sampling at a site.

Proposed Approach: Three sampling locations were selected within the boundaries of the playground or turf field in areas close to the anticipated activity. These sampling locations were positioned such as to not interfere with normal activity or use. An additional background sampling location was selected near (~20 to 100 m) and upwind from the playground or turf field to characterize ambient background levels. This approach was implemented successfully for air samples with the exception that only two "on playground" sampling locations were set up at the playground because of the small size of the area. In addition, the tire crumb infill material and synthetic turf blades were collected where available, rather than at predetermined locations.

3.5.2 Activity Variability

Factor: Activities could vary over time at a site. Activity levels for the sites and sample collection locations could be highly variable within and between sites. This study was a scoping environmental measurement and methods development study. Therefore, no scripted activities and no personal measurements were implemented. Activity levels may affect air particle measurements. However, activity levels are unlikely to affect air VOC measurements, surface wipes, and tire crumb and turf blade material grab sampling. The normal use or activities at one site might require one or more of the samplers to be deployed near but not directly on the

playground or turf field. In this case, the samplers were placed as close to the activity as permitted.

3.5.3 Sample Volume for Detection Limit

Adequate sample volumes are needed to obtain reasonable detection limits for airborne particles and metals on particles. Therefore, equipment and methods for particle air sampling were selected to provide flow rates of 20 L/min over a nominal 8-h sample collection period. If the particle air sampling duration was applied to match an ongoing site activity of less than 8 h, then detection limits for those samples would be higher. A minimum sample collection time of 2 h for air particle samples was selected.

Proposed Approach: Where practical and permitted, air particle samples were collected in close proximity to where activities were ongoing, but without interfering with the normal activities or use of the playground or turf field. Information about extant activities at each site was collected. This approach was implemented successfully.

3.5.4 Meteorological Conditions

Factor: Meteorological conditions, particularly moisture, temperature, and wind speed and direction, might impact sample collection decisions and potential emissions. Meteorological conditions may influence air particle and air VOC measurements at playground and turf field sites. Wind will transport airborne pollutant species away from the site and will transport ambient pollutant species onto the site. Suspension and resuspension of particles likely will be affected by meteorological conditions. Temperature likely will influence the VOCs that might be emitted from tire crumb or other synthetic turf materials. With higher temperatures, higher levels of VOCs emissions would be anticipated.

Proposed Approach: This study was designed to collect air samples during those meteorological conditions that likely would result in the highest emissions (i.e., hot, dry, and calm days). Sampling was scheduled in August and September on days when no rain had occurred on the previous day and when no rain was anticipated, with anticipated wind speeds <10 mph. Air VOC samples were collected during the hottest time of day (~2 p.m.) at each "on field" or "on playground" sampling location at the site. Air sampling locations were selected, where possible, to offset potential changes in wind direction. Portable meteorological measurement stations were not deployed. Basic information about meteorological conditions (temperature, wind speed, and approximate wind direction) was collected at each site using a handheld measurement device. In general, this approach was implemented successfully.

3.5.5 Background Contribution

Factor: Ambient background pollutant levels could contribute to onsite measurements, particularly for air samples. Ambient contaminants also may contribute to the total burden at the playground or turf field as a result of aerosol or dust deposition.

Proposed Approach: At each playground or synthetic turf field site, one background sampling location was collected upwind from the playground or turf field. A set of air samples identical to the other sample collection locations was collected at the background sample collection location. The resulting data were intended to be used to characterize the potential contribution of ambient or background air contaminants to the playground or turf field. This approach was implemented successfully.

4. Methods

4.1 Air VOC Samples

Grab air samples for VOCs were collected using evacuated 6-L Summa-polished stainless steel canisters during the hottest time of day (~2 p.m.). This collection time was selected for likely highest air and surface temperature conditions promoting VOC emissions. Air temperature and surface temperature were measured and recorded at the time the samples were collected. Each sample was collected by opening the canister valve and allowing the evacuated canister to fill with air over an interval of approximately 20 s. The canister valve then was closed, and the canisters stored at ambient temperature until analysis. The canister samples were analyzed at the EPA Region 1 Office of Environmental Measurement and Evaluation for 56 VOCs by gas chromatography/mass spectrometry (GC/MS) following EPA Method TO-15.

4.2 Air PM₁₀ Particle Samples for Mass and Metals Concentrations

 PM_{10} is defined as airborne particulate matter with an aerodynamic size less than 10 μ m. Ambient air was sampled at a nominal flow rate of 20 L/min (LPM) using metered, direct-current-supplied active samplers (SKC-HV-30 air pumps) and Harvard Impactor inlets (Air Diagnostic and Engineering), enabling PM_{10} mass loading on 47-mm Teflo filter media (Williams et al., 2008). Air monitoring was initiated for all monitors in quick order on their setup and calibration and continued without interruption through the monitoring event (day). At the conclusion of the sampling event, filter samples were recovered, stored in sealed transportation containers, and returned to the laboratory under ambient temperatures. The sampler ending flow rate was checked.

Filters were returned to the NERL Research Triangle Park, NC, gravimetric weighing facility, which operates under Federal Reference guidelines for temperature and relative humidity specifications (22 ± 0.5 °C, $35 \pm 1\%$ RH). The filters underwent a 24-h equilibration period prior to mass loading determination (Chen et al., 2007). Filter mass loadings were determined as the difference between presampling (tare) weights and those obtained following postsampling using a Sartorius MC 5 microbalance. The differential mass loading and data pertaining to the total volume of air sampled through each individual filter then was used to calculate the air mass PM₁₀ concentration in units of micrograms per cubic meter for each sampling location. Immediately following gravimetric analysis, the PM₁₀ mass concentration filters were released to the NERL X-ray fluorescence (XRF) laboratory for metals analysis. Metals analysis was performed for 44 selected metals using the NERL's unique Lawrence Berkeley National Laboratory-designed spectrometer (Williams et al., 2008).

4.3 Air PM₁₀ Particle Sample Collection for Scanning Electron Microscopy

Sample collection for assessing air particle morphology and selected particle metals composition using SEM was conducted similarly to the primary PM₁₀ mass and metals sample collection method. Identical SKC HV-30 pumps and similar Harvard Impactor samplers were used, the only differences being the operation of the units at a lower flow rate (10 LPM) to overcome observed filter pressure drop issues affecting pump battery life and run time. Specialized 37-mm polycarbonate filter material (Nuclepore) needed for SEM analyses was used.

4.4 Surface Wipe Sample Collection—Synthetic Turf Fields

Surface wipe samples were collected at synthetic turf field sites using a wet (water) wipe (Environmental Express, Ghost Wipe No. 4210) conforming to American Society for Testing and Materials (ASTM) E1792-03 requirements. Samples were collected at times when it was safe to

do so with regard to any activities occurring on the field. Sample collection time was not critical for these samples. Two co-located samples were scheduled for collection at the three "on field" sampling locations. No background sampling location wipe sample was collected. Samples were collected following the ASTM E1728-03 method, a standard wet-wipe method for collecting dust from indoor floor surfaces that used water as the wetting agent. Specifically, a 1-ft² template was placed on the surface of the field. Using clean, powderless plastic gloves, the field sampling technician removed the wet wipe from the foil packet. Using one side of the wipe, the turf surface was wiped in a U-shaped pattern within the template area. After folding the wipe in half to get a fresh wipe surface, the area was wiped again in a U-shaped pattern perpendicular to the first wipe pattern. The wipe was then folded in half again and the edges near the interior portion of the template were wiped. Finally, the wipe was folded again and placed in a precleaned 50-mL polyethylene tube (Environmental Express, Disposable Digestion Cup No. SC475) for storage. The tube was tightly capped and transported at ambient temperature to the laboratory, where the samples were placed in a freezer at -20 °C.

4.5 Tire Crumb Infill Material Sample Collection—Synthetic Turf Fields

Tire crumb infill material was collected at the synthetic turf field sites. Samples were collected from one or more areas primarily based on availability of infill material (small tire crumb granules) at the surface of the field. These sampling locations did not necessarily correspond to the air particle sampling sites. Samples were collected at times when it was safe to do so with regard to any activities occurring on the field. Sample collection time was not critical for these samples. No background sample was collected. Infill material was scooped into a precleaned 50-mL polyethylene tube (Environmental Express, Disposable Digestion Cup No. SC475) for storage. The tube was tightly capped and transported at ambient temperature to the laboratory, where the samples were placed in a freezer at -20 °C.

4.6 Blade Material Sample Collection—Synthetic Turf Fields

An attempt was made to collect samples of the loose "grass blades" at synthetic turf field sites. No destructive sample collection was allowed, so blades were not cut or harvested from the turf fields. Where possible, samples were to be taken for each color of turf blades on the field. Sampling locations did not necessarily correspond to the air particle sampling sites. Samples were collected at times when it was safe to do so with regard to any activities occurring on the field. Sample collection time was not critical for these samples. No background sample was collected. Blades were collected using cleaned plastic forceps and were placed into a precleaned 50-mL polyethylene tube (Environmental Express, Disposable Digestion Cup No. SC475) for storage. The tube was tightly capped and transported at ambient temperature to the laboratory, where the samples were placed in a freezer at -20 °C.

4.7 Tire Crumb Material Sample Collection—Playgrounds

Two different approaches were used for sample collection at playgrounds. For the first approach, sample collection locations were approximately adjacent to the "on playground" sampling locations. Sample collection time was not critical for these samples. No background sample was collected. Tire crumb material was intended for collection from an approximate 4" x 4" square, with material collected from the surface down to ground level at each site. Material was collected using forceps or another appropriate tool, and crumbs were placed into a 250- or precleaned 500-mL, high-density polyethylene wide-mouth bottle (SciSpec Scientific Specialties Service, Inc, No. 353008 or No. 353016) for storage. The bottle was tightly capped and transported at ambient temperature to the laboratory, where the samples were placed in a freezer at -20 °C. At the second playground, a simple collection of tire crumb rubber material was performed. Samples were placed in polyethylene bags and were mailed to the laboratory.

Once received there, the samples were placed into high-density polyethylene bottles and stored in a freezer at -20 °C.

4.8 SEM Sample Preparation and Analysis

4.8.1 Sample Preparation

Ambient samples: 5 mm x 5 mm sections were cut from each polycarbonate filter using a stainless steel scalpel. Each section was affixed to a standard 12-mm aluminum specimen stub using a double-sided, sticky C tab. The samples were then coated with ~200 Å of C to minimize sample charging by the electron beam during SEM analysis.

Source material samples: Individual crumbs from the bulk material sample, typically 2 to 3 mm in size, were deposited "as is" on a sticky C tab. Source particles closer in size to the ambient sample were generated by shaving pieces from larger crumbs using a stainless steel razor blade. Source samples were coated with ~200-Å film of conductive C to minimize charge buildup on the sample during SEM analysis.

4.8.2 SEM Sample Analysis

Samples were analyzed by SEM and Energy-Dispersive X-ray Spectrometry (EDX) using the Personal SEM (R.J. Lee Instruments Ltd.) in the NERL Electron Microscopy Laboratory. Manual SEM/EDX analysis was first conducted on the bulk tire crumb source samples. Chemistry and morphological features characteristic of the tire crumb material were identified to help identify tire crumb particles in the ambient samples. Ambient samples were analyzed by computer-controlled SEM (CCSEM/EDX). Instrument parameters for the CCSEM analyses included 20-kV accelerating voltage, backscattered electron (BSE) imaging mode, 16-mm working distance, and zero tilt. The BSE mode yields a more uniform background than the secondary electron (SE) mode, necessary for computer-controlled SEM, but at the expense of some loss in sensitivity for small carbonaceous particles; carbonaceous tire crumb particles about 1 μ m or smaller can be difficult to distinguish from the polycarbonate filter substrate in CCSEM analyses. Thus, small carbonaceous particles may be underreported in these analyses.

The CCSEM analysis was set up to analyze particles with average diameters between 1 and 20 μ m. Few particles >10 μ m, however, were observed in any sample. All particles within this size range were sized automatically and analyzed by EDX for chemistry. Based on the analyses of the tire crumb source samples sulfur (S), Zn, and C were identified as possible indicators of tire crumb material. Rules were developed to optimize the search for tire-crumb-like particles by extending the X-ray analysis time (10 s) and saving low-resolution images for all particles containing S, Zn, or C. Images and spectra for these particle types were reviewed manually offline, and particles were judged subjectively to be either tire-crumb-like, or not tire crumb material based on the particle morphology and chemistry.

Only a small fraction of the 6.7 cm² deposit area of each ambient filter was analyzed by CCSEM, typically about 1 mm², to complete each analysis in a reasonable time. Following CCSEM analyses, the EDX spectra and images of the particles of interest were reviewed manually, particles were relocated in the SEM for further examination, and suspected tire crumb particles were flagged.

4.9 Surface Wipe, Tire Crumb, and Turf Blade Sample Metals Analysis

Surface wipe samples, tire infill, tire crumb, and turf blade samples were received and prepared for analysis. Detailed sample descriptions were recorded because it was observed that the blade, infill, and crumb samples were not homogeneous. The playground tire crumb sample pieces were quite large and heavier than the normal sample size used for Pb in vitro

bioaccessibility extractions (typically 1 g). Five turf blade samples were not processed because they did not meet the minimum sample size of 0.7 g.

Sample Selection and Processing. For wipe samples, the entire wipe was used after removing any obvious turf blades or pieces of the infill material. For the crumb rubber infill samples from synthetic turf fields, 1.0 g aliquots were weighed out after rotating the field collection tubes in the x, y, and z axes for 1 min. For synthetic turf blade samples, the samples were processed with consideration of blade color. If more than one color of blade was present in the sample, representative blades of each color were selected for extraction. For the crumb rubber samples from playgrounds, pieces that appeared representative of the entire sample were chosen for extraction. In the situation where all pieces were very heavy, the piece closest to 1 g was used. A decision was made not to cut the samples as that would introduce fresh unaged surfaces, which potentially could impact the Pb bioaccessibility of the sample. Duplicate sample aliquots were chosen for extraction and spiking where there were adequate quantities.

Sample Extraction. The samples were treated as "soil" for the scoping study. Existing extraction procedures already in place were used. A consecutive extraction approach was taken because of the small number and amount of samples collected in the field.

First, the Pb in vitro extraction procedure EPA 9200.1-86 May 2008 "Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil" was used. Two 10-mL aliquots from the 100-mL extract total were removed for analysis and storage. The extracts were adjusted to 2% nitric acid (HNO₃; v/v) prior to analysis.

To obtain total leachable metals, SW-846 Method EPA 3050B "Acid Digestion of Sediments, Sludges and Soils" was used next. The method was used as written with the following minor modifications.

- After quantitative transfer of the 80 mL of Pb in vitro extract and solids from the in vitro extraction bottles to 250-mL glass beakers, 5 mL of concentrated HNO₃ was added to the in vitro extracts, and the extracts were reduced in volume to 5 mL on a hot plate.
- A maximum of 2 h of acid refluxing was performed (similar to the hot block option extraction time). Neither the tire crumb infill nor tire crumb samples from playgrounds completely dissolved.
- Samples were filtered through a Whatman 25-mm GD/X 0.45-µm cellulose acetate membrane syringe filter, as centrifugation was not adequate to separate the particulates from the solution for analysis.
- Final samples extracts for EPA Method 3050B were in 5% HNO₃ (v/v).

Analysis by ICP/MS. A new X-Series II quadrapole ICP/MS was designated as the preferred instrument despite it still being in "start-up" mode. Therefore, instrument, software, and data processing routines were developed and evaluated concurrent with the samples' analysis. After dilution, the in vitro extracts were 2% HNO₃ (v/v). The EPA 3050B extracts were received as 5% HNO₃ (v/v), and all subsequent dilutions were made by weight with 5% HNO₃ (v/v).

Quantitative analysis for total extractable mass concentration was performed for the primary metals of interest: Cr, Pb, and Zn. In addition, the metals aluminum (Al), arsenic (As), barium (Ba), cadmium (Cd), copper (Cu), iron (Fe), manganese (Mn), and nickel (Ni) were reported; however, QC assessment was not as extensive for these metals.

The instrumental details for the Thermo X-Series ICP/MS are shown in Appendix B as follows: Table B-4 lists the operating parameters, Table B-5 lists masses used and interference correction information, Table B-6 lists the calibration standards used, and Table B-7 lists method detection limits.

The sample extract analysis followed procedures outlined in EPA SW-846 Method 6020A (http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm). Instrument performance indicators are the QC solutions listed in Appendix B, Table B-8. For samples that

needed multiple dilutions to insure that all metals of interest were in the calibration range, the lowest dilution was used for a metal, where it did not exceed the top standard. Total extractable concentrations are calculated from the prorated combination of the in vitro extract concentrations and the EPA 3050B extract concentrations.

4.10 Pb Bioaccessibility Analysis

4.10.1 In Vitro Bioaccessibility Background Information

Methods for assessing Pb bioavailability in soil include in vivo animal studies, in vitro (referred to as bioaccessibility) studies, and mineralogical/speciation studies. In vivo studies quantify the metal present in various tissues and excreta of animals after an animal feeding bioassay is conducted. In vitro methodologies are physiologically based extraction tests designed to mimic the human gastrointestinal system. In vitro methods measure the bioaccessibility (e.g., solubility) of metals during a simulated gastric extraction process.

Bioavailability for this study is defined in the Guidance for Evaluating the Oral Bioavailability of Metals in Soils for Use in Human Health Risk Assessment (OSWER 9285. 7-80) as "The fraction of an ingested dose that crosses the gastrointestinal epithelium and becomes available for distribution to internal target tissues and organs (http://www.epa.gov/superfund/bioavailability/guidance.htm)." A related term pertaining to bioavailability assessment is bioaccessibility. Bioaccessibility refers to a measure of the physiological solubility of the metal at the portal of entry into the body (NRC, 2003). The U.S. EPA guidance document describes the methodologies for predicting lead bioavailability in soil using either an in vivo swine bioavailability bioassay or an in vitro bioaccessibility assay (IVBA). These methods have undergone extensive testing and evaluation, and they "are scientifically sound and feasible methodologies for predicting bioavailability of lead in soil" (OSWER 9285. 7-77). EPA recently published a standard operating procedure (SOP) for an in vitro bioaccessibility extraction for Pb that has been validated against the juvenile swine model (EPA Method 9200.1-86). The in vivo and in vitro methods described are specific to Pb-contaminated soils and Pb bioavailability. Currently, these methods have not been validated for testing other contaminants or media (e.g., tire crumb materials), and these have only been validated by EPA for Pb in soil.

4.10.2 In Vitro Pb Bioaccessibility Methodology

As noted above, validated in vitro methods did not exist for tire crumb samples when this study was conducted. The samples were extracted according to EPA Method 9200.1-86 May 2008 "Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil." This SOP defines the proper analytical procedure for the validated in vitro bioaccessibility assay for Pb in soil (soil which has been homogenized and processed for optimal reproducibility) to describe the typical working range and limits of the assay and to indicate potential interferences. Users of this SOP are cautioned that deviations in the assay method may impact the results (and the validity of the method). Two 10-mL aliquots were removed from the 100-mL extract for analysis and storage. Samples were analyzed by ICP/MS following procedures outlined in EPA SW-846 Method 6020A.

Calculations. The amount of Pb in the in vitro bioaccessibility extraction is calculated by multiplying the extract concentration by the total volume of the bioaccessible extract, which was 100 mL. The in vitro percent bioaccessibility values were determined by dividing the amount of Pb extracted in the in vitro extraction by the total extractable amount of Pb in the sample and multiplying by 100.

4.11 Meteorological and Activity Information

Meteorological information, including air and surface temperatures, wind speed, and wind direction information was collected periodically during each monitoring period when integrated air sampling was conducted. Air and surface temperatures were always measured at the time of air VOC sample collection. Meteorological measurements were made using a handheld Kestral 3000 device. This portable device was used so that multiple measurements could be made at various sampling locations on and around a site. Surface temperatures were made by laying the device on the field or playground surface and waiting for the temperature reading to stabilize. Activities occurring at the synthetic turf field and playground locations, if any, were noted periodically during each monitoring daytime period.

5. Quality Control and Quality Assurance

Appendix B includes the data resulting from the QC/QA procedures implemented in this study and as outlined in the approved QA project plan developed for this study (U.S. EPA 2008). The following narrative summarizes these results.

5.1 Air VOC Quality Control

Air VOC QC results are shown in Tables B-1, B-2, and B-3. Potential background contamination in the sampling and analysis procedure was assessed using field and laboratory blanks (Table B-1). Field blanks were 6-L canisters filled with clean air at the laboratory and transported and stored with the samples collected at three sampling locations. Potential background in the laboratory was assessed using laboratory blanks that were clean air delivered to the analytical system. A laboratory blank was analyzed with each of the five sets of samples. Except for methyl ethyl ketone (MEK), no analytes were detected in any laboratory blank. MEK was measured at levels below the method detection limit in two of the five laboratory blanks. MEK was the only analyte detected in two of the three field blanks, at levels that were about 5 to 10 times lower than the concentrations measured in air samples. The third field blank contained numerous target analytes, often at concentrations exceeding those measured in the samples. For example, the concentration of benzene was 1.1 ppbV, and the concentration of toluene was 29 ppbV. It is possible that this canister had a leak and was contaminated during the storage and transport process. The pressure of each evacuated canister used for sample collection was measured prior to collecting the sample to ensure that the canister did not leak. Two canisters intended for sampling were found to be at ambient pressure prior to use and were not used for sample collection. It is possible that this field blank canister also had a leak, but, because it was filled to ambient pressure at the laboratory, it was not possible to directly assess this prior to field deployment.

Recovery of target analytes (Table B-2) was assessed through the analysis of field and laboratory controls. Field controls were 6-L canisters filled with air fortified with a subset of 30 target analytes. These field controls were transported and stored with the samples collected at three sampling locations. The same mixture of analytes also was analyzed as three laboratory controls to assess recoveries at the analytical step. Except for 1,1,1-trichloroethane and 1,2,4-trichlorobenzene, the mean recoveries of target analytes in field and laboratory controls were within the range of 84% to 114%. The mean recoveries for 1,1,1-trichloroethane and 1,2,4-trichlorobenzene in field controls were 125% and 62%, respectively.

Sampling and analysis precision was assessed using duplicate sample collection and repeat analysis of samples in the laboratory (Table B-3). Duplicate samples were collected at the same site and within a minute of the collection time of the primary sample at three sampling locations to assess the precision of the sampling and analysis procedures. Air collected in sample canisters at three sampling locations was analyzed a second time to assess laboratory analysis precision. Laboratory precision was assessed for 11 analytes with sufficient measureable results. Mean relative percent differences (RPDs) ranged from 2.9% to 15.6% for repeat analyses. Field sampling and analysis precision was assessed for 12 analytes with measurable results. For eight of those analytes, mean RPD values ranged from 1.8 to 21.1%. The mean RPD values were higher for four analytes, including hexane (59.7%), meta- and para-(m&p)-xylenes (38.4%), toluene (85.8%), and methylene chloride (32.4%). Two of the duplicate samples had concentrations of hexane, toluene, and m&p-xylenes that were much lower than their corresponding samples. As discussed below, these two field samples also had higher concentrations of these three analytes than the other samples collected at different sites at the same sampling locations. It is possible that these two field samples were contaminated, resulting in the poor measurement precision.

5.2 Air PM₁₀ Mass Quality Control

A single certified flow calibration device (Bios) was used to document the set point and final flow rate of each sampler deployed in this effort. Flow rates, start and stop times, and sample identification notations were logged into predesignated data collection sheets employed for the field effort. A two-party check system was used during the field sampling effort to ensure that proper recovery of this data was performed, and that sample identification was correct. Two-party checks involved the primary researcher making the initial notation with the reviewer (second party) checking the notation independently for correctness. In addition to the data review conducted in the field, duplicate (collocated) samples were collected during each monitoring episode. This involved setting up a second monitoring station beside the original unit at one of the designated site sampling locations (A,B,C,D). The duplicate sampler was run under the same conditions as the primary unit, and its data were used to determine the precision of the employed methodology. Likewise, field and laboratory blank samples were utilized. Laboratory blanks (filter samples from the same single lot of filters used in the study) were maintained in the gravimetric laboratory to determine the amount of filter mass changes under control conditions. Similarly, field blank samples were deployed in the field at all sites. These samples were transported to the field, placed in the sampling apparatus but did not have any volume of air pulled through them. Filters treated in such a manner would represent the "artifact" mass associated with the sampling effort itself. Following the review of the data validation component of this study, it was determined that both the laboratory blanks and the field blanks had consistently insignificant quantities of mass loadings (≤2 μg/m³), and, therefore, no blank correction was performed on the sample data. Results from comparisons of the field duplicate samples indicate that precision errors ≤10% or mass concentration differences of ~1 to 2 µg/m³ were observed between replicates across all sites. All of these are highly acceptable values relative to normal data quality indicators for field monitoring efforts.

5.3 Air PM₁₀ Metals Quality Control

The NERL XRF laboratory employs a sophisticated QA/QC review of instrumentation during all analyses. For example, the unit associated with this analysis underwent audit trials during the sample analysis runs. Such audits, using National Bureau of Standards or other reference materials, provide the means to determine the accuracy of the current instrument calibration, as well as other parameters. The instrument has to have both precision ($\pm 5\%$) and accuracy ($\pm 10\%$) values from such trials to be considered operational. All such parameters were achieved for the reported analysis results.

5.4 Air PM₁₀ SEM Quality Control

As previously described for the PM₁₀ gravimetric sample collections, field checks were conducted pre- and postsampling relative to flow rates. Duplicate samples were collected at every regional site to assess precision. The two-party review system again was used to ensure proper documentation of field data collections. Laboratory and field blanks were obtained and used to assess data quality (reported in Appendix D. These samples indicate that no sample handling or storage issues impacted SEM data quality.

5.5 Wipe, Tire Crumb, and Turf Blade Sample Quality Control and Quality Assurance

5.5.1 Instrument Performance

Data summarizing the ICP/MS operating parameters, calibration, and method detection limits are provided in Tables B-4 through B-7. Table B-8 provides the ICP/MS criteria for acceptable data. All ICP/MS instrument QCs met the criteria shown in Table B-8 with the

exception of one serial dilution result for Zn (15%) and one serial dilution result for Al (26%) that exceeded the 10% target value. Additional data regarding the results of instrument duplicate aliquot analysis, postdigestion spike recoveries, and agreement between serial dilutions are provided in the sections below.

5.5.2 Recoveries

Extraction performance indicators consisted of recoveries from solution spikes and spikes of sample media (Tables B-9, B-10, and B-11 and National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 2710 spikes (Tables B-12 and B-13). For the eight spikes prepared in the extraction reagent blank (Table B-10), recoveries for Cr. Pb. and Zn were all above 92%. Results for analyte recovery from solutions spiked onto the media of interest varied by media (Table B-11). The Ghost wipes were a uniform media. Recoveries for the three Ghost wipe spiked samples for Cr, Pb, and Zn were greater than 93%. For the tire crumb rubber infill and tire crumb material from playgrounds, there was considerable heterogeneity of the material. There was a visual difference between the sample aliquot used for spiking and the aliquot designated as "sample," which was used to correct for the sample's contribution to the total spiked sample concentration. Recoveries for the four spiked tire crumb rubber infill samples for the three metals of interest varied from 16% to 553%. For the tire crumb samples from playground samples, recoveries range from 17% to 255%. It is probable that these recovery ranges reflect the variability in existing metal content across samples and the inability to correctly subtract the existing content uniformly without additional sample homogenization.

Extraction reagent blanks and Ghost wipe samples were spiked with NIST SRM 2710, where the values for Pb and Zn are certified, and the Cr concentration is provided as "information only" in Tables B-12 and B-13, respectively. For the six extraction reagent blanks spiked with NIST SRM 2710 (Table B-12), the average recoveries for Pb, Zn, and Cr were 87%, 76%, and 37%, respectively. For the three Ghost wipe samples spiked with NIST SRM 2710 (Table B-13), average recoveries for Pb, Zn, and Cr, were 87%, 79%, and 32%, respectively. According to the NIST SRM 2710 certificate addendum and EPA 3050B, the median recoveries for Pb, Zn, and Cr in 2710 using EPA 3050B are 92%, 85%, and 49%, respectively. Note that EPA 3050B is a not total digestion technique but designed to dissolve almost all metals that could become "environmentally available." By design, metals bound in silicate structures normally are not dissolved by the EPA 3050B procedure, as they are not usually mobile in the environment.

5.5.3 Analysis of Blank Materials

Nine extraction reagent blanks (identified as "bottle blanks"), one with each batch of samples, were processed with the samples (Table B-14). The sample data are reagent blank corrected using the bottle blank for the specific batch. Contamination in bottle blanks used for correction may cause overcorrection for some samples on some metals. However, the field samples also may be subject to this apparently random contamination. Table B-14 shows some situations where some metals, such as Cr, Fe, Mn, and Ni, are quite high in the 3050B bottle blank data compared to other bottle blank concentrations. The in vitro bottle blanks overall had very low concentrations but also showed a few high values.

Five 3050B-only reagent blanks were prepared with processing beginning at the hotplate step. Data from these samples were not used for sample correction. Results were similar to the 3050B bottle blanks. However, one sample did show very elevated concentrations of Cd, Cr, Fe, Mn, and Ni.

Three laboratory and three field blank ghost wipes were analyzed as samples (Table B-15). The two sets of wipes show similar concentrations. The 3050B extracts also

showed a few very high concentrations. The data from these blanks were not used to correct the ghost wipe sample data.

5.5.4 Measures of Precision

Analytical precision was assessed through the repeat analysis of sample extracts. The RPD for repeat analyses was less than the <20% target for the eleven metals (Table B-9).

Sampling precision also often is assessed by the collection of duplicate (collocated) samples. For the surface wipe and tire crumb material collected in this scoping study, this type of precision assessment also may assess the homogeneity of the sample media. Results for analysis of duplicate aliquots of tire infill material from a sample container or from different pieces of tire crumb collected from a playground are reported in Table B-16. For some media, there was a large RPD. Given the good analytical precision, the large RPD for duplicate sample aliquots or pieces strongly suggests that the materials, as collected and analyzed in this study, were not homogeneous with regard to the total extractable amount of some metals.

5.6 In Vitro Bioaccessibility Analysis Quality Control and Quality Assurance

A series of procedures and limits were followed to ensure the quality of the in vitro extractions and analyses. These QA/QC protocols are reviewed below, and any occurrence of nonconformance is noted and addressed. QA/QC for the extraction procedure consisted of a series of QC samples (controls, control limits, and corrective actions), as listed in Table B-17.

All bioaccessibility QC results are summarized in Tables B-18 through B-24) and meet the criteria shown in Table B-18, with the exception of the RPD values for the tire crumb duplicates (Tables B-23 and B-24) that is believed to result from sample heterogeneity issues. This extraction method was designed specifically for soils that have been processed in a manner used to create homogeneous samples. The RPD between duplicate extractions of these samples ranged from 2.7% to 124% for the infill samples and 4% to 183% for the crumb samples. Duplicate extractions of the wipes and blades were not possible either because there was a unique sample (wipes) or insufficient sample quantity (blades).

For the eight Pb spikes prepared in the in vitro extraction solutions (Table B-19), recoveries for Pb were all above 90%. The six NIST SRM 2710 extractions (Table B-20) resulted in an average RPD of 4.5% (range 0.4% to 8.9%). The RPDs for the three Ghost wipes spiked with NIST SRM 2710 (Table B-21) resulted in an average of 4.7% (range 3.5% to 6.2%). The NIST SRM RPD values are based on the mean in vitro bioaccessibility values of 75% for this SRM (EPA Method 9200.1-86).

Recoveries for blank Ghost wipes and tire crumb samples spiked with Pb solutions are listed in Table B-22. The media were extracted without spikes and used to correct for the sample's contribution to the total spiked samples. Recoveries for the four spiked infill samples for Pb ranged from 89% to 104%. Recoveries for the spiked crumb samples, ranged from 87% to 103% for Pb, whereas the recoveries for the spiked wipe samples ranged from 87% to 99%. Analysis of duplicate tire crumb infill and playground tire crumb aliquots (Tables B-23 and B-24, respectively) likely reflects the significant difference in heterogeneity of these samples.

5.7 Data Quality Assurance Review

Data generated in NERL/ORD laboratories in this scoping study were reviewed independently by a trained QA officer. The review included data generation, calculations, and transcriptions for a subset of the data. The Region 1 laboratory followed established laboratory QA/QC procedures in their analysis and review of air VOC results.

6. Results

6.1 Sampling Sites

The full study protocol (collection of all planned air, wipe, and material samples) was completed at two synthetic turf field sites (including repeated sampling on a second consecutive day at one site, F1). A reduced set of samples that did not include the integrated air particle sampling systems was completed at a third synthetic turf field site (F2). In addition, there were multiple synthetic turf fields at two sites (F2 and F3 at one site and F4, F5, and F6 at the other), and selected wipe and material samples were collected across different fields at these sites. The full study protocol, including collection of air samples, was completed at one playground (P1). However, because of the size of this playground, only two "on playground" sampling locations were operated instead of the three planned. Tire crumb material was obtained from a second playground, but no other sampling or site characterization was performed at this playground site. Information about each sampling site is provided in Table 2. The samples collected at each site are shown in Table 3.

Table 2. Sampling Site Information and Assigned Codes

Site	Туре	Assigned Code
EPA Region 5, Field 1, Day 1 ^a	Synthetic turf field	F1D1
EPA Region 5, Field 1, Day 2	Synthetic turf field	F1D2
EPA Region 4, Field 2 ^b	Synthetic turf field	F2
EPA Region 4, Field 3	Synthetic turf field	F3
EPA Region 4, Field 4 ^c	Synthetic turf field	F4
EPA Region 4, Field 5	Synthetic turf field	F5
EPA Region 4, Field 6	Synthetic turf field	F6
EPA Region 3	Playground	P1
EPA Region 4	Playground	P2

^aSamples were collected at one synthetic turf field on 2 consecutive days.

Table 3. Overview of the Types of Samples Collected at Each Site

Site	Air VOC	Air PM ₁₀ Mass	Air PM ₁₀ Metals	Air PM ₁₀ SEM	Surface Wipes	Tire Infill	Turf Blades	Tire Crumb
F1, Day 1	•	•	•	•	•	•	•	
F1, Day 2	•	•	•	•	•			
F2	•				•	•	•	
F3							•	
F4	•	•	•	•	•	•	•	
F5					•	•	•	
F6					•	•	•	
P1	•	•	•	•				•
P2								•

^bTwo synthetic turf fields (F2 and F3) were part of the complex at this site.

^cThree synthetic turf fields (F4, F5, and F6) were part of the complex at this site.

Fewer synthetic turf fields and playgrounds were monitored than originally planned, and at sites near only three of the four NERL facilities. This was primarily because of difficulties in identifying and arranging access to sites in combination with the logistical difficulties posed by the extensive array of equipment and skill required to operate the active particle sampling equipment at multiple sampling locations per site. Sampling at fewer sites had minimal impact on accomplishing the study goals, as the original design was very limited. Implementing the full protocol at only two synthetic turf field sites resulted in little impact in the methods evaluation study. Samples were collected at these two sites during relatively hot, dry days and with high activity levels (conditions favorable for the evaluation). The consecutive day measures at one site yielded additional data for assessing the methodology and understanding potential changes in site conditions from day to day. Sampling at only one playground site, although one with relatively high levels of activity, did provide insights regarding the practical issues regarding implementing the protocol at playground sites but resulted in limited data for the workgroup.

6.2 Site Characteristics

Descriptive information about each sampling site, including the age and type of material, maximum temperatures, wind speed, and activity information is provided in Table 4. Some anecdotal information may be relevant with regard to interpreting the study results. Sampling was conducted at field F1 on 2 consecutive days (F1D1 for day 1 and F1D2 for day 2). New tire crumb infill material recently had been applied to field F1. During the first day at field F1 (F1D1), heat thermals were observed coming off of the field during the hottest times of day, and there was a smell that generally is associated with tires or tire crumbs. There was considerable activity on this field throughout the day, including multiple physical education classes, as well as football and soccer practices. On the second day at field F1 (F1D2), the temperature was somewhat cooler, no thermals were observed, a similar smell was noted, and there were lower activity levels. Air PM sampling equipment was either on or immediately adjacent to field F1 during the activities. Fields F2 and F3 were adjacent fields at the same regional site. There was no activity at these fields during the monitoring period; therefore, no air PM samples were collected. Fields F4, F5, and F6 were collocated at another regional site. Air PM samples were collected on the sidelines of field F4. There was sporadic moderate activity on field F4 and on the immediately adjacent field (F5) during the monitoring day, including physical education classes, flag football, and a soccer game. One air PM sampling location was placed between fields F4 and F5. Two air PM sampling locations were placed on the opposite field F4 sideline based on wind direction at the beginning of the monitoring. The wind shifted later in the day and may have transported VOCs and particles to these two sampling locations (particularly to sampling location A) from the adjacent parking deck and nearby road with moderate commuter traffic. At playground P1, the playground was used by up to 60 preschool and early elementary students twice during the school day, and sampling continued for approximately 90 min into after-school use by approximately 12 to 20 students. The tire crumb material at playground P1, with embedded and visual fibers, was prepared and provided by a local supplier.

The air monitoring equipment setup at one sampling location at site F1 is shown in Figure 1, wipe sampling is shown in Figure 2, the turf blade and tire crumb infill at the surface is shown in Figure 3, and the multiple colors of turf blade are shown in Figure 4. A laboratory close-up of a sample vial containing tire crumb infill granules collected at site F2 is shown in Figure 5. Tire crumb material at site P1 is shown in Figure 6, and a laboratory close-up of this tire crumb material, with exposed fibers, is shown in Figure 7. A laboratory close-up of the "wood bark" tire crumb material collected at site P2 is shown in Figure 8.

Table 4. Site Information

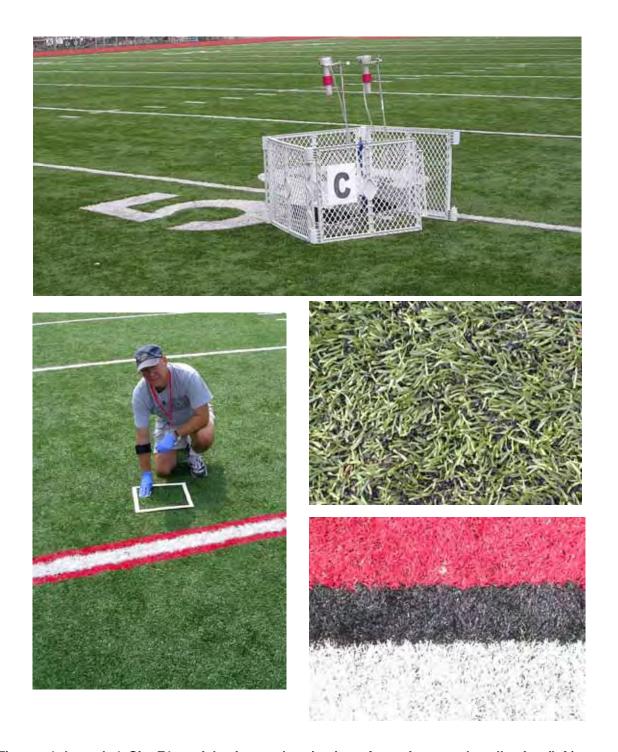
Site	Surface		Temperature at Time of Air VOC	General Activity Levels on
F1 Day 1 (F1D1)	Age 2 years ^b	Type of Surface Material Polyethylene turf blades ^c ; green field, red end zones, black and white lines; with granular tire crumb rubber infill	Sample Collection ^a 32 °C Air above field 50 °C Field surface Wind 2-11 mph	Monitoring Day Est. number: 20-70 at a time Est. duration: 45-120 min at a time
F1 Day 2 (F1D2)	2 years ^b	Polyethylene turf blades ^c ; green field, red end zones, black and white lines; with granular tire crumb rubber infill	35 °C Air above field 46 °C Field surface Wind 1-6 mph	Est. number: 20-70 at a time Est. duration: 30-45 min at a time
F2	4 years	Polyethylene turf blades ^c ; green field, red center circle, white lines; with tire crumb rubber infill	28 °C Air above field 44 °C Field surface Wind 1-11 mph	Number: 0 Duration: 0
F3	5 years	Polyethylene turf blades ^c ; green field, red center circle, white lines; with tire crumb rubber infill	d	_
F4	4 years	Polyethylene turf blades ^c ; green field; yellow and white lines; with tire crumb rubber infill	30 °C Air above field 44 °C Field surface Wind calm (2 mph)	Est. number: 10-35 at a time Est. duration: 45-120 min at a time
F5	3 years	Polyethylene turf blades ^c ; green field, yellow and white lines; patched area appeared to be green nylon turf blades ^c ; with tire crumb rubber infill	_	_
F6	2 years	Polyethylene turf blades ^c ; green field, yellow and white lines; with tire crumb rubber infill	_	_
P1	4 years	Shredded tire material; black color; much of the tire crumb had fiber material still included.	30 °C Air above playground 36 °C Playground surface Wind calm (1 mph)	Est. number: 12-60 at a time Est. duration: 30-90 min at a time
P2	Not known	Tire crumb material processed and formed to simulated bark appearance; multiple green and brown colors.	_	_

6.3 Sample Collection

The numbers and types of samples collected at each site are shown in Table 5. Air samples for VOCs were collected in evacuated 6-L Summa-polished stainless steel canisters at a 1-m sampling height. Four air VOC samples were collected at each of three synthetic turf field sites

aVOC air samples collected at hottest time of day (~2 p.m.).
bAdditional new tire infill applied during summer prior to sampling.
cType of turf blade material based on visual assessment (not confirmed through material analysis).

^dNot measured or not monitored.



Figures 1 through 4. Site F1 particle air samplers (top), surface wipe sample collection (left), green turf blade with black granular tire crumb (middle right), and multiple turf blade colors (lower right).

(F1, F3, and F4) with tire rubber infill material. Three air samples were collected at one playground site (P1) with tire crumb rubber material. Samples were collected at three different sampling locations (designated as A, B, and C, respectively) directly above each of the synthetic turf fields and at two different sites (A and B) directly above the playground. A background sample (designated as D) also was collected at each site a short distance upwind from the field









Figures 5 through 8. Tire crumb infill granules from site F2 (top left), shredded tire crumb at site P1 (top and bottom right), and tire crumb material from site P2 (bottom left).

or playground. With one exception, all air samples were collected at each site on 1 day and at approximately the same time of day. The exception was the collection of a set of four air samples on 2 consecutive days at one synthetic turf field. All grab air VOC samples were collected during the hottest time of day (~2 p.m.). In general, these grab air VOC samples were simple to collect, required little onsite collection time, and modest technical expertise. We found it was very important to verify the canister pressure prior to sampling to ensure that the evacuated canisters had not leaked prior to sample collection.

Integrated air PM₁₀ samples for mass and metals concentration measurement, as well as separate integrated air samples for SEM analysis, were collected at a 1-m sampling height at the four VOC sampling locations at synthetic turf field F1 on 2 consecutive days, and on 1 day at field F4. Air PM₁₀ samples were collected at the three VOC sampling locations on 1 day at playground P1. The limited sampling performed at playground P1 was based on the small space that did not allow the full complement of the normal sampling routine to be performed. In all events, active sampling locations were established quickly on the site, as well as from a background sampling location. Air monitoring was initiated for all monitors in quick order on their setup and calibration and continued without interruption through the monitoring event (daytime). This resulted in sample collections ranging from approximately 5.8 to 7.8 h and corresponding individual air volumes ranging from approximately 7.0 to 9.2 m³ over the course of a sampling day. Collection of up to 10 air particle samples at each site required considerable equipment (enough to fill a van), considerable setup and retrieval time (approximately 1 h each), extensive staff time onsite (typically 8 to 10 h for two people), and moderate technical monitoring expertise. The monitoring approach, equipment, and sampling durations were selected to obtain

Table 5. Number of Samples Collected at Each Site

Site ^a	Onsite Samples	Background Samples	Duplicate Samples	Archival Samples	Field Blanks	Field Controls
Air VOC						
F1D1	3	1	1	_	1	1
F1D2	3	1	_	_	_	_
F2	3	1	_	_	_	_
F4	3	1	1	_	1	1
P1	2	1	1	_	1	1
Air PM ₁₀ for M	lass and Metals	3				
F1D1	3	1	1	_	1	_
F1D2	3	1	1	_	1	_
F4	3	1	1	_	1	_
P1	2	1	1	_	1	_
Air PM ₁₀ for S	SEM					
F1D1	3	1	1	_	1	_
F1D2	3	1	1	_	1	_
F4	3	1	1	_	1	_
P1	2	1	1	_	1	_
Surface Wipe	s					
F1D1	3	_	3	3	1	_
F1D2	3	_	3	3	_	_
F2	3	_	3	_	1	_
F4	1	_	1	1	1	_
F5	1	_	_	1	_	_
F6	1	_	_	1	_	_
Tire Infill						
F1D1	3	_	_	_	_	_
F2	3	_	3	_	_	_
F4	1	_	1	_	_	_
F5	1	_	_	_	_	_
F6	1	_	_	_	_	_
Turf Blades						
F1D1	3	_		_	_	_
F1D2	1	_	_	_	_	_
F2	3	_	_	_	_	_
F3	4	_	_	_	_	_
F4	1	_	_	_	_	_
F5	1		_			
F6	1		_			_
Tire Crumb						
P1	2	_	1	2	_	
P2	1	_	_	_	_	_
	rf field D = day 1	or day 2, P = play	around			

 $^{^{}a}F$ = synthetic turf field, D = day 1 or day 2, P = playground.

adequate limits of detection for PM_{10} mass and a range of metal analytes to ensure that levels typically found in ambient air would be measurable.

Surface wipe samples were collected at three "on field" sampling locations at synthetic turf fields F1 (on 2 consecutive days) and F2. At a third site, a single surface wipe sample was

collected from each of three fields (F4, F5, and F6), as the three fields making up this complex were of different ages. A standard wet-wipe method (ASTM E1728-03) that is used to measure residential surface dust Pb levels was employed for sample collection in this study. In the absence of a validated wipe method for synthetic turf surfaces, this method was selected for evaluation in this study. In general, the advantages of this method were the availability of standard wipe material and existing analytical methodology. In practice, samples could be collected by those with moderate technical expertise at multiple sampling locations in a relatively short period of time. However, the sampling procedure was not as simple as for smooth floor materials, with the synthetic turf blades requiring additional patience and control. The relationship between the dust collected on the wipe sample that comes from turf blades versus the dust from the infill and is available for human contact is not clear and may need further investigation. Most wipes also picked up a few pieces of the rubber infill material and turf blades. A decision was made to remove these relatively large discrete materials in the laboratory prior to extraction as these larger materials would be characterized as part of the additional material samples. It is not expected that removal of these large materials would impact the measurement of the small dust particles that the surface wipe sample is designed to collect.

Tire crumb rubber infill used in these synthetic turf field installations was collected from three sampling locations at fields F1 and F2 and at single sampling locations from each of three fields (F4, F5, and F6) at the third site. Sample collection could be completed in a short time by persons with minimal technical expertise. In this study, it was decided to collect infill material that was already available at the surface rather than by dislodging material trapped deep within the turf blades. This decision was based partly on avoiding potential damage to field components but primarily because the material on the surface was more available for potential human contact. However, infill material was not available uniformly across the field surface.

Synthetic turf blade samples were collected at all field sites in this study. The synthetic turf blades were not a primary interest in this study, as the characterization of this type of material is being performed by other organizations. However, samples of turf blades were collected to enable an improved understanding of the surface wipe measurements with regard to the potential differential contributions from the infill and blades. A decision was made not to perform destructive collection (i.e., there was no cutting of material from the fields). Instead, the collection relied on the availability of loose blades found at the surface of the fields. Collection and analysis decisions were complicated by the limited availability of loose blades and a later decision that a minimum of 0.7 g of material was required for analysis. Collection of blades of each color type was attempted. For fields F1, F2, and F3, the colors were collected separately and kept separate for analysis. None of the green blade samples from the site complex comprised of fields F2 and F3 achieved the "postsampling" requisite 0.7-g sample size, and they, therefore, were not analyzed. In retrospect, a decision to combine green blades from several different sampling locations would have enabled the analysis of a composite site sample. For fields F4, F5, and F6 the different colors for each field were mixed together in the sample to best achieve adequate sample sizes, while, at the same time, obtaining samples from fields of different ages.

Tire crumb rubber samples were obtained from two playground sites. The material used at the P1 playground consisted of shredded tire particles containing fibers, whereas the material from playground P2 was processed and colored to simulate tree bark. Collection of tire crumb material is a simple process. However, the material is relatively heterogeneous and it is not clear how many pieces or which pieces need to be collected for site characterization. It is also not clear whether it is most appropriate to collect tire crumb pieces at the surface that may have experienced different weathering and contact than underlying pieces. A further challenge is that relatively small amounts (1 g or less) are required for analysis because larger amounts may overwhelm digestion and analytical systems. Intact tire crumb rubber pieces are generally larger

than 1 g. A decision was made not to cut the tire crumb pieces because exposing unweathered surfaces might impact the Pb bioaccessibility measurement.

6.4 Summary Measurement Results

Tables 6 through 8 provide key data summaries for the synthetic turf field and playground sites. All of the individual site sample data are provided in Appendix C.

6.5 Air VOC Measurement Results (Appendix C, Table C-1)

Key highlights from the grab air VOC measurements are summarized below.

- Twelve of the 56 target VOC analytes were present in multiple samples. Thirty-seven of the 56 analytes were not detected in any sample. Seven of the remaining 19 analytes had detectable levels in only one or two samples.
- Measured VOC concentrations were generally low. No analyte concentration exceeded 1 ppbV in any sample. Detection limits ranged from 0.070 to 0.079 ppbV for most analytes, and 0.14 to 0.16 ppbV for 1,3-butadiene and m&p-xylenes.
- MIBK previously has been identified as a tire-related VOC. MIBK was present in three "on field" samples collected at one synthetic turf field (F1) on the first monitoring day when heat thermals were observed and in two "on field" samples on the second consecutive day of monitoring. MIBK concentrations were low (<0.2 ppbV) in all five samples. MIBK was not detected in the background samples collected near the field on either day. MIBK was not detected at any other site in this study.
- Concentrations of the other VOCs routinely measured over the field or playground sampling locations were similar to the concentrations measured in corresponding upwind background samples collected nearby, or they likely could be explained by documented local sources near the site.
- MEK was measured in all the study samples, with the "on field" MEK levels being similar to levels in the upwind background samples at each site.
- Hexane was present in most of the "on field" and background samples. At the F4 synthetic turf field, the hexane concentrations at all three "on field" sites were higher than the background site, but all concentrations were low (<0.2 ppbV).
- The aromatic analytes benzene, toluene, and m&p-xylenes are ubiquitous atmospheric pollutants and were present at measureable levels in most samples collected in this study.
- Benzene concentrations were similar for the background and "on field" samples at all sites.
 The highest benzene concentration (0.32 ppbV) was measured at location A on site F4. This
 concentration was higher than at sites B or C or the background site D. Other aromatic VOC
 concentrations also were elevated somewhat at this sampling location and site. Sampling
 location A was closest to a parking garage exit and may have been impacted by traffic and
 vehicle exhaust.
- Toluene, m&p-xylenes, and hexane concentrations at site P1 location B, and at site F1 location B, were higher than concentrations at the other sampling locations for these sites. The reason for this is not clear; however, elevated levels (>4 ppbV) of these compounds were measured in one field blank, and contamination of the sampling canisters cannot be ruled out. Both of these samples had a corresponding duplicate sample collected at location B. Concentrations of these three analytes were present in the duplicate samples at ratios ranging from 0.15 to 0.63 of the concentrations in the samples. In fact, the concentrations measured in the duplicate samples were similar to the concentrations measured in the other samples at each site, further suggesting that these two samples with slightly higher concentrations may have been contaminated.
- Several halogenated VOCs were measurable in all or most of the samples. These included carbon tetrachloride, methylchloride, methylene chloride, dichlorodifluoromethane (Freon 12),

Table 6. Summary Results for Selected Analytes in Air Samples Collected at Synthetic Turf Fields and a Playground

						Synthetic	Turf Field	s			Playg	round
			F1	D1	F1	D2	F	2	ı	F4	F	21
			On	Back-								
Air Samples	Analyte	Unit	Field ^a	grnd. ^b	Play. ^a	grnd. ^b						
Air PM Mass	Particle Mass	μg/m ³	27.8	29.5	29.8	29.5	_	_	31.8	28.6	26.7	14.2
Air PM Metals	Pb	ng/m³	ND°	ND	7.7 ^d	6.3	_		ND	ND	5.1 ^d	ND
	Cr	ng/m ³	2.9	2.0	3.6	3.3	_	_	ND	ND	3.4	ND
	Zn	ng/m ³	10.8	23.8	11.8	11.6	_	_	31.4	21.7	104	10.5
Air VOCs	Methyl Isobutyl Ketone	ppbV	0.13	ND	0.12	ND						
	Benzene	ppbV	0.09	0.07	0.08	0.09	0.11	0.12	0.20	0.12	0.09	0.09
	Toluene	ppbV	0.42	0.15	0.11	0.12	0.18	0.19	0.28	0.19	0.29	0.16
	m&p-Xylenes	ppbV	0.17	0.08	0.10	ND	0.07	0.08	0.14	ND	0.13	0.05
	Hexane	ppbV	0.21	0.06	0.08	0.08	0.08	0.05	0.14	0.05	0.30	ND
	Methyl Ethyl Ketone	ppbV	0.47	0.44	0.38	0.36	0.41	0.37	0.43	0.44	0.41	0.38
	Carbon Tetrachloride	ppbV	0.09	0.10	0.10	0.10	0.09	0.08	0.09	0.10	0.08	0.09
	Dichlorodifluoromethane	ppbV	0.52	0.55	0.50	0.56	0.56	0.51	0.48	0.54	0.53	0.54
	Methylchloride	ppbV	0.47	0.48	0.47	0.46	0.45	0.45	0.48	0.52	0.46	0.45
	Trichloro-fluoromethane	ppbV	0.26	0.28	0.26	0.27	0.27	0.25	0.24	0.30	0.26	0.26
	Trichloro-trifluoroethane	ppbV	0.08	0.08	0.08	0.08	0.08	0.07	0.07	0.15	0.07	0.07
1	Methylene Chloride	ppbV	0.07	0.06	ND	ND	0.06	0.06	0.06	0.06	0.09	0.07

^aAverage of two or three "on field" or "on playground" measurements (any nondetect values were not included in the average). ^bSingle measurement from upwind background location. ^cNot detected.

^dPb was measured in only one of three "on field" samples at F1D2 and one of two "on playground" samples at P1.

Table 7. Summary Results for Total Extractable Pb, Cr, and Zn in Samples Collected at Synthetic Turf Fields and

Playgrounds

				Playgi	rounds			
			F1D1	F1D2	F2, F3	F4, F5, F6	P1	P2
Sample	Analyte	Unit	Range	Range	Range	Range	Range	Range
Turf Field Surface Wipe Samples	Pb	µg/ft ²	0.3-1.9	0.4-1.4	0.3-0.5	0.1-0.2	NC ^a	NC
	Cr	μg/ft ²	0.1-0.5	0.1-0.3	ND.b	ND-0.3	NC	NC
	Zn	μg/ft ²	21.3-43.3	26.4-40.6	9.2-19.3	4.3-13.6	NC	NC
Turf Field Infill Crumb Rubber	Pb	μg/g	13.1-34.7	NC	20.6-61.2	10.7-47.7	NC	NC
	Cr	μg/g	0.3-1.0	NC	0.4-0.9	0.3-1.0	NC	NC
	Zn	μg/g	5,050-19,200	NC	3,120-12,300	2,660-11,400	NC	NC
Turf Field Blades	Pb	μg/g	2.8-389 ^c	NC	2.4-2.8 ^d	2.1-701 ^e	NC	NC
	Cr	μg/g	1.0-73.1	NC	1.2-1.9	3.7-177	NC	NC
	Zn	μg/g	316-730	NC	199-255	131-206	NC	NC
Playground Tire Crumb	Pb	μg/g	NC	NC	NC	NC	1.0-443 ^f	3.4-7.8 ⁹
	Cr	μg/g	NC	NC	NC	NC	0.3-1.7	1.6-3.0
	Zn	µg/g	NC	NC	NC	NC	4,300-17,500	12,100-18,000

^aNot collected.

^bNot detected.

[°]Differences noted for different blade colors (red = 389 μ g/g; white, green, and black all <4.3 μ g/g).

^dAnalysis of red and white blades.

^eHighest level (701 μg/g) found in a field with a repaired area; levels in blades from two adjacent fields ranged from 2.0 to 77 μg/g. ^fAnalysis of seven pieces of tire crumb; six of those pieces had Pb levels <50 μg/g.

^gAnalysis of two pieces of tire crumb.

Table 8. Summary Results for Estimates of Pb Bioaccessibility in Samples Collected at Synthetic Turf Fields and

Playgrounds^a

			Syntheti	c Turf Fields		Playgr	ounds
		F1D1	F1D2	F1D2 F2, F3		P1	P2
Sample	Analyte	Range	Range	Range	Range	Range	Range
Turf Field Infill Crumb Rubber	Pb	1.6%-9.6%	NC ^b	1.7%-7.6%	1.7%-10.1%	NC	NC
Turf Field Blades	Pb	2.3%-86.8% ^c	NC	38.7%-40.3% ^d	0.2%-54.4% ^e	NC	NC
Playground Tire Crumb	Pb	NC	NC	NC	NC	0.3%-10.7% ^f	1.8%-7.4%

^aThe in vitro bioaccessibility method has not been validated for these materials.

^bNot collected.

^cDifferences noted for different blade colors (red = 2.3%, white and green = 40.9% to 43.0%, and black = 86.8%); also, the lowest bioaccessibility value (2.3%) corresponds to the sample with the highest total extractable Pb (389 µg/g).

^dAnalysis of red and white blades.

^eLowest bioaccessibility value (0.2%) corresponds to the sample with the highest total extractable Pb (701 μg/g).

Lowest bioaccessibility value (0.3%) corresponds to the sample with the highest total extractable Pb (443 µg/g)

trichlorofluoromethane (Freon 11), and trichlorotrifluoroethane (Freon 113). In all cases, the levels in the "on field" samples were similar to levels in the upwind background sample at each site.

6.6 Air PM₁₀ Mass Measurement Results (Appendix C, Table C-2)

 PM_{10} concentrations observed across all of the sites ranged from approximately 24 to 33 $\mu g/m^3$. PM_{10} mass concentrations collected on or adjacent to synthetic turf fields were generally equivalent to concentrations in ambient air measured at the upwind background sites. Mass concentrations across a given synthetic turf field or playground were often consistent within themselves. That is, PM_{10} concentrations from field sampling locations A, B, and C at a given site often varied by only 2 to 3 $\mu g/m^3$, which was within the precision error typically observed for the duplicates. This mass consistency was generally true regardless of the range of activities taking place on the field and the proximity of such activities to a given monitor. Such a statement, however, cannot be made for the one playground site monitored. Comparison of data from the P1 site indicates an approximately 15 $\mu g/m^3$ higher PM_{10} mass concentration was obtained from the monitor located near the highest density of playground activity (location B).

6.7 Air PM₁₀ Metal Measurement Results (Appendix C, Table C-3)

Air PM₁₀ sample filters were analyzed for 44 metals. As part of the analysis, the statistical uncertainty of the measurement was determined; the measured metal concentrations must be at least three times the uncertainty concentration to be considered a measured result. Based on this assessment, the full list of 44 metals was reduced to 12 that are reported in Appendix C-3, including the primary analytes Pb, Cr, and Zn (see Table 6), as well as 8 other elements (Ca [calcium], Cl [chlorine], Cu, Fe, K [potassium], Mn, S, Si [silicon], and Ti [titanium]) with sufficient number of measurable results for assessment within and across sampling sites.

Measurement results from synthetic turf field F1 show that upwind background levels of Si, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Cu, Zn, and Pb were similar to the "on field" concentration measurements at thee sampling locations (A, B, and C). This would indicate that the infill tire crumb rubber and other materials on the turf field examined in F1 (on both day 1 and day 2) provided little contribution to the measured metal concentrations associated with airborne aerosols. Slightly higher concentrations of Ti (~36%), Cr (~90%), Mn (~100%), Fe (~74%), Cu, (~57%), and Zn (~65%) were measured at the "on field" monitors relative to the upwind background sampling location at the F4 synthetic turf field. This site was bordered by both a busy urban commuter road, as well as a parking deck. Because of this, the additional contribution of some of the metals to the collected "on field" air samples, notably those that often are observed in near-roadway air samples (Fe, Zn, and Cu) might not be singularly reflective of contributions from a single source (i.e., this might be indicative of near-road influence, as well as of any contribution from any resuspended tire crumb rubber infill aerosol).

It would appear that the "on playground" samples associated with the P1 site had consistently higher levels of the 12 selected metals discussed above, as compared with the background site. For example, "on playground" aerosol metal concentrations for Si, Cl, K, and Ca were sometimes 50% to 700% higher, as compared with the background. These metals often are associated with crustal (soil) related sources. "On playground" samples had much higher levels of metals that might be of relevance to tire crumb rubber components. For example, Ti concentrations were more than four times higher for one "on playground" sampling location, with nonspeciated Cr concentrations (~3 ng/m³) substantially higher than the background monitor.

Mn concentrations were ~7 times higher (15 ng/m³), with Fe as much as 4 times more concentrated (~1,000 ng/m³). Of particular interest are Zn levels, which were 8 to 11 times higher than background levels for the two "on playground" monitors (82 to 117 ng/m³). On the

other hand, these metals also may be found in soils, so the relative contribution of tire crumb particles to the increased levels could not be determined readily from these results.

Pb concentrations were at measurable levels in only 3 of the 18 air PM samples collected in this study. These three samples had concentrations (≤7.7 ng/m³) that were near the method detection limit. The maximum concentration of 7.7 ng/m³ was measured in a sample collected at a synthetic turf field and, given the precision of the method, is considered indistinguishable from the corresponding background level (6.3 ng/m³). A Pb level of 5.1 ng/m³ was measured at one "on playground" sampling location; again, a level near the method detection limit. The samples for another "on playground" sample, its duplicate sample, and the background sampling location had Pb values below the method detection limit.

6.8 Air PM₁₀ SEM Measurement Results (Appendix C, Table C-4)

Air PM_{10} samples were collected on filters at three sites with tire crumb material for SEM analysis. Air samples collected at two "on field" or "on playground" sampling locations and at one upwind background sampling location were analyzed for synthetic turf fields F1 and F4 and playground P1. In preparation for analyzing the air samples, samples of the tire infill material from a field and the crumb material from a playground were analyzed to determine whether metal or morphological "source profiles" could be identified that would assist in identifying tire crumb-related particles in the air samples. A detailed report describing the SEM analysis procedure and measurement results is provided in Appendix D. Key findings from the SEM analysis are described below.

- Prior to analyzing the air filter samples, particles were generated from the tire crumb materials collected at the turf fields and playground to try to identify a signature morphology and metals composition for tire crumb particles. These particles did not show a unique, easily identifiable X-ray spectrum for metals composition or supporting a definitive source attribution analysis. C and S were consistently present in the tire crumb particles. Zn usually but not always was observed in tire crumb particles, often at trace levels. Tire crumb particles from the source material varied considerably in morphology, making it difficult to identify a typical or characteristic tire crumb morphology.
- Very few fibers were observed in any of these air samples, and none could be attributed to tire crumb. This was true even for the playground site (P1) air samples, which had tire crumbs with exposed and embedded fibrous material.
- The ability to quantify the tire crumb concentration in these samples hinges on the tire crumb
 particles having a unique composition or morphology that would enable the analyst to identify
 tire crumb particles with a high degree of confidence. This does not seem to be the case for
 tire crumb particles collected in this study, as seen in the variety of morphologies and
 compositions on air filters.
- At the two synthetic turf fields, mass concentrations for postulated tire crumb particles were
 estimated to be only a very small fraction of the total PM₁₀ mass concentrations measured at
 these sites. At the playground site, estimated mass concentrations for postulated tire crumb
 particles were a relatively small fraction of the total PM₁₀ mass concentrations, but a higher
 fraction than was measured at the synthetic turf fields. However, the variability in tire crumb
 particle composition and morphologies introduces large uncertainties in these results.

6.9 Total Extractable Metals in Synthetic Turf Field Surface Wipe, Tire Crumb Infill, and Turf Blade Samples and Playground Tire Crumb Rubber Samples (Appendix C, Tables C-5 through C-8)

The total extractable measurement results for 11 metals in wipe, tire crumb, and synthetic turf blades collected in the scoping study are shown in Appendix C. Primary target metals were Pb, Cr, and Zn (see Table C-5), and metals of secondary interest included Al, As,

Ba, Cd, Cu, Fe, Mn, and Ni. Results from surface wipe samples collected at synthetic turf fields are shown in Table C-5, those for tire crumb infill from synthetic turf fields are shown in Table C-6, and those for synthetic field turf blades are shown in Table C-7. Results for tire crumb material at playgrounds are shown in Table C-8.

6.9.1 Surface Wipes from Synthetic Turf Fields (Table C-5)

Surface wipe samples were collected at different sampling locations from several synthetic turf fields. The sampling locations were in close proximity to the air monitoring "on field" locations. In some cases, duplicate samples were collected side-by-side. Some wipe samples were collected from different color turf blade areas at one site (F1) where there were large areas of different color turf blades. A wipe sample also was collected at the field (F5) with visually different turf materials. Total extractable metal measurement result highlights include those that follow.

- Total extractable Pb was less than 2.0 μ g/ft² in all surface wipe samples collected at the synthetic turf fields in this study. Most results were less than 1.0 μ g/ft².
- Many of the sample analysis results were similar to levels measured in field blanks, which had Pb values ranging from 0.14 to 0.54 μ g/ft².
- The highest total extractable Pb value (1.9 μg/ft²) was measured on a surface wipe collected at site F1 on red synthetic turf blades.
- Surface wipe samples collected side-by-side, in some cases, had similar total extractable Pb levels; in other cases, the differences in side-by-side measurements ranged up to approximately twofold. Some of the side-by-side measures taken at one turf field site (F1) were from an area of mixed turf blade colors.
- Surface wipe samples collected at different locations on synthetic turf fields had up to sixfold differences in total extractable Pb concentrations. The greatest differences appeared to be associated with wipes taken from areas with different blade colors.
- Total extractable Cr was <0.6 µg/ft² in all surface wipe samples.
- Total extractable Zn in surface wipe samples ranged from 4.0 to 43 μg/ft².
- Measurements of As were very low (≤0.1 μg/ft²) and similar to amounts found on field blanks.
- Most Cd measurements were less than the method detection limit; the remainder were ≤0.025 μg/ft².

6.9.2 Tire Crumb Infill at Synthetic Turf Fields (Table C-6)

Samples of tire crumb infill granules were collected at different sampling locations from several synthetic turf fields. The sampling locations did not necessarily correspond to the sampling locations where the other samples were collected. In some cases, duplicate samples were collected side-by-side. In addition, second aliquots of material collected in each sample container were analyzed, so that there were two analysis results for each sample or duplicate sample.

It is important to remember that the methods for collection and analysis have not been validated. Total extractable metal measurement result highlights include the following.

- Total extractable Pb concentrations in tire crumb infill from synthetic turf fields ranged from 11 to 61 μg/g.
- There was considerable variability (up to an approximately fourfold difference) in total extractable Pb concentrations for different aliquots randomly taken from the same sample container.
- The variability among locations at a site and among different sites was similar to the variability in Pb measurement results for aliquots taken from the same container (up to approximately fourfold).
- Total extractable Cr concentrations ranged from not detected to 1.0 µg/g.

- Total extractable Zn concentrations ranged from 2,600 to 19,000 μg/g.
- The variability in Zn concentrations for aliquots taken from the same container was less than the variability observed in Pb concentrations.
- Concentrations of As ranged from not detected to 0.55 µg/g.
- Cd concentrations ranged from not detected to approximately 1.5 μg/g.

6.9.3 Turf Blades at Synthetic Turf Fields (Table C-7)

Characterization of the turf blade material was not a primary goal of this scoping study; the focus of this study was on the tire crumb components. However, these samples were collected and analyzed to help improve interpretation of the surface wipe measurements. It is important to remember that the methods for collection and analysis have not been validated for this material. Samples of synthetic turf blades were collected at different sampling locations from several synthetic turf fields. Where possible, samples of different colors were collected but, because of the small sample sizes, could not always be analyzed separately. The sampling locations did not necessarily correspond to the locations where the other samples were collected. Total extractable metal measurement result highlights are as follows.

- Total extractable Pb concentrations from synthetic turf blades ranged from 2.4 to 700 μg/g.
- The highest total extractable Pb concentration (700 μg/g) was measured from blades in a sample collected at a turf field (F5), which included an area that had apparently been repaired with a section of turf material visually different from the rest of the field. Mixed-color turf blade samples taken from two adjacent fields (F4 and F6) at the same site had Pb levels ranging from 2.0 to 77 μg/g.
- The second highest total extractable Pb concentration (389 μg/g) was measured in red blades at another turf field site (F1). Pb concentrations for green, white, and black blades collected from this same site ranged from 2.8 to 4.3 μg/g.
- Total extractable Cr concentrations ranged from 0.1 to 180 μg/g. The level of Cr generally appeared to be lower than but correlated with the corresponding Pb concentrations.
- Total extractable Zn concentrations ranged from 130 to 730 µg/g. Zn levels in white and black blades collected at one site (F1) were about twofold higher than in green and red blades at the same site and about three to five times higher than levels measured in blades from the other two synthetic turf field sites.
- Concentrations of As and Cd were less than 0.6 μg/g in all samples.

6.9.4 Tire Crumb Material from Playgrounds (Table C-8)

Samples of tire crumb pieces were collected at different sampling locations at two playgrounds. Seven pieces of crumb rubber from one playground (P1; shredded tires with exposed fibers) and two pieces from the second playground (P2; with simulated bark tire crumb material) were analyzed. It is important to remember that the methods for collection and analysis have not been assessed or validated. Total extractable metal measurement result highlights include those described below.

- Total extractable Pb concentrations in five pieces of shredded tire crumb from P1, the
 playground with fibrous materials, ranged from 1.0 to 6.3 μg/g. A Pb concentration of 46 μg/g
 was measured in a sixth piece, and 440 μg/g Pb was measured in a seventh piece,
 documenting the heterogeneity of Pb in these site samples.
- Total extractable Pb concentrations from two simulated bark tire crumb samples collected at a second playground (P2) were 3.4 and 7.8 µg/g.
- Total extractable Cr concentrations ranged from 0.3 to 3.0 μg/g.
- Total extractable Zn concentrations ranged from 4,300 to 18,000 μg/g.
- The variability in Zn concentrations was less than the variability observed in Pb concentrations.

- Concentrations of As ranged from 0.04 to 0.96 μg/g, except for a value of 15 μg/g that was measured in the same P1 crumb piece with the highest Pb level (440 μg/g Pb).
- Cd concentrations ranged from 0.09 to 10.5 μg/g, with the highest concentrations corresponding to samples with the highest Pb concentrations.

6.9.5 Lessons Learned with Regard to the Sampling and Analysis of Tire Crumb Materials

Again, there are no validated sample collection and analytical procedures for characterizing tire crumb materials for assessing potential environmental concentrations or potential exposures by various routes and pathways at synthetic turf fields and playgrounds. An objective of this scoping study was to apply an existing wipe sample collection method and analytical procedures developed for soil media and to assess their performance. A few of the lessons learned are described below.

- There was no evaluated protocol available for measuring tire crumb rubber constituents at turf fields and playgrounds. As such, this scoping study was conducted to evaluate the methods and identify key factors (e.g., resources, accessibility, practicality, activity levels) that would need to be considered in designing future studies. Some factors that may need to be considered for future research include the number and placement of sampling locations (i.e., how many samples at how many locations need to be collected and analyzed to adequately characterize a site), representative and duplicate wipe samples for turf fields with mixed colors, retaining or not retaining turf infill and fibers on wipe samples, representative material samples, the relationship between the wipe sample and material sample results.
- In some cases, the amount of material available for analysis was not optimal, generally a 1-g sample is specified in methods. For tire crumb material from playgrounds, most of the crumb pieces were much larger than 1 g. It was decided that the material would not be cut because that would open fresh surfaces that potentially would result in different extractable amounts. Decisions not to cut up samples prevented sample size matching to extraction procedure requirements and prevented homogenization procedures. In addition, the larger samples created some extraction and analysis problems with regard to the extraction vessel and need for multiple dilutions of some sample extracts. On the other hand, some samples of synthetic turf blades collected in this study were not adequate for analysis. Only nondestructive collection of loose blades was performed in this study. In future work, sample sizes and decisions regarding tire crumb subsampling should be considered.
- Information from this and other studies regarding the metals of most interest would improve analytical optimization, reporting, and the selection of appropriate QC materials.
- The heterogeneity of these samples create analysis and data interpretation challenges. For
 example, multiple dilutions and reanalyses of many samples were required to obtain
 measurements in the instrument calibration range. Based on excellent results from analytical
 QC analyses (serial dilution and postdigestion spikes), the new ICP/MS instrument used for
 this study appeared to produce quantitative total extractable results for the wipe samples, turf
 blades, tire infill, and tire crumb media for multiple metals with low detection limits.
- Spiking levels appropriate to the tire material and blade material concentrations need to be
 considered in future study designs to improve results. However, given the heterogeneity of
 the materials, it is not clear whether spiking samples to assess recoveries will be feasible.
 Spike recoveries need to be reevaluated with truly homogeneous samples to determine
 whether the extraction procedure or the sample heterogeneity was the source of variable
 recoveries during the scoping study.
- Sporadic contamination of laboratory bottle blanks was found, especially for metals that might be associated with steel. In this study, the blank result for each analysis batch was subtracted from measured results, following the standard procedure. In the future, blank correction could be based on the average of the bottle blanks, with the option of removing outliers.

Investigation is needed of possible sources of contamination and, perhaps, changing to a less "open" extraction process.

6.10 Pb Bioaccessibility Results (Appendix C, Tables C-9, C-10, and C-11)

6.10.1 Analysis of Turf Field Wipe, Tire Crumb, and Turf Blade Samples

The bioaccessible values for Pb in the field samples are provided in Appendix C. Because of the variability observed for different sample aliquots, duplicate samples, and analyses of multiple sample pieces, we have reported all analyses individually rather than averaging the results across the two measurements for each sample. It is important to recognize that different bioaccessibility procedures may yield different Pb bioaccessibility results. The EPA method employed in this study has been validated for Pb in soil, but no methods have been validated for tire crumb or synthetic turf blade materials. Highlights from the bioaccessability analyses include those below.

- Synthetic turf field tire crumb infill Pb bioaccessibility ranged from 1.6% to 10.7% (mean 4.7%; Table C-9).
- Synthetic turf field blade Pb bioaccessibility (Table C-10) ranged from 0.2% to 86.8% (mean 34.2%). The three samples with the highest total extractable Pb (77 to 700 µg/g) had the lowest Pb bioaccessibility values (0.2% to 2.3%). Gaining a clear understanding of this observation requires additional research.
- Playground tire crumb Pb bioaccessibility (Table C-11) ranged from 0.3% to 7.4% (mean 4.3%). The two samples with the highest total extractable Pb (46 and 440 μg/g) had the lowest Pb bioaccessibility values (both 0.3%). Gaining a clear understanding of this observation requires additional research.
- Up to a fourfold difference in Pb bioaccessibility was found between two aliquots of tire crumb infill analyzed from the same sample vial. Up to a 36-fold difference was found between the analyses of seven pieces of tire crumb material from the same playground. These results suggest substantial heterogeneity in Pb bioaccessibility from tire crumb rubber samples. Gaining a clear understanding of this observation requires additional research.
- The in vitro Pb bioaccessibility method was judged to be inappropriate for the surface wipe samples. The blank media bioaccessible Pb values were similar to the values observed in the field samples. Given the relatively small amount of dust collected on the wipe, as compared with the large mass of wipe material, and the relatively low amounts of Pb measured, it is likely that any calculated bioaccessibility attributed to the dust likely is to be impacted significantly by the background levels in the sampling and analysis procedures. Gaining a clear understanding of this observation requires additional research.

6.10.2 Lessons Learned with Regard to Bioaccessibility Data

An objective of this scoping study was to apply existing wipe collection and material analytical procedures developed for soil media and to assess their performance. A few of the lessons learned are described below.

- Sufficient quantities of samples are needed to meet extraction requirements for EPA SOP 9200.1-86; this should be considered in sampling designs.
- Limitations on cutting samples prevented further homogenization of samples.
- Better method detection limits (MDLs) for the in vitro extractions were achieved relative to the EPA SOP 9200.1-86 requirements and values previously published.
- Additional methods development and validation research is recommended before this wipe method is applied in future studies.

6.11 Methods Evaluation SummaryA summary evaluation of the sample collection and analysis methods for the several types of samples collected in this scoping study is provided in Table 9.

Sample Type	Sample Collection	nent of Methods Applied in Sample Analysis	Other Comments
Air VOCs	 Grab samples simple to collect. Short time needed. Can be collected directly over field or playground. 	 Standard Method TO-15 (GC/MS) provided data for 56 analytes with good precision and accuracy. Sensitivity was sufficient to measure analytes in ambient air. 	Overall, this method was simple to implement and provided adequate data.
Air PM ₁₀ (mass, metals, SEM)	 Large amount of equipment needed. Technical expertise required. Required 8-10 h of time to collect. Could not always be placed directly on field because of activity. 	 Standard research methods provided data for mass concentration and for multiple metals. Sensitivity sufficient to measure analytes in ambient air. Precision and accuracy were good. 	 Overall, this method was somewhat complex and time consuming to implement and provided adequate data. Identification of tire crumb particles was difficult because of lack of standard morphology or composition.
Surface Wipes (turf fields)	 Moderately simple to collect. Short time needed. Method not validated for synthetic turf surfaces. Wipes can collect infill particles and turf blades (that were removed for these analyses). 	 Standard EPA Methods 3050B and 6020 applied with good recovery and analytical precision. Sensitivity was very good and sufficient to measure low levels of multiple metals. EPA Pb in vitro bioaccessibility method 9200.1-86 found not to be appropriate for wipe samples. 	 Overall, this method was relatively simple to implement. The method provided quantitative measurement results, but the method has not been validated for use on synthetic turf field surfaces. The in vitro Pb bioaccessibility method was judged to be inappropriate for the surface wipe samples.
Tire Crumb Infill (turf fields)	 Simple to collect. Short time needed. Decisions needed on area and depth of material collection. 	 Standard EPA Methods 3050B and 6020 applied with good analytical precision. EPA Pb in vitro bioaccessibility method 9200.1-86 applied with good analytical precision. Nonhomogeneous material made assessment of analyte recoveries difficult. Sensitivity was very good and sufficient to measure low levels of multiple metals. 	 Overall, this method was relatively simple to implement. The method provided quantitative measurement results, but the method has not been validated for use on tire crumb particles. Improved quality control methods and materials required to assess metal recoveries. Nonhomogeneity of Pb has implications for site sampling, analysis, and data interpretation.

Table 9. Overall Summary and Assessment of Methods Applied in This Scoping Study (cont'd.)

(cont'd.)		T	
Sample Type	Sample Collection	Sample Analysis	Other Comments
Blades (turf fields)	 Simple to collect. Relatively short time needed. Limited in this study to collecting loose blades where available. Larger sample size needed for analysis (>1 g each) than were collected for some samples in this study. Collection of different colors revealed different Pb levels at some fields; this should be considered in site sampling plans. 	 Standard EPA Methods 3050B and 6020 applied with good analytical precision. EPA Pb in vitro bioaccessibility method 9200.1-86 applied with good analytical precision. Nonhomogeneous material made assessment of analyte recoveries difficult. Sensitivity was very good and sufficient to measure low levels of multiple metals of interest. 	 Overall, this method was relatively simple to implement. The method provided quantitative measurement results, but the method has not been validated for use on synthetic turf blades. Improved quality control methods and materials required to assess metal recoveries. Differences for Pb depending on blade color have implications for site sampling and analysis.
Tire Crumb Rubber (playgrounds)	 Simple to collect. Short time needed. Decisions required on site sampling plan with regard to location and number of areas and depth of material for collection. 	 Standard EPA Methods 3050B and 6020 applied with good analytical precision. EPA Pb in vitro bioaccessibility method 9200.1-86 applied with good analytical precision. Sensitivity was very good and sufficient to measure low levels of multiple metals of interest. Nonhomogeneous material made assessment of analyte recoveries difficult. Tire crumb pieces were larger than the 1 g desired for analysis; homogenization and subsampling may be needed in future work. 	 Overall, sample collection was simple to implement, but analysis was more difficult because of sample size issues. The method provided quantitative measurement results, but the method has not been validated for use on tire crumb particles. Improved quality control methods and materials required to assess metal recoveries. Nonhomogeneity of Pb has implications for site sampling, analysis, and data interpretation.

7. References

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Appendix A

List of Sample Collection and Analysis Methods

NOTE: The following methods have not been evaluated for collecting and analyzing samples from synthetic turf fields or from playgrounds with tire crumb rubber.

Research Operating Procedure for the Collection of Particulate Matter (PM) Air Samples at Playgrounds and Synthetic Turf Fields

Research Operating Procedure for the Collection of Tire Crumb Material at Playgrounds

Research Operating Procedure for the Collection of "Grass Blade" Fibers from Synthetic Turf Fields

Research Operating Procedure for the Collection of Infill Material from Synthetic Turf Fields

Research Operating Procedure for the Collection of Volatile Organic Chemicals in Air Using Canisters

ASTM E1792-03: Standard Specification for Wipe Sampling Materials for Lead in Surface Dust

ASTM E1728-03: Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination

Recommended Operating Procedure for Elemental Analysis of Particulate Matter on Membrane Filters by the LBL XRF Spectrometer

Standard Operating Procedure for the Gravimetric Determination of Particle Mass on Teflon Air Sampling Filters

Standard Operating Procedures for the USEPA-NERL Scanning Electron Microscopy/ Energy-Dispersive X-Ray Analysis (SEM/EDX) Laboratory

Compendium Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS); (Region 1 EIASOP-AIRCAN9: "Standard Operating Procedure for the Analysis of Volatile Organic Compounds in Air by Gas Chromatography/Ion Trap Detector)"

EPA SW-846 Method 3050B: Acid Digestion of Sediments, Sludges, and Soils. Revision 2. December 1996

EPA SW-846 Method 6020A: Inductively Coupled Plasma-Mass Spectrometry. Revision 1. February 2007

EPA 9200.1-86. Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil. May 2008

Appendix B

Quality Control and Quality Assurance Results

- Table B-1. Field and Laboratory Blank Measurement Results for Volatile Organic Compounds in Air
- Table B-2. Percent Recovery of Air Volatile Organic Compounds in Field and Laboratory Controls
- Table B-3. Relative Percent Difference in Measurement Results for Air Volatile Organic Compounds in Field Duplicate Canister Samples and in Repeat Analysis of Canister Samples in the Laboratory
- Table B-4. ICP/MS Operating Parameter Settings
- Table B-5. ICP/MS Isotopes and Interference Corrections
- Table B-6. Concentrations of Individual Metals in Working Calibration Standards
- Table B-7. ICP/MS Method Detection Limits
- Table B-8. Summary of ICP/MS QC Criteria
- Table B-9. Summary of ICP/MS Instrumental QC Results
- Table B-10. Recovery of Metals Spiked in Extraction Reagent Blank
- Table B-11. Recovery of Metals-Spiked Solution on Matrix of Interest
- Table B-12. Total Extractable Recoveries for NIST SRM 2710 Spiked into Extraction Solution
- Table B-13. Total Extractable Recoveries for NIST SRM 2710 Spiked onto Ghost Wipe Media
- Table B-14. Bottle Blank Data Used for Sample Correction and Reagent Blank Data Method 3050B
- Table B-15. Laboratory and Field Ghost Wipe Blank Samples
- Table B-16. Relative Percent Differences for the Analysis of Duplicate Aliquots of Tire Crumb Infill from Synthetic Fields and Playground Tire Crumb Samples
- Table B-17. Recommended Control Limits for In Vitro Soil Quality Control Samples According to EPA Method 9200.1-86
- Table B-18. Summary Control Limit Results for In Vitro Pb Bioaccessibility Quality Control Samples
- Table B-19. Percent Recovery Results for In Vitro Blank Spikes

Table B-20. Summary of Pb In Vitro Extractable Values for NIST SRM 2710

Table B-21. Summary of Pb In Vitro Extractable Values for NIST SRM 2710 Spiked onto Ghost Wipe Media

Table B-22. Results for In Vitro Pb Solution Spikes onto Blank Ghost Wipes and Tire Crumb Samples

Table B-23. In Vitro Pb Bioaccessibility Extraction Duplicates for Synthetic Turf Field Tire Crumb Infill

Table B-24. In Vitro Pb Bioaccessibility Extraction Duplicates for Playground Tire Crumb

Table B-1. Field and Laboratory Blank Measurement Results (ppbV) for Volatile Organic Compounds (VOCs) in Air

Table B 1.1 Told and Ear	Field Blank	Sample		Field Blanks				oratory Blai		
voc	MDL	MDL	P1	F4	F1D1	P1	F2	F4	F1D1	F1D2
1,1,1-Trichloroethane	0.05	0.076 ^a	ND⁵	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethylene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4-Trichlorobenzene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4-Trimethylbenzene	0.05	0.076 ^a	ND	ND	0.360	ND	ND	ND	ND	ND
1,2-Dibromoethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane	0.05	0.076 ^a	ND	ND	0.073	ND	ND	ND	ND	ND
1,2-Dichloropropane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,3,5-Trimethylbenzene	0.05	0.076 ^a	ND	ND	0.120	ND	ND	ND	ND	ND
1,3-Butadiene	0.10	0.15 ^c	ND	ND	0.250	ND	ND	ND	ND	ND
1,3-Dichlorobenzene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
2-Hexanone	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
4-Ethyltoluene	0.05	0.076 ^a	ND	ND	0.380	ND	ND	ND	ND	ND
Acrylonitrile	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Allyl Chloride	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	0.05	0.076 ^a	ND	ND	1.11	ND	ND	ND	ND	ND
Benzylchloride	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Bromodichloromethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Bromoform	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
c-1,2-Dichloroethylene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
c-1,3-Dichloropropylene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Carbon Tetrachloride	0.05	0.076 ^a	ND	ND	0.040	ND	ND	ND	ND	ND
Chlorobenzene	0.05	0.076 ^a	ND	ND	0.510	ND	ND	ND	ND	ND
Chloroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	0.05	0.076 ^a	ND	ND	0.049	ND	ND	ND	ND	ND
Cyclohexane	0.05	0.076 ^a	ND	ND	12.0	ND	ND	ND	ND	ND
Dibromochloromethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Dichlorodifluoromethane	0.05	0.076 ^a	ND	ND	0.270	ND	ND	ND	ND	ND
Dichlorotetrafluoroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	0.05	0.076 ^a	ND	ND	1.63	ND	ND	ND	ND	ND
Heptane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Hexachloro-1,3-butadiene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Hexane	0.05	0.076 ^a	ND	ND	6.20	ND	ND	ND	ND	ND
m&p-Xylenes	0.10	0.15 ^c	ND	ND	4.27	ND	ND	ND	ND	ND
Methyl Ethyl Ketone	0.05	0.076 ^a	0.100	0.059	3.83	0.040	0.044	ND	ND	ND

Table B-1. Field and Laboratory Blank Measurement Results (ppbV) for Volatile Organic Compounds (VOCs) in Air (cont'd.)

	Field Blank	Sample		Field Blanks	3		Lab	oratory Bla	nks	
VOC	MDL	MDL	P1	F4	F1D1	P1	F2	F4	F1D1	F1D2
Methyl Isobutyl Ketone	0.05	0.076 ^a	ND	ND	0.190	ND	ND	ND	ND	ND
Methylbromide	0.05	0.076 ^a	ND	ND	0.042	ND	ND	ND	ND	ND
Methylchloride	0.05	0.076 ^a	ND	ND	0.440	ND	ND	ND	ND	ND
Methylene Chloride	0.05	0.076 ^a	ND	ND	0.400	ND	ND	ND	ND	ND
Methyl-t-butyl ether	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
o-Xylene	0.05	0.076 ^a	ND	ND	1.35	ND	ND	ND	ND	ND
Styrene	0.05	0.076 ^a	ND	ND	1.10	ND	ND	ND	ND	ND
t-1,2-Dichloroethylene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
t-1,3-Dichloropropylene	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethylene	0.05	0.076 ^a	ND	ND	0.160	ND	ND	ND	ND	ND
Tetrahydrofuran	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	0.05	0.076 ^a	ND	ND	29.0	ND	ND	ND	ND	ND
Trichloroethylene	0.05	0.076 ^a	ND	ND	0.180	ND	ND	ND	ND	ND
Trichlorofluoromethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Trichlorotrifluoroethane	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl Bromide	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND
Vinylchloride	0.05	0.076 ^a	ND	ND	ND	ND	ND	ND	ND	ND

^a Range from 0.070 to 0.079 ppbV. ^bND = not detected. ^c Range from 0.14 to 0.16 ppbV.

Table B-2. Percent Recovery^a of Air Volatile Organic Compounds (VOCs) in Field and Laboratory Controls

	Spiking		Fie	eld Contr	ols			Labor	atory Co	ntrols	
voc	Level (ppbV)	P1	F4	F1D1	Mean	Std. Dev.	P1	F4	F1D2	Mean	Std. Dev.
1,1,1-Trichloroethane	4.83	124	114	136	125	10.8	120	125	128	124	4.3
1,1,2,2-Tetrachloroethane	4.99	98	86	99	94	7.5	93	93	93	93	0.1
1,1,2-Trichloroethane	4.82	112	104	119	112	7.8	108	110	109	109	1.1
1,1-Dichloroethane	4.90	112	99	108	106	6.8	106	99	93	99	6.2
1,1-Dichloroethylene	4.78	103	97	122	108	12.8	106	101	109	105	3.9
1,2,4-Trichlorobenzene	4.09	72	44	70	62	15.7	69	61	70	66	5.0
1,2,4-Trimethylbenzene	5.04	101	84	102	96	10.1	96	98	96	97	1.2
1,2-Dichlorobenzene	4.97	91	77	91	86	8.4	88	85	86	86	1.7
1,2-Dichloropropane	4.85	112	105	119	112	6.7	102	114	109	108	5.8
1,3,5-Trimethylbenzene	4.87	111	97	112	107	8.8	111	108	109	109	1.3
1,3-Dichlorobenzene	5.00	92	77	92	87	8.5	89	83	86	86	2.9
Benzene	4.83	116	105	120	114	7.7	114	118	111	114	3.5
c-1,2-Dichloroethylene	4.80	107	97	113	106	8.1	106	99	99	101	3.9
Carbon Tetrachloride	4.84	101	26	132	87	55.0	76	107	120	101	22.7
Chlorobenzene	4.86	103	97	105	101	4.4	102	102	104	102	1.3
Chloroethane	4.81	96	66	112	91	23.2	90	101	91	94	6.1
Chloroform	4.90	110	99	106	105	5.8	103	104	96	101	4.6
Dichlorodifluoromethane	4.05	113	95	107	105	9.0	97	95	101	98	3.0
Dichlorotetrafluoroethane	3.84	103	96	109	103	6.6	100	104	99	101	2.5
Ethylbenzene	4.88	79	94	109	94	15.0	84	104	105	97	12.0
Hexachloro-1,3-butadiene	4.41	92	72	89	84	10.7	90	81	103	91	11.1
m&p-Xylenes	4.97	106	95	111	104	8.3	106	108	106	107	1.0
Methylchloride	4.38	90	103	98	97	6.5	89	87	82	86	3.8
Methylene Chloride	4.91	111	101	114	108	6.7	105	105	101	104	2.4
o-Xylene	4.60	108	93	111	104	9.7	102	105	104	104	1.2
Tetrachloroethylene	4.86	109	100	108	106	5.0	101	107	105	104	3.0
Toluene	4.83	107	101	115	108	7.1	107	108	108	108	0.3
Trichloroethylene	4.92	113	103	112	109	5.3	98	107	109	105	5.5
Trichlorofluoromethane	4.76	117	106	120	114	7.3	114	116	110	113	3.1
Vinylchloride	3.09	104	97	116	106	9.2	103	105	102	103	1.4

^a Found * 100

Table B-3. Relative Percent Difference (RPD)^a in Measurement Results for Air Volatile Organic Compounds (VOCs) in Field Duplicate Canister Samples and in Repeat Analysis of Canister Samples in the Laboratory

		Field Duplicates				Laboratory Repeat Analysis						
	RPD	RPD	RPD		Mean	Std. Dev	RPD	RPD	RPD		Mean	Std. Dev.
VOC	P1	F4	F1D1		RPD	RPD	P1	F4	F1D1		RPD	RPD
Benzene	4.8	13.3	9.5		9.2	4.3	12.8	0.0	7.9		6.9	6.4
Carbon Tetrachloride	9.3	3.4	8.3		7.0	3.1	12.4	1.1	1.0		4.9	6.6
Dichlorodifluoromethane	3.8	0.0	7.7		3.8	3.8	1.9	4.1	5.6		3.9	1.9
Hexane	44.9	28.6	105.5		59.7	40.5	_	0.0	31.2		15.6	22.1
m&p-Xylenes	-	2.6	74.2		38.4	50.6	_	8.7	3.7		6.2	3.5
Methyl Ethyl Ketone	11.8	4.8	28.6		15.0	12.2	0.0	2.0	14.6		5.6	7.9
Methyl Isobutyl Ketone	_	_	21.1		21.1		_	_	_			
Methylchloride	8.5	2.1	4.1		4.9	3.3	0.0	0.0	8.7		2.9	5.0
Methylene Chloride	44.4	20.3	_		32.4	17.0	4.3	8.7	12.0		8.3	3.8
Toluene	107.1	6.5	143.9		85.8	71.1	6.9	5.4	30.8		14.4	14.2
Trichlorofluoromethane	0.0	0.0	11.3		3.8	6.5	0.0	0.0	11.3		3.8	6.5
Trichlorotrifluoroethane	2.7	1.4	1.2		1.8	0.8	2.7	2.8	7.7		4.4	2.8

^a100 * ABS(M1- M2) / [(M1+M2) / 2}

Table B-4. ICP/MS Operating Parameter Settings

Instrument Settings						
RF Power	1,200-1,260 W					
Ar Gas Flow Rates:						
Cool	13 Lpm					
Auxiliary	0.9-1.0 Lpm					
CCT Gas Flow (He 93%/ H 7%)	~10 µL/min					
Sampler Cone (Ni/Cu)	1.1-mm diameter orifice	sample cone				
Skimmer Cone (Ni/Cu)	0.75-mm diameter skimn	0.75-mm diameter skimmer cone				
Nebulizer	Concentric nebulizer, 35	PSI, 1 mL/min				
Spray Chamber	Air-cooled cyclone					
Detector Dead Time	55 ns					
Internal Standard Solution	40-200 ppb solution of 6	Li 45 Sc 89 Y In115 and 1	159Tb			
Acquisition Parameters (Norma	l Mode)	1				
Major	Minor	Global	Add. Gases			
Extraction	Lens	Standard resolution	CCT - 0			
Lens 1	Forward power 1,400	High resolution				
Lens 2	Horizontal	Analogue detector				
Focus	Vertical	PC detector				
D1	DA					
D2 -140	Cool					
Pole Bias 0	Auxiliary					
Hexapole Bias -4.0	Sampling depth 130-140					
Acquisition Parameters (Collision	on Cell Technology [CCT]	Mode)	1			
Major	Minor	Global	Add. Gases			
Extraction	Lens	Standard resolution	CCT – ~10 µL/min			
Lens 1	Forward power 1,400	High resolution				
Lens 2	Horizontal	Analogue detector				
Focus	Vertical	PC detector				
D1	DA					
D2 -140	Cool					
Pole Bias -17.0	Auxiliary					
Hexapole Bias -20.0	Sampling depth 130-140					
Operating Peremeters						
Operating Parameters	0.75 - 0.4					
Standard Resolution	0.75 ± 0.1 amu					
High Resolution	0.30 ± 0.1 amu					
Integration Type	Average					
Calibration Type	Linear					
Number of scans per replicate	1					
Number of replicates (runs)	3-7					

Table B-5. ICP/MS Isotopes and Interference Corrections

i able 6-3. ICF/INS ISOlopes and interferen					
Analyte	Mass	Interference			
Pb	207				
Pb	208				
Cr	52				
Zn	66				
Al	27				
As	75				
Ва	137				
Cd	111				

Analyte	Mass	Interference
Cd	114	-0.027 * 118 Sn
Cu	65	
Fe	54	-0.028 * 52 Cr
Fe	56	-0.15 * 43 Ca
Mn	55	
Ni	60	-0.002 * 34 Ca
Ni	62	

Table B-6. Concentrations of Individual Metals in Working Calibration Standards

Table B c.	LoCal1	LoCal2	HiCal1	HiCal2	LLQC CRI	LoCal3	LoCal2
Metal	(ppb)		(ppm)	(ppm)	(ppb)	(ppb)	1:100 (ppb)
-		(ppb)	(ppiii)	(ppiii)	(ppb)		
Pb	250	500			1	1,000	5
Cr	250	500			2	1,000	5
Zn	250	500			2	1,000	5
Al	250	500			30	1,000	5
As	250	500			1	1,000	5
Ва	250	500			10	1,000	5
Cd	250	500			1	1,000	5
Cu	250	500			2	1,000	5
Fe	250	500	50	100		1,000	5
Mn	250	500			1	1,000	5
Ni	250	500			1	1,000	5
Internal Standards							
Li	200						
Sc	200						
Υ	40						
In	40						
Tb	40						

Table B-7, ICP/MS Method Detection Limits (ppb)^a

Metal	Isotope	In Vitro Method	EPA Method 3050B
Pb	208	0.082	0.092
Cr	52	0.201	0.175
Zn	66	0.409	0.388
Al	27	1.99	2.47
As	75	0.366	0.246
Ва	137	0.668	0.634
Cd	111	0.219	0.224
Cu	65	0.335	0.350
Fe	56	6.4	4.3
Mn	55	0.178	0.144
Ni	60	0.268	0.332

^aCalculated by formula in 40 CRF Part 136. MDLs reported here are for sample extracts delivered to the instrument. For In vitro matrix: First CRI chosen per day over 6 analysis days plus 1 extra. (Note: One metal (Fe) not spiked into CRI so concentration at reagent blank level.

For EPA 3050B matrix: One CRI chosen per day over 7 analysis days.

Table B-8. Summary of ICP/MS QC Criteria^a

Name	Acceptance Criteria	Minimum Value Requirement
Initial Calibration Verification	90%-110%	-
Initial Calibration Blank	<crql<sup>b</crql<sup>	
Continuing Calibration Verification	90%-110%	
Continuing Calibration Blank	<crql< td=""><td></td></crql<>	
-	±3CRQL or ±20%	
Interference Check Solution A	true value	
	±3CRQL or ±20%	
Interference Check Solution AB	true value	
	±30%, except ±50%	
Contract Required Quantitation Limit Check	for Co, Mn, Zn	
Duplicate Samples Relative Percent Difference	<20%	50 times CRQL
Serial Dilution Samples Percent Difference	<10%	50 times CRQL
Post Digestion Spike Samples	75%-125%	
Sample Extract QC		
Spike	75%-125%	
SRM	75%-125%	

^aBased on EPA Method 6020A QC criteria. ^bCRQL = contract required quantitation limit.

Table B-9. Summary of ICP/MS Instrumental QC Results

III EPA 3030I	B 5% HNO ₃ Matr	1X, n=4		
Metal	Mass (amu)	DUP ^a Criteria	DUP RPD ^b Found	Status (pass/fail)
Pb	208	<20%	0.117%-7.83%	P
Cr	52	<20%	0.145%-3.54%	Р
Zn	66	<20%	0.081%-1.26%	Р
Al	27	<20%	0.023%-5.26%	Р
As	75	<20%	0.929%-12.8%	Р
Ва	137	<20%	0.563%-1.41%	Р
Cd	111	<20%	1.048%-1.26%	Р
Cu	65	<20%	0.011%-1.40%	Р
Fe	56	<20%	0.110%-2.37%	Р
Mn	55	<20%	1.09%-3.61%	Р
Ni	60	<20%	0.478%-2.50%	Р
Metal	Mass	_		
	(amu)	SER ^c Criteria	SER Difference	Status (pass/fail)
Pb	208	<10%	2.54%-7.24%	Р
Cr	52	<10%	0.167%	Р
Zn	66	<10%	2.41%-7.74%, 14.8%	F
Al	27	<10%	1.50%-4.31%	P
As	75	<10%	*d	N/A
Ва	137	<10%	0.938%-4.99%	Р
Cd	111	<10%	*	N/A
Cu	65	<10%	2.34%-5.72%	Р
Fe	56	<10%	*	N/A
Mn	55	<10%	*	N/A
Ni	60	<10%	3.43%	Р
Metal	Mass (amu)	PDS ^e Criteira	PDS Recovery	Status (pass/fail)
Pb	208	75%-125%	100%-104%	Р
Cr	52	75%-125%	86.7%-89.0%	Р
Zn	66	75%-125%	99.6%-109%	Р
Al	27	75%-125%	*	N/A
As	75	75%-125%	98.7%-105%	Р
Ba	137	75%-125%	77.2%-100%	Р
Cd	111	75%-125%	94.6%-103%	Р
Cu	65	75%-125%	96.5%-101%	Р
Fe	56	75%-125%	*	N/A
Mn	55	75%-125%	*	N/A
	60	1	92.8%-99.2%	Р

Table B-9. Summary of ICP/MS Instrumental QC Results (cont'd.)

III III VILIO Z	<mark>% HNO₃ Matrix, r</mark> Mass	i - 3		
Metal	(amu)	DUP Criteria	DUP RPD Found	Status (pass/fail)
Pb	208	<20%	0.684%-4.70%	P
Cr	52	<20%	*	N/A
Zn	66	<20%	0.104%-1.98%	Р
Al	27	<20%	1.90%-11.0%	Р
As	75	<20%	*	N/A
Ва	137	<20%	0.142%-2.38%	Р
Cd	111	<20%	*	N/A
Cu	65	<20%	0.142%-9.38%	Р
Fe	56	<20%	*	N/A
Mn	55	<20%	*	N/A
Ni	60	<20%	*	N/A
Metal	Mass			
wetai	(amu)	SER Criteria	SER Difference	Status (pass/fail)
Pb	208	<10%	0.062%	Р
Cr	52	<10%	*	N/A
Zn	66	<10%	0.169%-9.40%	Р
Al	27	<10%	8.49%, 26.0%	F
As	75	<10%	*	N/A
Ва	137	<10%	0.045%-2.51%	Р
Cd	111	<10%	*	N/A
Cu	65	<10%	*	N/A
Fe	56	<10%	*	N/A
Mn	55	<10%	*	N/A
Ni	60	<10%	*	N/A
Metal	Mass (amu)	PDS Criteria	PDS Recovery	Status (pass/fail)
Pb	208	75%-125%	94.7%-106%	P
Cr	52	75%-125%	91.2%-102%	Р
Zn	66	75%-125%	95.4%-110%	Р
Al	27	75%-125%	*	N/A
As	75	75%-125%	90.0%-103%	Р
Ва	137	75%-125%	88.0%-108%	Р
Cd	111	75%-125%	97.3%-107%	Р
Cu	65	75%-125%	91.2%-111%	Р
Fe	56	75%-125%	*	N/A
Mn	55	75%-125%	*	N/A
Ni	60	75%-125%	97.2%-108%	Р

aDUP = duplicate aliquot of extract analyzed independently.
bRPD = relative percent difference.
cSER = serial dilution of extract.

dConcentrations too low to meet QC criteria.

ePDS = postdigestion spike.

Table B-10. Recovery of Metals Spiked in Extraction Reagent Blank

Sample ID	Description	Spiked Amount (total ug spiked)	Net Total Extractable (total ug found)	Percent Recovery
	•	Primary Metals of Int	terest	
Cr				
TC1-2	Blank Spike	640	600	93.7
TC2-2	Blank Spike	640	616	96.3
TC3-2	Blank Spike	2,500	2,340	93.8
TC4-2	Blank Spike	2,500	2,350	94.1
TC5-2	Blank Spike	2,500	2,320	92.7
TC6-2	Blank Spike	2,500	2,390	95.8
TC7-2	Blank Spike	2,500	2,370	94.8
TC8-2	Blank Spike	2,500	2,380	95.0
X (n=8)	biatik Spike	2,300	2,380	94.5
` '				94.5
Pb				
TC1-2	Blank Spike	1,000	949	94.9
TC2-2	Blank Spike	1,000	1,070	107
TC3-2	Blank Spike	1,000	987	98.7
TC4-2	Blank Spike	1,000	985	98.5
TC5-2	Blank Spike	1,000	980	98.0
TC6-2	Blank Spike	1,000	974	97.4
TC7-2	Blank Spike	1,000	999	99.9
TC8-2	Blank Spike	1,000	1,000	100
X (n=8)				99.3
Zn				
TC1-2	Blank Spike	800	774	96.8
TC2-2	Blank Spike	800	747	93.4
TC3-2	Blank Spike	2,500	2,390	95.7
TC4-2			,	95.5
	Blank Spike	2,500	2,390 2,370	94.8
TC5-2	Blank Spike	2,500		
TC6-2	Blank Spike	2,500	2,420	96.7
TC7-2	Blank Spike	2,500	2,400	95.9
TC8-2	Blank Spike	2,500	2,400	96.1
X (n=8)				95.6
<u></u>		Secondary Metals of I	nterest	
AI				
TC1-2	Blank Spike	0	5.40	Not spiked
TC2-2	Blank Spike	0	31.4	Not spiked
TC3-2	Blank Spike	0.2	44.2	Not spiked
TC4-2	Blank Spike	0.2	38.3	Not spiked
TC5-2	Blank Spike	0.2	36.7	Not spiked
TC6-2	Blank Spike	0.2	29.3	Not spiked
TC7-2	Blank Spike	0.2	32.9	Not spiked
TC8-2	Blank Spike	0.2	20.6	Not spiked
X	Diain Opino	V. <u>L</u>	20.0	Not spiked
As				opinou
TC1-2	Blank Spike	0	23.1	Not spiked
TC2-2	Blank Spike	0	22.9	Not spiked
TC3-2	Blank Spike	500	449	89.8
			459	91.8
TC4-2	Blank Spike	500		
TC5-2	Blank Spike	500	449	89.8
TC6-2	Blank Spike	500	423	84.5
TC7-2	Blank Spike	500	453	90.6
TC8-2	Blank Spike	500	463	92.5
X (n=6)				89.8

Table B-10. Recovery of Metals Spiked in Extraction Reagent Blank (cont'd.)

Sample ID	Description	Spiked Amount (total µg spiked)	Net Total Extractable (total µg found)	Percent Recovery
Ва		(10 m p 3 op m c m)	(real page and a	
TC1-2	Blank Spike	0	2.97	Not spiked
TC2-2	Blank Spike	0	4.58	Not spiked
TC3-2	Blank Spike	2,510	2,420	96.6
TC4-2			,	98.1
	Blank Spike	2,510	2,460	96.3
TC5-2 TC6-2	Blank Spike	2,510	2,420	
TC7-2	Blank Spike	2,510	2,480	98.6
	Blank Spike	2,510	2,490	99.2
TC8-2	Blank Spike	2,510	2,490	99.1
X (n=6)				98.0
Cd				
TC1-2	Blank Spike	160	157	98.0
TC2-2	Blank Spike	160	150	93.6
TC3-2	Blank Spike	497	475	95.6
TC4-2	Blank Spike	497	480	96.6
TC5-2	Blank Spike	497	465	93.5
ГС6-2	Blank Spike	497	483	97.3
TC7-2	Blank Spike	497	480	96.6
TC8-2	Blank Spike	497	480	96.5
X (n=8)	'			96.0
Cu				
TC1-2	Plank Chika	800	702	99.1
	Blank Spike	800	793	100
TC2-2 TC3-2	Blank Spike	800	800	
	Blank Spike	2,540	2,410	95.0
TC4-2	Blank Spike	2,540	2,410	95.0
TC5-2	Blank Spike	2,540	2,400	94.6
TC6-2	Blank Spike	2,540	2,450	96.3
TC7-2	Blank Spike	2,540	2,420	95.4
TC8-2	Blank Spike	2,540	2,430	95.5
X (n=8)				96.4
Fe				
TC1-2	Blank Spike	0	0	Not spiked
TC2-2	Blank Spike	0	27.7	Not spiked
TC3-2	Blank Spike	0	3.00	Not spiked
ΓC4-2	Blank Spike	0	0.222	Not spiked
TC5-2	Blank Spike	0	2.12	Not spiked
TC6-2	Blank Spike	0	0.213	Not spiked
TC7-2	Blank Spike	0	0.668	Not spiked
ГС8-2	Blank Spike	0	0.219	Not spiked
	opo			Not spiked
X				тиот зыкеа
VIn				
ΓC1-2	Blank Spike	400	377	94.2
ΓC-2-2	Blank Spike	400	386	96.4
ГС3-2	Blank Spike	0.02	0.311	Not spiked
TC4-2	Blank Spike	0.02	0.004	Not spiked
ΓC5-2	Blank Spike	0.02	0.028	Not spiked
TC6-2	Blank Spike	0.02	0	Not spiked
ГС7-2	Blank Spike	0.02	0.010	Not spiked
TC8-2	Blank Spike	0.02	0.121	Not spiked
X (n=2)	•			95.3

Table B-10. Recovery of Metals Spiked in Extraction Reagent Blank (cont'd.)

Sample ID	Description	Spiked Amount (total µg spiked)	Net Total Extractable (total µg found)	Percent Recovery
Ni				
TC1-2	Blank Spike	0	0.869	Not spiked
TC-2-2	Blank Spike	0	0.312	Not spiked
TC3-2	Blank Spike	1,250	1,220	97.2
TC4-2	Blank Spike	1,250	1,220	97.7
TC5-2	Blank Spike	1,250	1,200	95.8
TC6-2	Blank Spike	1,250	1,240	99.2
TC7-2	Blank Spike	1,250	1,220	97.5
TC8-2	Blank Spike	1,250	1,230	98.3
X (n=6)				97.6

Table B-11. Recovery of Metals-Spiked Solution on Matrix of Interest

		Net Spiked	Net Media	Media	
M. P 10 1	Spiked	Total	Total	Corrected	Percent
Media and Sample	Amount (µg)	Extractable (µg)	Extractable (µg)	Spike (µg)	Recovery
Pb on Wipes Blank Ghost Wipe			0.227		
Wipe with Spike	1,000	921	0.221	920	92.0
wipe with Spike	1,000	321		920	92.0
Blank Ghost Wipe			0.694		
Wipe with Spike	991	994	0.001	993	100
					,,,,,
Blank Ghost Wipe			1.08		
Wipe with Spike	991	995		994	100
Mean					99.4
Pb on Infill	ı				
F4-L1-S1-A1	4.000	07.4	41.1	222	20.0
F4-L1-S1-A1	1,000	974		933	93.3
E4 1 D1 A1			24.0		
F4-L1-D1-A1 F4-L1-D1-A1	1,000	214	24.8	190	19.0
F4-LI-DI-AI	1,000	214		190	19.0
F1-L3-S1-A1			20.6		
F1-L3-S1-A1	991	854	20.0	834	84.1
				00.	<u> </u>
F2-L2-D1-A1			36.4		
F2-L2-D1-A1	991	889		853	86.0
Pb on Crumb					
P1-LA-TC1			0.989		
P1-LA-TC1	1,000	317		316	31.6
D4 I A T00			2.24		
P1-LA-TC2 P1-LA-TC2	991	233	6.31	227	22.9
F 1-LA-102	991	233		221	22.9
P1-LB-TC1			443		
P1-LB-TC1	991	276	110	-167	N/A
1 1 25 101	331	210		107	14/71
Cr on Wipes	I				
Blank Ghost Wipe			0.091		
Wipe with Spike	640	580		580	90.7
Blank Ghost Wipe			6.24		
Wipe with Spike	2,490	2,400		2,390	96.1
DI LOI (M			10.0		
Blank Ghost Wipe	0.400	0.070	13.0	0.000	04.0
Wipe with Spike Mean	2,490	2,370		2,360	94.8 93.9
ivicali					93.9
Cr on Infill	1				
F4-L1-S1-A1			0.602		
F4-L1-S1-A1	640	596	3.332	595	93.0
<u> </u>					23.0
F4-L1-D1-A1			0.352		
F4-L1-D1-A1	640	106		106	16.6
F1-L3-S1-A1			0.544		
F1-L3-S1-A1	2,490	2,160		2,160	86.7

Table B-11. Recovery of Metals-Spiked Solution on Matrix of Interest (cont'd.)

		Net Spiked	ion on Matrix of Net Media	Media	
	Spiked	Total	Total	Corrected	Percent
Media and Sample	Amount (µg)	Extractable (µg)	Extractable (µg)	Spike (µg)	Recovery
F2-L2-D1-A1			0.018		
F2-L2-D1-A1	2,490	2,080		2,080	83.6
Mean					69.9
Cr on Crumb					
Cr on Crumb P1-LA-TC1	T		0.281		
P1-LA-TC1	640	115	0.201	115	17.9
I I-LA-IOI	040	110		110	17.5
P1-LA-TC2			0.721		
P1-LA-TC2	2,490	598		598	24.0
DA LD TOA			0.704		
P1-LB-TC1 P1-LB-TC1	2,490	788	0.761	787	31.6
Mean	2,490	700		101	24.4
Mean					24.4
Zn on Wipes					
Blank Ghost Wipe			4.06		
Wipe with Spike	800	763		759	94.8
DI LOI (M"			40.0		
Blank Ghost Wipe	2.400	0.400	13.0	0.400	00.5
Wipe with Spike	2,490	2,420		2,400	96.5
Blank Ghost Wipe			23.6		
Wipe with Spike	2,490	2,430		2,410	96.7
Mean	,	,		,	96.0
Zn on Infill	1		0.040		
F4-L1-S1-A1 F4-L1-S1-A1	000	0.000	9,940	-989	NI/A
F4-L1-51-A1	800	8,960		-909	N/A
F4-L1-D1-A1			4,880		
F4-L1-D1-A1	800	6,040	1,000	1,159	1,459
		,		•	·
F1-L3-S1-A1			7,930		
F1-L3-S1-A1	2,490	21,700		13,800	555
F2-L2-D1-A1			10,300		
F2-L2-D1-A1	2,490	11,700	10,000	1,420	56.8
Mean		, ,		.,0	251
Zn on Crumb	1				
P1-LA-TC1	000	5.070	4,330	0.40	4
P1-LA-TC1	800	5,270		940	117.5
P1-LA-TC2			6,730		
P1-LA-TC2	2,490	13,100	2,1.00	6,380	256
	,	,		,	
P1-LB-TC1			17,500		
P1-LB-TC1	2,490	14,000		-3,430	N/A
Mean					186

Table B-12. Total Extractable Recoveries for NIST SRM 2710 Spiked into Extraction Solution

Metal	Batch	SRM Certified Concentration (ug/g)	Found Concentration (ug/g)	Percent Recovery	SRM Percent Leachable Recovery
Mean Cra Mean Pb Mean Zn	1	626	535	85.5	94
-	2	626	502	80.3	94
	3	626	514	82.0	94
	4	626	492	78.5	94
	6	626	502	80.2	94
	8	626	498	79.5	94
Mean				81.0	
Cr ^a	1	39	14.2	36.3	49
<u> </u>	2	39	16.3	41.8	49
	3	39	15.1	38.6	49
	4	39	10.1	26.0	49
	6	39	14.8	38.0	49
	8	39	13.8	35.4	49
Mean				37.5	
Pb	1	5,530	5,100	92.2	92
	2	5,530	4,740	85.7	92
	3	5,530	4,830	87.4	92
	4	5,530	4,830	87.3	92
	6	5,530	4,740	85.6	92
	8	5,530	4,630	83.7	92
Mean		·	·	87.0	
Zn	1	6,950	5,640	81.2	85
	2	6,950	5,040	72.5	85
	3	6,950	5,580	80.3	85
	4	6,950	5,300	76.2	85
	6	6,950	5,140	73.9	85
	8	6,950	5,200	74.8	85
Mean				76.5	
Al	1	64,400	18,300	28.3	28
	2	64,400	18,900	29.4	28
	3	64,400	18,400	28.5	28
	4	64,400	16,900	26.3	28
	6	64,400	17,600	27.3	28
	8	64,400	17,200	26.8	28
Mean				27.8	
Ва	1	707	329	46.6	51
	2	707	311	44.0	51
	3	707	267	37.8	51
	4	707	298	42.1	51
	6	707	304	43.0	51
	8	707	322	45.5	51
Mean				43.2	
Cd	1	21.8	17.0	78.1	92
	2	21.8	17.8	81.4	92
	3	21.8	20.2	92.7	92
	4	21.8	18.8	86.3	92
	6	21.8	17.9	82.2	92
	8	21.8	18.7	85.7	92
Mean			1	84.4	

Table B-12. Total Extractable Recoveries for NIST SRM 2710 Spiked into Extraction Solution (cont'd.)

Metal	Batch	SRM Certified Concentration (ug/g)	Found Concentration (ug/g)	Percent Recovery	SRM Percent Leachable Recovery
Cu	1	2,950	2,630	89.0	92
	2	2,950	2,490	84.3	92
	3	2,950	2,570	87.2	92
	4	2,950	2,500	84.6	92
	6	2,950	2,490	84.4	92
	8	2,950	2,510	85.0	92
Mean		·	·	85.8	
Fe	1	33,800	28,500	84.4	80
	2	33,800	24,100	71.2	80
	3	33,800	27,500	81.4 75.0	80
	4	33,800	25,400		80
	6	33,800	23,400	69.4	80
	8	33,800	23,000	67.9	80
Mean		·	·	74.9	
Mn	1	10,100	7,250	71.8	76
	2	10,100	6,890	68.2	76
	3	10,100	7,080	70.1	76
	4	10,100	6,610	65.4	76
	6	10,100	6,860	67.9	76
	8	10,100	6,880	68.2	76
Mean				68.6	
Ni	1	14.3	7.89	55.2	71
	2	14.3	9.89	69.2	71
	3	14.3	8.70	60.8	71
	4	14.3	5.50	38.5	71
	6	14.3	8.39	58.7	71
	8	14.3	7.96	55.6	71
Mean				56.3	

^aNot certified for SRM.

Table B-13. Total Extractable Recoveries for NIST SRM 2710 Spiked onto Ghost Wipe Media

Media Metal	Туре	Batch	SRM Certified Total Conc. (ug/g)	Found Conc. (ug/sample)	Blank Corrected Conc. (ug/g)	Percent Recovery	SRM Percent Leachable Recovery
As	SRM	1	626	507	507	80.90	94
7.0	Blank	1	020	0.15	001	00.00	<u> </u>
As	SRM	8	626	543	543	86.80	94
	Blank	8		0.13			
As	SRM	9	626	496	496	79.2	94
	Blank	9	<u> </u>	0.12			
Mean						82.3	
Cr ^a	SRM	1	39	14.1	14.0	36.0	49
	Blank	1		0.09		00.0	
Cr	SRM	8	39	17.7	11.4	29.3	49
	Blank	8		6.23			
Cr	SRM	9	39	13.8	0.76	1.96	49
Oi	Blank	9	- 55	13.0	0.70	1.50	40
Mean (n=3)		_				22.4	
Mean (n=2)						32.6	
Pb							
	SRM	1	5,530	4,950	4,950	89.5	92
	Blank	1	·	0.23	,		
	SRM	8	5,530	5,040	5,040	91.1	92
	Blank	8		0.69			
	SRM	9	5,530	4,500	4,500	81.4	92
	Blank	9	0,000	1.08	.,000		
Mean						87.3	
Zn							
<u> </u>	SRM	1	6,950	5,570	5,560	80.0	85
	Blank	1	,	5.11	,		
	SRM	8	6,950	5,800	5,780	83.2	85
	Blank	8		12.9			
	SRM	9	6,950	5,260	5,240	75.4	85
	Blank	9	0,000	23.7	0,240	70.7	- 55
Mean						79.5	
Al							
7.51	SRM	1	64,400	18,100	18,100	28.2	28
	Blank	1	,	0.37	,		
	SRM	8	64,400	20,000	20,000	31.1	28
	Blank	8		2.14			
	SRM	9	64,400	18,500	18,500	28.8	28
	Blank	9	01,400	7.71	10,000	20.0	20
Mean						29.3	

Table B-13. Total Extractable Recoveries for NIST SRM 2710 Spiked onto Ghost Wipe Media (cont'd.)

Media (co Metal	Туре	Batch	SRM Certified Total Conc. (ug/g)	Found Conc. (ug/sample)	Blank Corrected Conc. (ug/g)	Percent Recovery	SRM Percent Leachable Recovery
Ва						_	
Du	SRM	1	707	316	309	43.8	51
	Blank	1		6.84			
	SRM	8	707	353	330	46.7	51
	Blank	8		22.7			
	SRM	9	707	324	298	42.2	51
	Blank	9		25.5			
Mean						44.2	
Cd							
	SRM	1	21.8	16.4	16.4	75.0	92
	Blank	1		0.03			
	SRM	8	21.8	18.2	18.1	83.2	92
	Blank	8		0.06			
	SRM	9	21.8	17.9	17.9	81.9	92
	Blank	9	21.0	0.02	17.0	01.0	02
Mean						80.1	
Cu							
	SRM	1	2,950	2,540	2,540	86.2	92
	Blank	1		0.02			
	SRM	8	2,950	2,630	2,630	89.3	92
	Blank	8		0.25			
	SRM	9	2.050	2.450	2.450	00.4	00
	Blank	9	2,950	2,450 0.44	2,450	83.1	92
Mean	Biarik	J		0.44		86.2	
Fe							
	SRM	1	33,800	27,500	27,500	81.4	80
	Blank	1		0.20			
	SRM	8	33,800	29,100	29,100	86.0	80
	Blank	8		26.7			
	SRM	9	33,800	23,800	23,700	70.2	80
	Blank	9	=5,555	61.2			
Mean						79.2	
Mn							
	SRM	1	10,100	7,050	7,050	69.8	76
	Blank	1		0.03			
	SRM	8	10,100	7,310	7,300	72.3	76
	Blank	8		10.3			
	SRM	9	10,100	6,980	6,960	68.9	76
	Blank	9	10,100	23.3	0,000	30.9	, 5
Mean						70.3	

Table B-13. Total Extractable Recoveries for NIST SRM 2710 Spiked onto Ghost Wipe Media (cont'd.)

Metal	Туре	Batch	SRM Certified Total Conc. (ug/g)	Found Conc. (ug/sample)	Blank Corrected Conc. (ug/g)	Percent Recovery	SRM Percent Leachable Recovery
Ni							
	SRM	1	14.3	7.86	7.86	55.0	71
	Blank	1		0.00			
	SRM	8	14.3	9.96	6.55	45.8	71
	Blank	8		3.42			
	SRM	9	14.3	7.98	0.91	6.34	71
	Blank	9	-	7.08			
Mean (n=3)						35.7	
Mean (n=2)						50.4	

^aNot certified for SRM.

Table B-14. Bottle Blank Data Used for Sample Correction and Reagent Blank Data Method 3050B (ng/mL)

	As	Cr	Pb	Zn	Al	Ва	Cd	Cu	Fe	Mn	Ni
For In Vitro 2	% HNO3				<u> </u>		<u> </u>				
Bottle Blank II											
TC 1-1	0.13	0.252	0.292	68.0	23.0	125	0.007	1.36	0.006	0.132	0.12
TC 2-1	-0.016	0.059	0.114	10.4	3.46	11	-0.001	0.926	0.0	0.08	0.03
TC 3-1	0.126	0.466	0.293	97.3	31.1	174	0.05	1.51	0.007	0.132	0.20
TC 4-1	0.023	0.035	0.05	7.72	3.29	8.18	0.001	0.23	0.000	0.017	0.01
TC 5-1	0.002	0.109	0.106	54.4	16.2	119	0.001	0.716	0.003	0.043	0.01
TC 6-1	0.005	0.132	0.138	58.5	17.8	126	0.001	1.27	0.003	0.061	0.07
TC 7-1	-0.022	0.027	0.087	9.60	2.81	11.4	0	0.372	0.001	0.023	0.02
TC 8-1	-0.015	0.043	0.07	8.04	3.22	9.04	0	0.42	0.001	0.06	0.00
TC 9-1	-0.031	0.041	0.071	8.63	1.70	7.31	0.002	0.261	0.000	0.012	0.07
Mean (n=9)	0.057	0.129	0.136	35.8	11.4	65.6	0.008	0.784	0.003	0.062	0.06
Std. dev.	0.065	0.145	0.093	34.1	10.9	68.4	0.017	0.499	0.002	0.046	0.06
%RSD	114	112	68.3	95.2	95.6	104.3	222	63.6	98.0	73.1	102
MDL	0.367	0.201	0.082	0.409	1.99	0.668	0.219	0.335	0.006	0.178	0.26
Eo. EDA 2050	D EO/ LINC		•	•	•				•	•	
For EPA 3050 Bottle Blank II		/3									
TC 1-1	1.04	1.45	6.91	192	128	179	0.088	13.4	0.113	9.40	3.7
TC 2-1	1.28	1.10	2.69	148	85.6	153	0.061	7.63	0.047	2.48	2.9
TC 3-1	0.755	1.29	1.84	157	91.6	197	0.034	3.61	0.042	1.47	2.7
TC 4-1	1.40	48.6	4.407	229	100	193	0.811	7.88	0.191	52.1	27.8
TC 5-1	0.761	5.49	1.65	288	102	192	0.413	14.8	0.034	2.70	2.90
TC 6-1	0.814	3.38	1.54	185	88.5	178	0.19	5.53	0.041	4.15	2.50
TC 7-1	0.383	40.5	0.791	208	90.7	183	1.40	2.90	0.194	48.8	25.6
TC 8-1	0.426	5.67	2.54	182	103	191	0.18	5.31	0.056	9.25	3.24
TC 9-1	1.47	6.97	3.22	170	82.0	169	1.06	8.52	0.029	4.13	7.73
Mean (n=9)	0.925	12.7	2.84	195	96.9	182	0.471	7.72	0.083	14.9	8.80
Std. dev.	0.398	18.3	1.86	42.4	13.9	14.0	0.500	4.08	0.067	20.4	10.3
%RSD	43.0	144	65.3	21.7	14.4	7.7	106	52.8	80.5	136	117
MDL	0.246	0.175	0.092	0.388	2.47	0.634	0.224	0.350	0.004	0.144	0.33
For EPA 3050)B Only Re	agent Bla	anks 5%	HNO ₂ No	ot Stored	l in Bottle	es				
Reagent Blanl		<u></u>									
TC-4-13	Lost	Lost	Lost	Lost	Lost	Lost	Lost	Lost	Lost	Lost	Los
TC 5-13	0.936	5.20	1.56	301	92.6	173	0.472	11.3	0.044	2.69	12.3
TC 6-13	0.971	1.02	7.04	166	95.9	168	0.035	6.31	0.109	11.2	2.93
TC 7-13	0.074	8.15	1.39	156	78.6	187	0.314	1.37	0.038	9.01	5.63
TC 9-13	0.382	271	2.76	285	60.8	167	2.74	3.44	1.043	460	152
Mean (n=4)	0.591	71.2	3.19	227	82.0	174	0.890	5.60	0.308	121	43.2
Std. dev.	0.438	133	2.64	76.3	16.0	9.15	1.24	4.28	0.491	226	72.
%RSD	74.1	187	82.8	33.6	19.5	5.3	140	76.6	159	187	168

Table B-15. Laboratory and Field Ghost Wipe Blank Samples (not blank corrected;	
ng/mL)	

Blank											
Wipe ID	As	Cr	Pb	Zn	Al	Ва	Cd	Cu	Fe	Mn	Ni

Laboratory	/ Blank Wipes
Laborator	Dialik Wibes

For In Vit	ro 2% HN	IO ₃									
TC 1-11	0.144	0.183	0.282	67.7	13.4	95.6	0.083	1.45	0.006	0.224	0.077
TC 8-12	0.065	0.13	0.695	55.5	12.9	118	0.012	0.944	0.005	0.167	0.034
TC 9-2	0.038	0.113	0.227	57.6	15.3	115	-0.001	0.902	0.003	0.086	0.064
Mean	0.082	0.142	0.401	60.3	13.8	110	0.048	1.099	0.005	0.159	0.058
std dev	0.055	0.037	0.256	6.53	1.29	12.4	0.050	0.306	0.002	0.069	0.022
%RSD	66.9	25.7	63.7	10.8	9.3	11.3	106	27.8	32.7	43.6	37.8
MDL	0.367	0.201	0.082	0.409	1.99	0.668	0.219	0.335	0.006	0.178	0.268
For EPA	3050B 5%	6 HNO₃									
TC 1-11	2.45	2.34	9.20	232	133	261	0.252	9.58	0.114	9.41	2.16
TC 8-12	1.61	67.9	8.10	207	97.5	177	0.777	6.61	0.313	112	37.4
TC 9-2	2.47	136	13.7	297	124	179	1.24	11.6	0.63	246	78.2
Mean	2.17	68.9	10.3	245	118	205	0.75	9.27	0.35	123	39.3
std dev	0.49	67.0	2.95	46.4	18.0	47.8	0.49	2.53	0.26	119	38.0
%RSD	22.6	97.3	28.5	18.9	15.3	23.3	65.2	27.2	73.9	96.8	96.9
MDL	0.25	0.17	0.09	0.39	2.47	0.63	0.22	0.35	0.00	0.14	0.33

Field Blank Wipes

	-										
For In Vit	tro 2% HN	IO ₃									
TC 8-4	0.043	0.045	0.242	12.9	4.16	9.60	0.084	0.402	0.003	0.157	0.055
TC 8-6	0.037	0.134	9.13	54.8	16.0	115	0.009	0.926	0.005	0.129	0.049
TC 8-7	0.037	0.144	0.2	60.4	20.3	126	0.006	0.857	0.005	0.133	0.058
Mean	0.04	0.11	3.19	42.7	13.5	83.7	0.03	0.73	0.004	0.1400	0.054
std dev	0.00	0.05	5.14	26.0	8.37	64.4	0.04	0.28	0.001	0.015	0.005
%RSD	8.9	50.6	161	60.8	62.0	77.0	134	39.1	26.6	10.8	8.5
Repeat E	Repeat Extract Analysis										
TC 8-6	0.034	0.146	9.27	55.0	15.8	115	0.009	0.926	0.005	0.153	0.048

For EPA 3050B 5% HNO₃

TC 8-4	1.72	9.68	9.75	246	109	189	1.40	7.75	0.15	19.6	5.960
TC 8-6	1.17	7.37	4.72	209	127	186	0.18	6.30	0.14	14.2	5.04
TC 8-7	1.35	665	7.84	371	138	173	3.01	7.12	2.63	1,170	358
Mean	1.42	227	7.44	276	125	183	1.53	7.06	0.97	401	123
std dev	0.28	379	2.54	85.1	14.9	8.79	1.42	0.73	1.43	666	203
%RSD	19.8	167	34.1	30.9	12.0	4.8	92.6	10.3	148	166	166

Table B-16. Relative Percent Differences (RPD) for the Analysis of Duplicate Aliquots of Tire Crumb Infill from Synthetic Fields and Playground Tire Crumb Samples (µg/g)

Fire Crumb Infill from Synthetic Fields and Playground Tire Crumb Samples (μg/g)							
Sample	Cr	Pb	Zn	As			
Synthetic Field Infill	Samples						
F4-L1-S1-A1	0.60	41.1	9,940	0.11			
F4-LI-S1-A2	0.25	10.7	5,320	0.08			
RPD	84.0	118	60.6	33.3			
		-					
F4-L1-D1-A1	0.35	24.8	4,880	NR ^a			
F4-L1-D1-A2	0.33	47.7	4,070	NR			
RPD	6.0	63.2	18.0				
F4-L2-S1-A1	0.14	13.6	2,660	NR			
F4-L2-S1-A2	0.37	19.3	4,310	NR			
RPD	88.9	34.4	47.6				
F4-L3-S1-A1	1.03	23.7	11,400	0.40			
F4-L3-S1-A2	0.98	20.0	8,190	0.28			
RPD	5.0	16.7	33.1	35.9			
		-					
F1-L1-S1-A1	1.01	29.2	17,200	0.55			
F1-L1-S1-A2	0.95	18.5	19,200	0.18			
RPD	6.2	44.9	10.9	102			
		1	1010				
F2-L1-S1-A1	0.35	20.6	5,690	0.23			
F2-L1-S1-A2	0.92	26.5	9,930	0.24			
RPD	90.2	25.3	54.4	3.9			
F2-L1-D1-A1	0.36	61.2	5,890	0.18			
F2-L1-D1-A2	0.24	36.0	3,120	0.22			
RPD	41.1	51.9	61.4	17.7			
F1-L3-S1-A1	0.54	20.6	7,930	0.29			
F1-L3-S1-A2	0.24	14.4	5,047	0.20			
RPD	76.0	35.2	44.4	38.1			
F1-L2-S1-A1	0.33	13.1	9,050	0.21			
F1-L2-S1-A2	0.54	34.7	8,541	0.25			
RPD	47.2	90.2	5.8	21.1			
F2-L3-S1-A1	NR	21.6	10,700	0.22			
F2-L3-S1-A2	NR	29.1	10,300	0.25			
RPD		29.6	3.5	11.4			
F2-L2-D1-A1	NR	36.4	10,300	0.44			
F2-L2-D1-A2	NR	27.5	10,700	0.24			
RPD		27.9	3.19	58.2			
F2-L2-S1-A1	NR	33.0	10,500	0.28			
F2-L2-S1-A2	NR	30.6	10,100	0.13			
RPD		7.7	3.6	74.7			
F2-L3-D1-A1	NR	43.7	10,200	0.20			
F2-L3-D1-A2	NR	22.4	12,300	0.26			
RPD		64.7	18.7	29.1			

Table B-16. Relative Percent Differences (RPD) for the Analysis of Duplicate Aliquots of Tire Crumb Infill from Synthetic Fields and Playground Tire Crumb Samples ($\mu g/g$) (cont'd.)

(00000	Cr	Pb	Zn	As
Playground Tire Cru	umb Samples		•	
P1-LA-TC1	0.52	2.43	9,720	0.15
P1-LA-TC2	1.66	46.3	11,100	0.96
RPD	104	180	13.7	146
P2-TC1	1.61	7.75	18,000	0.25
P2-TC2	2.97	3.42	12,100	0.28
RPD	59.5	77.5	39.4	10.1
P1-LA-TC4	0.72	6.31	6,730	0.28
P1-LA-TC5	0.76	4.64	8,250	0.59
RPD	5.8	30.4	20.3	72.1
P1-LB-TC1	0.76	443	17,500	15.0
P1-LB-TC2	0.26	0.99	6630	0.08
RPD	98.2	199	89.9	198

^aNR = Not reported.

Table B-17. Recommended Control Limits for In Vitro Soil Quality Control Samples According to EPA Method 9200.1-86

In Vitro QCs	Frequency	Control Limits	Corrective Actions
Reagent Blank	Once per batch	<25 μg/L Pb	Make new fluid and rerun all
	-		analyses.
Bottle Blank	5% ^a	<50 μg/L Pb	Check calibration and reanalyze
			as necessary.
Blank Spike (10 mg/L)	5% ^a	85%-115% recovery	Check calibration and/or source
		-	of contamination and reanalyze.
Matrix Spike (10 mg/L)	10% ^a	75%-125% recovery	Flag
Duplicate Sample	10% ^a	±20% RPDb	Flag
Control Soil (NIST 2710)	5% ^a	± 10% RPD°	Flag

^aMinimum 1 in 20.

bRPD = relative percent difference.

^cThe National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) RPD is based on certified values and mean RBA-Pb values of 75% for SRM 2710.

Table B-18. Summary Control Limit Results for In Vitro Pb Bioaccessibility Quality Control Samples (Note: This method has not been validated for the types of samples

collected in this scoping study.)

In Vitro QC Parameters	Analysis Frequency	Control Limit Results	Corrective Actions
Reagent Blank	9 Reagent blank (batches 1-9)	<5 μg/L Pb	None
Bottle Blank	9 Blank runs (batches 1-9)	<5 μg/L Pb	None
Blank Spike (10 mg/L)	8 Blank spike runs (batches 1-8)	90-105% recovery	None
Blank Ghost Wipe with Spike (10 mg/L)	3 Blank wipe with spike runs (batches 1, 8, 9)	87%-99% recovery	None
Blank Ghost Wipe with NIST SRM 2710 ^b	3 Wipes with SRM runs (batches 1, 8, 9)	3%-6% RPD°	None
NIST SRM 2710 ^b	6 SRM runs (batches 1-4, 6, 8)	0%-9% RPD°	None
Infill Spike (10 mg/L)	4 Infill spike runs (batches 1-4)	89%-104% recovery	None
Crumb Spike (10 mg/L)	3 Crumb spike runs (batches 1, 5, 9)	87%-103% recovery	None
Duplicate Samples	13 Pairs of infill samples and 4 pairs of crumb samples	2.7%-124% for infill samples and 4%-183% for crumb samples	Noted in report
Blank Ghost Wipe	3 Blank ghost wipe runs (batches 1, 8, 9)	<10 μg/wipe Pb	None

^aMinimum 1 in 20; matrix spikes for crumbs and infill only performed if enough material was available.

Table B-19. Percent Recovery Results for In Vitro Blank Spikes

Batch Number	Sample ID	Spiked Amount (mg/L)	Recovered In Vitro Extractable Pb (mg/L)	Percent Spike Recovery
1	Blank Spike	10	9.0	89.7
2	Blank Spike	10	9.9	99.5
3	Blank Spike	10	10.2	102
4	Blank Spike	10	10.2	102
5	Blank Spike	10	10.2	102
6	Blank Spike	10	10.5	105
7	Blank Spike	10	10.0	100
8	Blank Spike	10	10.1	101
Mean				100

^bThe National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) RPD is based on certified values and mean RBA-Pb values of 75% for SRM 2710.

^cRPD = relative percent difference.

Table B-20. Summary of Pb In Vitro Extractable Values for NIST SRM 2710

Batch Number	SRM Certified Concentration (ug/g)	Found Concentration (ug/g)	RPD
1	75.0	81.7	8.9
2	75.0	73.6	1.9
3	75.0	74.1	1.2
4	75.0	79.5	6.0
6	75.0	81.3	8.3
8	75.0	75.3	0.4
Mean	75.0	77.6	4.5

Table B-21. Summary of Pb In Vitro Extractable Values for SRM 2710 Spiked onto Ghost Wipe Media

Туре	Batch	SRM %IVBA ^a (based on EPA SOP)	Found (%IVBA)	RPD
SRM	1	75	79.7	6.22
SRM	8	75	78.3	4.39
SRM	9	75	77.6	3.45
Mean			78.5	4.69

^a In vitro bioaccessibility.

Table B-22. Results for In Vitro Pb Solution Spikes onto Blank Ghost Wipes and Tire Crumb Samples

Crumb Samples	Spiked Amount	Net Spiked In Vitro Extractable	Net Media In Vitro Extractable	Media Corrected	Percent
Media and Spike	(mg/L)	(mg/L)	(mg/L)	Spike	Recovery
Pb on Wipes	40	0.05		0.05	00.5
Ghost Wipe with Spike	10	8.65	0.00	8.65	86.5
Blank Ghost Wipe			0.00		
Ghost Wipe with Spike	10	9.93		9.93	99.3
Blank Ghost Wipe			0.01		
Ghost Wipe with Spike	10	9.66		9.66	96.6
Blank Ghost Wipe			0.00		
Pb on Tire Crumb Infill					
F4-L1-S1-A1 Spiked	10	8.86		8.86	88.6
F4-L1-S1-A1 Unspiked			0.01		
F4-L1-DS1-A1 Spiked	10	10.0		10.0	100
F4-L1-DS1-A1					
Unspiked			0.01		
F1-L3-S1-A1 Spiked	10	9.91		9.90	99.0
F1-L3-S1-A1 Unspiked			0.01		
F2-L2-DS1-A1 Spiked	10	10.5		10.5	105
F2-L2-DS1-A1					
Unspiked			0.02		
Pb on Tire Crumb					
P1-LA-TC1 Spiked	10	8.88		8.87	88.7
P1-LA-TC1 Unspiked			0.00		
P1-LA-TC4 Spiked	10	10.3		10.3	103
P1-LA-TC4 Unspiked			0.00		
P1-LB-TC1 Spiked	10	10.2		10.2	102
P1-LB-TC1 Unspiked			0.02		

Table B-23. In Vitro Pb Bioaccessibility Extraction Duplicates for Synthetic Turf Field Tire Crumb Infill

Sample	Pb (%IVBA) ^a
F4-L1-S1-A1	1.7
F4-L1-S1-A2	7.3
RPD	124
F4-L1-DS1-A1	3.9
F4-L1-DS1-A2	2.4
RPD	46.3
F4-L2-DS1-A1	3.8
F4-L2-DS1-A2	2.7
RPD	34.7
F4-L3-S1-A1	8.5
F4-L3-S1-A2	10.1
RPD	17.4
F1-L1-S1-A1	9.6
F1-L1-S1-A2	5.3
RPD	57.2
F2-L1-S1-A1	3.7
F2-L1-S1-A2	3.8
RPD	2.7
F2-L1-DS1-A1	1.7
F2-L1-DS1-A2	2.9
RPD	52.9

Sample	Pb (%IVBA)
F1-L3-S1-A1	4.2
F1-L3-S1-A2	4.4
RPD	4.6
F1-L2-S1-A1	5.0
F1-L2-S1-A2	1.6
RPD	105
F2-L3-S1-A1	5.0
F2-L3-S1-A2	4.2
RPD	17.9
F2-L2-DS1-A1	5.0
F2-L2-DS1-A2	4.5
RPD	11.3
F2-L2-S1-A1	7.6
F2-L2-S1-A2	3.6
RPD	72.3
F2-L3-DS1-A1	3.1
F2-L3-DS1-A2	5.5
RPD	54.7
Maximum RPD	124
Minimum RPD	2.7
Number	13

Table B-24. In Vitro Pb Bioaccessibility Extraction Duplicates for Playground Tire Crumb

	<i>n</i> = 100.000001					
Sample	Pb (%IVBA) ^a					
P1-LA-TC2	4.6					
P1-LA-TC3	0.3					
RPD	176					
P2-TC1	1.8					
P2-TC2	7.4					
RPD	122					
P1-LA-TC4	2.4					
P1-LA-TC5	5.2					

Sample	Pb (%IVBA)
P1-LB-TC1	0.3
P1-LB-TC2	6.4
RPD	183
Maximum RPD	183
Minimum RPD	73.2
Number	4

^a%IVBA = percent in vitro bioaccessibility.

^a%IVBA = percent in vitro bioaccessibility.

Appendix C

Compilation of Environmental Sample Analysis Results

- Table C-1. Results of Analysis for Grab VOC Air Samples
- Table C-2. Results of Analysis for Particle Mass on Integrated Air Samples
- Table C-3. Results of Analysis for Metals in Integrated Air Samples
- Table C-4. Results of SEM Analysis for Postulated Tire Crumb Particles on Integrated Air Samples
- Table C-5. Results of Analysis of Wet Wipe Samples for Total Extractable Metals
- Table C-6. Results of Analysis of Synthetic Turf Field Tire Crumb Infill for Total Extractable Metals
- Table C-7. Results of Analysis of Synthetic Turf Field Blades for Total Extractable Metals
- Table C-8. Results of Analysis of Playground Tire Crumb for Total Extractable Metals
- Table C-9. Results of Analysis of Synthetic Turf Field Infill Sample Analysis for Bioaccessible Pb
- Table C-10. Results of Analysis of Synthetic Turf Field Blade Sample Analysis for Bioaccessible Ph
- Table C-11. Results of Analysis of Playground Tire Crumb Sample Analysis for Bioaccessible Pb

voc	Site ^a	Sa	pp ampling Loc	bV cation at Sit	e ^b	Sa	μ g , ampling Loc	/m³ cation at Sit	e ^b
		Α	В	С	D	Α	В	С	D
Benzene	F1D1	0.090	0.110	0.074	0.073	0.287	0.351	0.223	0.223
	F1D2	0.081	0.081	0.088	0.092	0.255	0.255	0.287	0.287
	F2	0.098	0.120	0.120	0.120	0.319	0.383	0.383	0.383
	F4	0.320	0.120	0.160	0.120	1.021	0.383	0.510	0.383
	P1	0.088	0.086	_c	0.087	0.287	0.287	_	0.287
Toluene	F1D1	0.140	0.98	0.140	0.150	0.527	3.688	0.527	0.564
	F1D2	0.120	0.110	0.110	0.120	0.452	0.414	0.414	0.452
	F2	0.180	0.180	0.190	0.190	0.677	0.677	0.715	0.715
	F4	0.510	0.190	0.150	0.190	1.919	0.715	0.564	0.715
	P1	0.140	0.430	_	0.160	0.527	1.618	_	0.602
m&p-Xylenes	F1D1	ND ^d	0.170	ND	0.083	ND	0.737	ND	0.347
	F1D2	0.075	0.130	ND	ND	0.303	0.564	ND	ND
	F2	0.047	0.074	0.091	0.077	0.217	0.303	0.390	0.347
	F4	0.290	0.055	0.077	ND	1.257	0.217	0.347	ND
	P1	ND	0.130	_	0.050	ND	0.564	_	0.217
o-Xylene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	0.120	ND	ND	ND	0.520	ND	ND	ND
	P1	ND	0.045	_	ND	ND	0.173	_	ND
Ethylbenzene	F1D1	ND	0.073	ND	ND	ND	0.303	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	0.110	ND	ND	ND	0.477	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,2,4-Trimethylbenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	0.082	ND	ND	ND	0.393	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
4-Ethyltoluene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	0.082	ND	ND	ND	0.393	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Hexane	F1D1	ND	0.320	0.092	0.063	ND	1.126	0.317	0.211
	F1D2	0.098	0.062	ND	0.078	0.352	0.211	ND	0.281
	F2	ND	0.049	0.110	0.045	ND	0.176	0.387	0.141
	F4	0.150	0.110	0.160	0.049	0.528	0.387	0.563	0.176
	P1	ND	0.300	_	ND	ND	1.055	_	ND

Voc	C:4-a			bV	b	μg/m ³ Sampling Location at Site ^b				
VOC	Site	A	B	cation at Sit	e D	A	B	Cation at Si	D D	
Cyclohexane	F1D1	ND	0.330	ND ND	ND	ND	1.134	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND ND	
	P1	ND ND	0.250	IND _	ND	ND	0.859	IND _	ND ND	
Methyl Ethyl Ketone	F1D1	0.400	0.560	0.440	0.440	1.180	1.651	1.298	1.298	
	F1D2	0.490	0.340	0.320	0.360	1.445	1.003	0.94	1.062	
	F2	0.400	0.440	0.380	0.370	1.180	1.298	1.121	1.091	
	F4	0.400	0.500	0.380	0.440	1.091	1.474	1.209	1.298	
	P1	0.340	0.480	0.410	0.380	1.003	1.415	1.209	1.121	
Methyl Isobutyl Ketone	F1D1	0.088	0.480	0.180	ND	0.368	0.450	0.736	ND	
	F1D2	0.088	0.110	ND	ND ND	0.308	0.450	0.730 ND	ND	
	F2	0.120 ND	ND	ND ND	ND ND	ND	0.450 ND	ND ND	ND	
	F4	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND	
	P1	ND ND	ND ND	IND	ND ND	ND ND	ND ND	IND	ND	
2-Hexanone	F1D1	ND ND	ND ND	ND	ND ND	ND ND	ND ND	ND	ND	
	F1D2	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
	F2	ND ND	0.046	ND ND	ND ND	ND ND	0.204	ND ND	ND	
	F4	ND ND	ND	ND ND	ND ND	ND ND	0.204 ND	ND ND	ND	
	P1			IND				IND		
Carbon Tetrachloride		ND 0.000	ND 0.002	0.004	ND 0.007	ND 0.566	ND 0.570	0.500	ND 0.610	
	F1D1	0.090	0.092	0.084	0.097	0.566	0.579	0.528	0.610	
	F1D2	0.097	0.100	0.093	0.098	0.610	0.629	0.585	0.616	
	F2	0.088	0.100	0.092	0.084	0.554	0.629	0.579	0.528	
	F4 P1	0.088	0.089	0.086	0.098	0.554	0.560	0.541	0.616	
Dichlorodifluoromethane		0.083	0.082	0.500	0.094	0.522	0.516	2.472	0.0.591	
	F1D1	0.519	0.540	0.500	0.550	2.566	2.670	2.472	2.720	
	F1D2 F2	0.490 0.500	0.530 0.640	0.470 0.540	0.560 0.510	2.423 2.472	2.621 3.165	2.324 2.670	2.769 2.522	
	F4	0.300	0.480	0.490	0.540	2.324			2.670	
	P1	0.470	0.460	0.490			2.373	2.423		
Methylchloride				0.470	0.540	2.571	2.670	0.07	2.670 0.99	
•	F1D1 F1D2	0.430 0.460	0.500 0.470	0.470 0.470	0.480 0.460	0.888	1.033 0.97	0.97 0.97	0.95	
	F2	0.420	0.460	0.470	0.450	0.95	0.97	0.971	0.93	
	F4	0.420	0.460	0.470	0.430	0.867		0.97	1.074	
	P1	0.470	0.500	0.470	0.520	0.97	1.033 0.93	0.91	0.93	
Trichlorofluoromethane	F1D1		0.430	0.260		1.405		1.461		
	F1D1	0.250	0.260		0.280		1.573		1.573	
		0.260		0.250	0.270	1.461	1.461	1.405	1.517	
	F2	0.250	0.290	0.270	0.250	1.405	1.630	1.517	1.405	
	F4	0.240	0.240	0.240	0.300	1.349	1.349	1.349	1.686	

Table C-1. Results			pp	bV	-		μg	/m ³	_ b
VOC	Site			ation at Sit				cation at Sit	
Trichlorotrifluoroethane	F4D4	Α	B	C 0.075	D 004	Α	B 0.040	C . 507	D 0.010
	F1D1	0.080	0.081	0.075	0.081	0.613	0.613	0.537	0.613
	F1D2	0.077	0.079	0.076	0.084	0.613	0.613	0.613	0.613
	F2	0.073	0.085	0.075	0.072	0.537	0.613	0.537	0.537
	F4	0.069	0.072	0.072	0.150	0.537	0.537	0.537	1.150
Methylene Chloride	P1	0.074	0.074		0.074	0.537	0.537		0.537
,	F1D1	ND	0.074	0.062	0.062	ND	0.243	0.208	0.208
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	0.058	0.069	0.062	0.059	0.208	0.243	0.208	0.208
	F4	0.055	0.055	0.065	0.061	0.208	0.174	0.208	0.208
Chloroform	P1	0.071	0.110	- ND	0.066	0.243	0.382		0.243
- Critici Critici	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	0.062	ND 	ND	ND	0.293	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1-Trichloroethane	P1	ND	ND		ND	ND	ND		ND
1,1,1 Thomoroculano	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-	P1	ND	ND	_	ND	ND	ND		ND
Tetrachloroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	P1	ND	ND	_	ND	ND	ND		ND
1,1,2-Trichioroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
A A D'ablana tha a a	P1	ND	ND	_	ND	ND	ND		ND
1,1-Dichloroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
4.4.5:11	P1	ND	ND	_	ND	ND	ND	_	ND
1,1-Dichloroethylene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
101T:11	P1	ND	ND	_	ND	ND	ND		ND
1,2,4-Trichlorobenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND

Table C-1. Results		, 0.0 .0.		bV	μg/m³				
VOC	Site ^a	Sa	ampling Lo	cation at Sit	e ^b	S	ampling Lo	cation at Sit	e ^b
		Α	В	С	D	Α	В	С	D
1,2-Dibromoethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,2-Dichlorobenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,2-Dichloroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,2-Dichloropropane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,3,5-Trimethylbenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,3-Butadiene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,3-Dichlorobenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
1,4-Dichlorobenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Acrylonitrile	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND		ND	ND	ND	_	ND

voc	Site ^a	S	pp ampling Loc	bV cation at Sit	e ^b	s	μg, ampling Loc	/m ³ cation at Sit	e ^b
		Α	В	С	D	Α	В	С	D
Allyl Chloride	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Benzylchloride	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Bromodichloromethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Bromoform	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
cis-1,2-Dichloroethylene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
cis-1,3-	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
Dichloropropylene	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Chlorobenzene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Chloroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
	F4	ND	ND	ND	ND	ND	ND	ND	ND
	P1	ND	ND	_	ND	ND	ND	_	ND
Dibromochloromethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND
	F2	ND	ND	ND	ND	ND	ND	ND	ND
		110	1 110	110	110				שוו
	F4	ND	ND	ND	ND	ND	ND	ND	ND

voc	Site ^a	ppbV Sampling Location at Site ^b				μg/m ³ Sampling Location at Site ^b				
		Α	В	С	D	Α	В	С	D	
Dichlorotetrafluoroethane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Heptane	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Hexachloro-1,3-	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
butadiene	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Methylbromide	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Methyl-t-Butyl Ether	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Styrene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
trans-1,2-	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
Dichloroethylene	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
trans-1,3-	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
Dichloropropylene	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Tetrachloroethylene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	

voc	Site	Sa		bV cation at Sit	e ^b	μg/m³ Sampling Location at Site ^b				
		Α	В	С	D	Α	В	С	D	
Tetrahydrofuran	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Trichloroethylene	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Vinyl Bromide	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
	P1	ND	ND	_	ND	ND	ND	_	ND	
Vinylchloride	F1D1	ND	ND	ND	ND	ND	ND	ND	ND	
	F1D2	ND	ND	ND	ND	ND	ND	ND	ND	
	F2	ND	ND	ND	ND	ND	ND	ND	ND	
	F4	ND	ND	ND	ND	ND	ND	ND	ND	
O'ta ida e'faa faa D	P1	ND	ND	_	ND	ND	ND	_	ND	

a Site identification: P = playground; F = synthetic turf field; D = day 1 or day 2.
b Sampling location: A, B, and C are "on field" or "on playground"; D is "upwind background."
c Only two "on playground" sampling locations.
d ND = not detected.

Table C-2. Results of Analysis for Particle Mass on Integrated Air Samples (µg/m³)

		Sampling Location ^b									
Site ^a	Α	A Dup	В	B Dup	С	D	Field Blank				
F1D1	27.5	c	28.6	29.0	27.4	29.5	0.6				
F1D2	29.6	_	30.0	_	_	29.5	0.4				
F4	33.4	31.0	30.9	_	31.0	28.6	0.7				
P1	23.9	24.3	29.5	_	_	14.2	0.2				

Table C-3. Results of Analysis for Metals in Integrated Air Samples (ng/m³)a

Table C-3. Results of Analysis for Metals in Integrated Air Samples (ng/m)												
Site and Sampling Location at the Site ^b	Pb	Cr	Zn	Ca	CI	Cu	Fe	К	Mn	S	Si	Ti
	PD	_									-	
F1D1 location A	_c	2.3	6.6	449	14.1	8.9	230	134	6.1	4,004	1,291	20.4
F1D1 location B	_	_	15.3	230	_	_	201	91.7	_	3,688	555	30.8
F1D1 location B duplicate	_	2.8	13.6	370	_	_	199	99.2	6.3	3,018	1,497	22.3
F1D1 location C	_	3.6	10.6	349		_	226	126	_	3,946	796	24.7
F1D1 location D	_	2.0	23.8	396	_	_	240	128	7.5	3,667	1,405	25.2
F1D1 field blank	_		_	34.5	12.2	_		8.6		_		-
F1D2 location A	7.7	4.3	17.0	834	21.0	10.3	364	164	9.3	3,680	1,248	29.4
F1D2 location B	_	2.8	6.6	812	_	_	364	158	10.6	3,784	858	27.9
F1D2 location C	NR ^d	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
F1D2 location D	6.3	3.3	11.6	881	20.9	_	356	178	8.4	3,933	1,548	31.9
F1D2 field blank	_		9.5	_	_	_		_	4.3	_		-
F4 location A	_	3.4	36.0	487	25.5	21.3	803	302	18.5	3,044	2,646	58.7
F4 location A duplicate	_	5.0	31.9	303	_	20.0	559	191	13.0	1,516	2,116	44.0
F4 location B	_	_	33.0	338	_	13.4	663	228	13.4	2,845	1,912	58.2
F4 location C	_	_	25.2	319	_	12.2	573	238	10.9	2,952	1,942	51.4
F4 location D	_	2.7	21.7	466	21.1	12.3	456	227	10.9	2,976	1,890	41.2
F4 field blank	_	_	_	12.9	_	_	_	_	_	_	_	
P1 location A	5.1	3.6	90.7	317	118	11.8	672	196	8.6	664	2,784	57.6
P1 location A duplicate	_	2.5	82.5	365	177	_	748	240	13.6	882	2,435	63.7
P1 location B	_	3.1	117	414	189		987	312	15.0	903	3,455	92.3
P1 location D	_	_	10.5	238	95.5	10.8	294	97.2	_	751	516	21.4
P1 field blank	_	_	_	_	_	_			_	_	_	_

^aF = turf field, P = playground, D = day, Dup = duplicate sample. ^bA, B, and C are "on field" or "on playground," D is "upwind background."

^cNot collected.

^aValues represent those less than three times the measurement uncertainty limit following field blank corrections.

^bF = turf field, P = playground, D = day, Dup = duplicate sample. ^bDesignations A, B, and C refer to "on field" or "on playground" locations; D locations represent background sites.

^c— Represents values less than three times the measurement uncertainty limit. ^dNR = sample filter inverted during collection; analysis results not reported.

Table C-4. Results of SEM Analysis for Postulated Tire Crumb Particles on Integrated Air

Samples

Sample ID ^b	Number of Postulated Tire Crumb Particles ^c	Mass of Postulated Tire Crumb Particles ^c (pg)	Scaled Mass of Postulated Tire Crumb Particles ^d (µg)	Estimated Concentration of Postulated Tire Crumb Particles (µg/m³)
F1D1 location A	5	6	0.01	0.001
F1D1 location A duplicate	15	80	0.02	0.004
F1D1 location A dup. Repeat	5	65	0.03	0.007
F1D1 location B	18	90	0.08	0.019
F1D1 location D	7	16	0.01	0.003
F4 location A	0	0	0.00	0.000
F4 location A duplicate	4	19	0.01	0.003
F4 location B	6	47	0.03	0.007
F4 location D	6	15	0.01	0.002
P1 location A	63 ^e	3,330	3.9	0.77
P1 location B	58 ^f	2,530	2.1	0.42
P1 location D	5	40	0.03	0.005

^aGiven the lack of unique composition and morphology for tire crumb particles from the collected materials, the estimates in this table have considerable uncertainty.

^bF= synthetic turf field; P = playground; A and B are "on field" or "on playground;" D = "upwind background."

^cRaw numbers, not normalized to the same analyzed area. (CCSEM areas analyzed ranged from 0.6 mm² to 3.6

 mm^2).

^dEstimated tire crumb mass scaled to total exposed filter area of 6.7 cm².

^eMass median diameter ~2.6 μm.

fMass median diameter ~2.2 µm.

Table C-5. Results of Analysis of Wet Wipe Samples for Total Extractable Metals ($\mu g/ft^2$; based on total extractable amounts from consecutive in vitro and Method 3050B

extractions)

Site and Locatio	d Sample n ^a	Pb	Cr	Zn	Al	As	Ва	Cd	Cu	Fe	Mn	Ni
F1D1	Loc. A, S	0.500	0.112	35.9	19.5	0.051	NR	<mdl<sup>b</mdl<sup>	2.97	77.1	0.738	0.241
F1D1	Loc. A, DS	0.489	0.178	43.3	26.3	0.055	0.351	<mdl< td=""><td>3.59</td><td>104</td><td>0.923</td><td>0.123</td></mdl<>	3.59	104	0.923	0.123
F1D1	Loc. B, S ^c	1.46	0.413	34.4	27.0	0.062	0.083	<mdl< td=""><td>2.77</td><td>91.0</td><td>1.09</td><td>0.221</td></mdl<>	2.77	91.0	1.09	0.221
F1D1	Loc. B, DS ^c	1.91	0.517	38.4	22.5	0.102	NR	0.022	2.61	81.3	1.13	0.184
F1D1	Loc. C, S	0.347	0.214	30.9	18.6	0.033	5.05	<mdl< td=""><td>2.06</td><td>74.2</td><td>0.690</td><td>0.149</td></mdl<>	2.06	74.2	0.690	0.149
F1D1	Loc. C, DS	0.323	0.077	21.3	17.3	0.028	NR	<mdl< td=""><td>1.69</td><td>71.5</td><td>0.598</td><td>0.177</td></mdl<>	1.69	71.5	0.598	0.177
F1D2	Loc. A, S	0.370	0.096	26.4	17.6	0.045	NR	<mdl< td=""><td>2.05</td><td>68.6</td><td>0.741</td><td>0.065</td></mdl<>	2.05	68.6	0.741	0.065
F1D2	Loc. A, DS	0.407	0.197	34.9	21.1	0.039	NR	<mdl< td=""><td>2.68</td><td>87.7</td><td>0.882</td><td>0.122</td></mdl<>	2.68	87.7	0.882	0.122
F1D2	Loc. B, S ^c	0.731	0.278	37.4	30.4	0.050	0.387	0.008	2.79	100	1.02	0.165
F1D2	Loc. B, DS ^c	1.39	NR ^d	29.1	28.9	0.058	0.564	NR	2.20	87.3	0.356	NR
F1D2	Loc. C, S	0.688	NR	40.6	30.0	0.049	0.280	NR	2.69	91.5	NR	NR
F1D2	Loc. C, DS	0.346	NR	25.7	18.2	0.045	NR	NR	1.92	75.6	NR	NR
F2	Loc. A, S	0.456	NR	19.3	52.1	0.049	0.125	NR	0.677	116	NR	NR
F2	Loc. B, S	0.280	NR	13.0	28.0	0.026	7.31	<mdl< td=""><td>0.549</td><td>78.9</td><td>NR</td><td>NR</td></mdl<>	0.549	78.9	NR	NR
F2	Loc. C, S	0.289	NR	9.24	33.3	0.024	1.16	NR	0.488	74.8	NR	NR
F4	Loc. A, S	0.177	NR	5.28	42.7	0.022	0.085	NR	0.326	61.3	NR	NR
F4	Loc. A, DS	0.139	NR	4.25	36.1	0.018	0.384	<mdl< td=""><td>0.280</td><td>49.5</td><td>NR</td><td>NR</td></mdl<>	0.280	49.5	NR	NR
F5	Loc. B, S	0.129	NR	6.29	19.6	0.020	6.10	NR	0.203	28.5	NR	NR
F6	Loc. C, S	0.184	0.245	13.6	16.4	0.027	5.94	0.006	0.305	33.7	0.520	NR
F1D1	Field Blank	0.181	0.096	1.79	0.17	0.034	NR	0.033	0.057	2.28	0.253	0.067
F4	Field Blank	0.541	0.046	3.17	1.25	0.021	5.60	<mdl< td=""><td>0.051</td><td>2.15</td><td>0.124</td><td>0.046</td></mdl<>	0.051	2.15	0.124	0.046
F4	Field Blank	0.135	16.0	7.36	1.76	0.025	5.84	0.068	0.067	62.1	27.1	8.54

^aF = synthetic turf field; D = day 1 or day 2; Loc. = "on field" sampling location at site, S = sample collected at the location, DS = duplicate sample collected immediately adjacent to the sample.

b<MDL = extract concentration less than the method detection limit in both in vitro and Method 3050B analyses.

^cLocation B wipe samples from red synthetic turf in end zone.

^dNR = not reported; analytical result <0 after blank correction.

Table C-6. Results of Analysis of Synthetic Turf Field Tire Crumb Infill for Total Extractable Metals (µg/g; based on total extractable amounts from consecutive in vitro

and Method 3050B extractions)

	Viethod 3050 Sampling	Бехпа	Clions)									
Loca	tion, Sample,	DL	C	7		A -	Da	Cd	C	F	Man	Ni
F1	Aliquot ^a L1, S1, A1	Pb 29.2	Cr 1.01	Zn 17,200	AI 218	As 0.55	Ba 68.4	0.87	Cu 23.7	Fe 416	Mn 4.54	8.95
F1	L1, S1, A2	18.5	0.95	19,200	233	0.18	7.01	0.97	24.0	535	13.6	7.55
F1	L2, S1, A1	13.1	0.33	9.050	168	0.21	NR	0.79	8.73	196	3.16	2.50
F1	L2, S1, A1	34.7	0.54	8,540	167	0.25	NR	0.73	6.54	168	3.25	2.04
F1	L3, S1, A1	20.6	0.54	7,930	170	0.29	NR	1.05	7.96	241	3.94	3.52
F1	L3, S1, A2	14.4	0.24	5,050	171	0.20	NR	0.69	2.64	150	3.48	2.44
F2	L1, S1, A1	20.6	0.24	5,690	345	0.23	NR	0.35	3.46	215	4.66	0.78
F2	L1, S1, A2	26.5	0.92	9,930	533	0.24	56.0	0.60	10.7	476	3.85	1.95
F2	L1, DS1, A1	61.2	0.36	5,890	473	0.18	37.5	0.55	2.63	209	3.71	0.82
F2	L1, DS1, A1	36.0	0.24	3,120	376	0.10	5.67	0.68	1.18	134	3.36	0.70
F2	L2, S1, A1	33.0	NR ^b	10,500	347	0.28	35.8	0.46	13.6	228	NR	NR
F2	L2, S1, A1	30.6	NR	10,100	330	0.20	18.3	0.40	15.8	210	NR	NR
F2	L2, DS1, A1	36.4	NR	10,100	289	0.13	26.4	1.05	16.8	429	NR	NR
F2	L2, DS1, A1		NR		309					227		NR
		27.5		10,700		0.24	12.3	0.47	14.1		NR	
F2	L3, S1, A1	21.6	NR	10,700	368	0.22	44.7	0.47	14.4	247	NR	NR
F2	L3, S1, A2	29.1	NR	10,300	386	0.25	43.5	0.45	14.2	283	NR	NR
F2	L3, DS1, A1	43.7	NR	10,200	426	0.20	98.5	0.52	14.6	239	NR	NR
F2	L3, DS1, A2	22.4	NR	12,300	373	0.26	76.6	0.68	17.2	269	NR	NR
F4	L1, S1, A1	41.1	0.60	9,940	307	0.11	21.0	0.29	7.32	160	2.27	0.94
F4	L1, S1, A2	10.7	0.25	5,320	290	0.08	15.7	0.30	1.54	125	1.59	0.37
F4	L1, DS1, A1	24.8	0.35	4,880	328	-0.03	71.1	<mdl< td=""><td>2.47</td><td>232</td><td>3.92</td><td><mdl< td=""></mdl<></td></mdl<>	2.47	232	3.92	<mdl< td=""></mdl<>
F4	L1, DS1, A2	47.7	0.33	4,070	268	<mdl< td=""><td>39.2</td><td><mdl< td=""><td>2.13</td><td>154</td><td>3.82</td><td><mdl< td=""></mdl<></td></mdl<></td></mdl<>	39.2	<mdl< td=""><td>2.13</td><td>154</td><td>3.82</td><td><mdl< td=""></mdl<></td></mdl<>	2.13	154	3.82	<mdl< td=""></mdl<>
F5	L1, S1, A1	13.6	<mdl<sup>c</mdl<sup>	2,660	201	<mdl< td=""><td>18.7</td><td><mdl< td=""><td>1.01</td><td>104</td><td>4.01</td><td><mdl< td=""></mdl<></td></mdl<></td></mdl<>	18.7	<mdl< td=""><td>1.01</td><td>104</td><td>4.01</td><td><mdl< td=""></mdl<></td></mdl<>	1.01	104	4.01	<mdl< td=""></mdl<>
F5	L1, S1, A2	19.3	<mdl< td=""><td>4,310</td><td>269</td><td><mdl< td=""><td>32.1</td><td><mdl< td=""><td>2.57</td><td>111</td><td>2.79</td><td><mdl< td=""></mdl<></td></mdl<></td></mdl<></td></mdl<>	4,310	269	<mdl< td=""><td>32.1</td><td><mdl< td=""><td>2.57</td><td>111</td><td>2.79</td><td><mdl< td=""></mdl<></td></mdl<></td></mdl<>	32.1	<mdl< td=""><td>2.57</td><td>111</td><td>2.79</td><td><mdl< td=""></mdl<></td></mdl<>	2.57	111	2.79	<mdl< td=""></mdl<>
F6	L1, S1, A1	23.7	1.03	11,400	456	0.40	42.6	1.55	13.6	539	7.91	2.17
F6	L1, S1, A2	20.0	0.98	8,190	555	0.28	33.9	1.36	8.5	745	8.73	1.41

^aF = synthetic turf field; L = site sampling location; S = sample collected at the location; DS = duplicate sample collected at the location; A = aliquot of tire crumb infill from sample (~1 g each).

^bNR = not reported; analytical result <0 after blank correction.

^c<MDL = extract concentration less than the method detection limit in both in vitro and Method 3050B analyses.

Table C-7. Results of Analysis of Synthetic Turf Blades for Total Extractable Metals (µg/g; based on total extractable amounts from consecutive in vitro and Method 3050B extractions)

Site a	and e Color ^a	Pb	Cr	Zn	Al	As	Ва	Cd	Cu	Fe	Mn	Ni
F1	Red	389	73.1	351	1,090	0.40	141	0.16	5.83	738	7.70	1.96
F1	Green	3.84	9.71	316	2,090	0.47	88	0.07	44.8	2,060	9.41	3.22
F1	White	4.28	0.99	688	1,320	0.60	114	NR ^b	7.42	721	6.83	3.98
F1	Black	2.76	1.91	729	1,290	0.29	111	NR	5.91	787	6.02	1.00
F3	Red	2.40	1.20	199	947	0.22	1,950	NR	3.19	449	8.35	0.10
F3	White	1.97	0.08	255	336	NR	38	NR	1.54	138	3.36	1.66
F4	Green, white, yellow mix	2.08	3.72	206	2,120	0.25	50	NR	74.1	4,950	12.07	2.94
F5	Green, white, yellow mix ^c	701	177	131	1,620	0.12	40	<mdl<sup>d</mdl<sup>	68.3	3,300	6.05	2.43
F6	Green, white, yellow mix	77.1	18.9	175	1,150	0.05	303	NR	34.9	3,230	4.94	14.3

^aF = synthetic turf fields.

Table C-8. Results of Analysis of Playground Tire Crumb for Total Extractable Metals (µg/g; based on total extractable amounts from consecutive in vitro and Method 3050B extractions)

Locat	Sampling tion, and Tire b Piece ^a	Pb	Cr	Zn	Al	As	Ва	Cd	Cu	Fe	Mn	Ni
P1	Loc. A, TC 1	0.99	0.28	4,330	96.4	0.04	5.70	0.09	0.05	37.2	0.47	0.24
P1	Loc. A, TC 2	2.43	0.52	9,717	92.3	0.15	0.72	0.34	1.02	82.3	1.29	0.25
P1	Loc. A, TC 3	46.3	1.66	11,100	372	0.96	18.9	6.14	1.82	384	4.58	1.60
P1	Loc. A, TC 4	6.31	0.72	6,730	138	0.28	3.75	0.84	2.83	154	2.81	0.62
P1	Loc. A, TC 5	4.64	0.76	8,250	799	0.59	3.46	0.11	1.66	274	1.86	2.09
P1	Loc. B, TC 1	443	0.76	17,500	350	15.0	13.3	10.5	3.74	320	8.83	2.48
P1	Loc. B, TC 2	0.99	0.26	6,630	44.3	0.08	1.89	0.05	1.26	57.4	2.24	0.79
P2	TC 1	7.75	1.61	18,000	126	0.25	8.46	0.21	2.74	1,900	7.76	1.69
P2	TC 2	3.42	2.97	12,100	170	0.28	11.0	0.67	4.98	6,180	4.27	1.11

^aP = playground; Loc. = sampling location; TC = different pieces of tire crumb from two sampling locations at site P1 were analyzed; two pieces of tire crumb from site P2 were analyzed.

^bNR = not reported; analytical result <0 after blank correction.

^cBlade samples collected from a field with an apparently patched area. Also, the Method 3050B extract for this sample spilled during processing.

d<MDL = extract concentration less than the method detection limit in both in-vitro and Method 3050B analyses.

Table C-9. Results of Analysis of Synthetic Turf Field Infill Sample Analysis for **Bioaccessible Pb**

Site, Sampli a	ing Location, Sample, nd Aliquot ^a	Total Extractable Pb (μg/g)	Pb In Vitro Bioaccessibility Value (%) ^b
F1	L1, S1, A1	29.2	9.6
F1	L1, S1, A2	18.5	5.3
F1	L2, S1, A1	13.1	5.0
F1	L2, S1, A2	34.7	1.6
F1	L3, S1, A1	20.6	4.2
F1	L3, S1, A2	14.4	4.4
F2	L1, S1, A1	20.6	3.7
F2	L1, S1, A2	26.5	3.8
F2	L1, DS1, A1	61.2	1.7
F2	L1, DS1, A2	36.0	2.9
F2	L2, S1, A1	33.0	7.6
F2	L2, S1, A2	30.6	3.6
F2	L2, DS1, A1	36.4	5.0
F2	L2, DS1, A2	27.5	4.5
F2	L3, S1, A1	21.6	5.0
F2	L3, S1, A2	29.1	4.2
F2	L3, DS1, A1	43.7	3.1
F2	L3, DS1, A2	22.4	5.5
F4	L1, S1, A1	41.1	1.7
F4	L1, S1, A2	10.7	7.3
F4	L1, DS1, A1	24.8	3.9
F4	L1, DS1, A2	47.7	2.4
F5	L1, S1, A1	13.6	3.8
F5	L1, S1, A2	19.3	2.7
F6	L1, S1, A1	23.7	8.5
F6	L1, S1, A2	20.0	10.1
Mean			4.7 ± 2.3
Minimum			1.6
Maximum			10.1

^aL = sampling location at site, S = sample collected at the location, DS = duplicate sample collected at the location, A = aliquot of tire crumb infill from sample (~1 g each).

^bThe in vitro bioaccessibility values were determined by dividing the amount of Pb extracted in the in vitro extraction

by the total extractable amount of Pb.

Table C-10. Results of Analysis of Synthetic Turf Field Blade Sample Analysis for **Bioaccessible Pb**

Site	a and Blade Color	Total Extractable Pb (µg/g)	Pb In Vitro Bioaccessibility Value (%) ^a
F1	Red	389	2.3
F1	Green	3.84	40.9
F1	White	4.28	43.0
F1	Black	2.76	86.8
F3	Red	2.40	40.3
F3	White	1.97	38.7
F4	Green, white, yellow mix	2.08	54.4
F5	Green, white, yellow mix	701	0.2
F6	Green, white, yellow mix	77.1	1.0
Mean			34.2 ± 28.8
Minimum			0.2
Maximum			86.8

^a F = field site.

Table C-11. Results of Analysis of Playground Tire Crumb Sample Analysis for **Bioaccessible Pb**

	ng Location, and umb Piece ^a	Total Extractable Pb (μg/g)	Pb In Vitro Bioaccessibility Value (%) ^b
P1	Loc. A, TC1	0.99	10.7
P1	Loc. A, TC2	2.43	4.6
P1	Loc. A, TC3	46.3	0.3
P1	Loc. A, TC4	6.31	2.4
P1	Loc. A, TC5	4.64	5.2
P1	Loc. B, TC1	443	0.3
P1	Loc. B, TC2	0.99	6.4
P2	TC1	7.75	1.8
P2	TC2	3.42	7.4
Mean			4.3 ± 3.5
Minimum			0.3
Maximum			10.7

^bThe in vitro bioaccessibility values were determined by dividing the amount of Pb extracted in the in vitro extraction by the total extractable amount of Pb.

^aL = sampling location at site, TC = tire crumb piece analyzed from the location.

^bThe in vitro bioaccessibility values were determined by dividing the amount of Pb extracted in the in vitro extraction by the total extractable amount of Pb.

Appendix D

Air PM₁₀ SEM Analysis Report

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

NATIONAL EXPOSURE RESEARCH LABORATORY Human Exposure and Atmospheric Sciences Division Research Triangle Park, NC 27711

SEM Analysis of Tire Crumb Samples

Bob Willis February 23, 2009

Sample Collection

PM $_{10}$ samples were collected by the EPA on playgrounds and turf fields that used tire crumb (TC) for a base. Samples were collected at two synthetic turf fields in EPA Regions 4 and 5 and at one playground in EPA Region 3. Samples were collected on 37mm polycarbonate filters (0.4 μ m pore) using a Harvard Impactor employing a 10 μ m inlet. Samples were collected for approximately 400 min at a flow rate of 10 lpm, giving a total sampled volume of about 4 m 3 . Duplicate samples were collected to assess precision and background samples were collected for comparison from nearby playgrounds/turf fields that did not use tire crumbs.

In addition to ambient samples collected on the playgrounds and turf fields, TC particles collected from the field's crumb base were provided from each of the three sites to provide "source profiles" to assist in identifying TC-related particles in the ambient samples.

Sample Preparation

Source samples: Individual "crumbs" from the bulk sample, typically a couple of mm in size, were deposited "as is" on a sticky carbon tab. Source particles closer in size to the ambient sample were generated by shaving pieces from larger crumbs using a stainless steel razor blade. Source samples were coated with ~200 Å film of conductive carbon to minimize charge build-up on the sample during SEM analysis.

*Air PM*₁₀ samples: 5 mm x 5mm sections were cut from each polycarbonate filter using a stainless steel scalpel. Each section was affixed to a standard 12-mm aluminum specimen stub using a double-sided sticky carbon tab. The samples were then coated with ~200 Å of carbon to minimize sample charging by the electron beam during SEM analysis.

Sample Analysis

Samples were analyzed by Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectrometry (EDS) using the Personal SEM (R.J. Lee Instruments Ltd.) in the NERL Electron Microscopy Laboratory. Manual SEM/EDX analysis was first conducted on the bulk TC source samples provided. Chemistry and morphological features characteristic of the TC material were

identified to help identify TC particles in the ambient samples. Ambient samples were analyzed by computer-controlled SEM (CCSEM/EDX). Instrument parameters for the CCSEM analyses included: 20 kV accelerating voltage, Backscattered electron (BSE) imaging mode, 16 mm working distance, zero tilt. The BSE mode yields a more uniform background than the secondary electron (SE) mode, necessary for computer-controlled SEM, but at the expense of some loss in sensitivity for small carbonaceous particles: carbonaceous TC particles about 1 micron or smaller can be difficult to distinguish from the polycarbonate filter substrate in CCSEM analyses. Thus, small carbonaceous particles may be under-reported in these analyses.

The CCSEM analysis was set up to analyze particles with average diameters between 1 and $20~\mu m$. Few particles >10 μm , however, were observed in any sample. All particles within this size range were automatically sized and analyzed by EDS for chemistry. Based on the analyses of the tire crumb source samples, Sulfur, Zinc, and Carbon were identified as possible indicators of TC material. Rules were developed to optimize the search for TC-like particles by extending the X-ray analysis time (10 s) and saving low-resolution images for all particles containing S, Zn, or C. Images and spectra for these particle types were manually reviewed off-line and particles were subjectively judged o be either TC-like, or not TC material based on the particle morphology and chemistry.

Only a small fraction of the 6.7 cm² deposit area of each ambient filter was analyzed by CCSEM, typically about 1 mm², to complete each analysis in a reasonable time. Following CCSEM analyses, the EDX spectra and images of the particles of interest were manually reviewed, particles were relocated in the SEM for further examination and suspected TC particles were flagged.

Results

Source samples: Figures 1-10 are SEM photos of tire crumb material used at the three sampling sites. The TC particles examined did not show a single, unique, easily identifiable x-ray spectrum or particle morphology. Many of the tire crumbs displayed an "exterior" and an "interior" surface (Figs. 1, 2, 3a, 4a). The exterior surface often had a rough, fractured surface decorated with super-micron crustal-like aluminosilicate particles, quartz, and Fe-rich particles. (Figs. 3b, 4a). Freshly exposed interior surfaces tend to have a smooth surface embedded with many sub-micron Zn-rich inclusions (Figs. 4b, 5-7). It is postulated that some of the sub-micron Zn-rich particles observed in the ambient samples (e.g., Figs. 15, 21, 24, 37, 39, 43, 45, 46) may have been liberated from the TC matrix as part of the mechanical wear process. Carbon and sulfur were consistently present in TC particles; zinc was usually, but not always observed in TC particles (Fig. 8), and often was found at a trace level (Fig. 9). TC particles varied considerably in morphology such that it is difficult to identify a typical or characteristic TC morphology. Infrequently, TC source sample particles had the appearance of a bundle of fibers (Fig. 10).

It is questionable whether the morphologies observed in these very large source particles are of any relevance in identifying PM_{10} TC particles collected on the air filters.

*Air PM*₁₀ samples: Particle loadings on ambient samples were excellent for CCSEM, with relatively few instances of particles touching or overlapping. Representative field images are shown in Figs. 11 and 12 (samples from Site F1 Location B and Site F1 Location D,

respectively). Figures 13-46 show images and X-ray spectra of a subset of postulated TC particles found in samples from the three sites.

Table 1 below summarizes the results of the CCSEM analyses of 11 samples. The third column of Table 1 (#TC) shows the number of TC-like particles identified in each sample after manual review of the CCSEM data. The area analyzed by CCSEM differed somewhat for each sample, and the number of TC-like particles reported is not normalized to the area, so these numbers cannot be strictly compared between samples. The TC mass associated with each particle is estimated by assuming the particle to be a prolate sphere, calculating the volume from the projected area of the particle, and assuming a density for each particle. (For particles which are primarily carbonaceous, a density of 1.5 was assumed. For particles which are mostly noncarbonaceous, the density is the average weighted density calculated from the EDS spectrum where the density of each metal detected in the particle (excluding carbon) is weighted by its fraction of X-ray counts in the EDS spectrum). The Scaled TC Mass is the estimated TC mass on the entire filter, assuming that the area analyzed is representative of the filter as a whole, and that the exposed filter area is 6.7 cm². The estimated tire crumb concentration in each sample

Table 1. Concentration of TC-like particles in ambient samples.

		2	Mass	Scaled TC	TC
Sample ID	Run#	# TC ^a	TC ^a pg	Mass ^b µg	μg/m³
F1D1 Location B	10C	18	90	0.08	0.019
F1D1 Location A	21D	5	6	0.01	0.001
F1D1 Location A Duplicate	9D	15	80	0.02	0.004
F1D1 Location A Dup					
Repeat	21B	5	65	0.03	0.007
F1D1 Location D					
(background)	21C	7	16	0.01	0.003
F4 Location B	28C	6	47	0.03	0.007
F4 Location A	28D	0	0	0.00	0.000
F4 Location A Duplicate	28B	4	19	0.01	0.003
F4 Location D					
(background)	28A	6	15	0.01	0.002
P1 Location A	22A	63 ^c	3330	3.9	0.77
P1 Location B	16E	58 ^d	2530	2.1	0.42
P1 Location D					
(background)	16D	5	40	0.03	0.005

^aThese are raw numbers, not normalized to the same analyzed area. (CCSEM areas analyzed ranged from 0.6 mm² to 3.6 mm²).

^bEstimated TC mass scaled to total exposed filter area of 6.7 cm².

^cMass median diameter ~2.6 µm.

dMass median diameter ~2.2 µm.

assumes a sample volume of 4 m³. Samples highlighted in yellow (D sites) were collected at playgrounds or turf fields which did not use TC material, but which were located near the A and B sites.

There is no independent means or SEM calibration standard to check the accuracy of the SEM analyses reported in Table 1. These are crude estimates whose uncertainty could be at least a factor of ± 5 . (See caveats in Discussion section).

Discussion

Even though the numbers in Table 1 have large uncertainties in absolute terms, the two P1 samples (Locations A and B) collected at a playground using TC material, clearly stand out from all other samples. These two samples showed much higher number and mass concentrations of TC-like particles than samples collected at the background sampling location (Location D) and at all F1 and F4 sampling locations. The TC-like mass concentrations at the two P1 sampling locations were estimated to be 90x and 160x higher than the background sample, with estimated concentrations of about 0.53 and 0.96 µg/m³, respectively in the PM₁₀ size fraction. In eight of the remaining ten samples, there were ≤ 7 TC-like particles, too few to support any conclusions regarding differences between the "on field" and background sampling locations. Although the data suggest possible enrichment of TC-like particles in F1 samples at Locations B and A (duplicate), an earlier analysis of the collocated sample from Location A did not show a significant enrichment in TC mass concentration compared to the background sample. The repeat analyses of the Location A duplicate sample provides a rough assessment of the overall analysis precision including the CCSEM analysis and the subjective, manual data interpretation. This precision is about 50% and is attributed to the small number of candidate particles coupled with the subjective nature of the particle classification. The estimated mass median diameters of the TC-like particles in samples in the P1 Location A and Location B samples were 2.6 µm (63 particles) and 2.2 µm (58 particles), respectively.

Concern has been expressed about the potential for inhalation exposure to fibers from TC material. Very few fibers were observed in any of these samples and none that could be attributed to TC.

A number of factors may contribute to the observed enrichment in TC-like particles at the P1 site and the lack of enrichment at the F1 and F4 sites: (1) The P1 site was a playground (as opposed to a turf field); (2) the P1 site may have had more and/or more vigorous activity during the sampling period than the other sites; (3) the P1 sampler may have been located in an area of unusually high ambient TC concentrations; (4) TC materials vary in composition and mechanical wear properties. In particular, Zn concentrations in tires can vary nearly a factor of 10: the TC material used in the P1 site may have had elevated Zn compared to the TC material used at the F1 and F4 sites. Since the range of Zn concentrations in tires approximates the EDS detection limit for Zn, any increase in the Zn concentration of the TC material would have a major effect on the number of TC-like particles identified by CCSEM. (ICP analyses of the bulk TC samples should answer this question).

Caveats: The ability to quantify the TC concentration in these samples by SEM/EDS hinges on the TC particles having distinct and unique composition and/or morphology which would enable

the analyst to identify TC particles with a high degree of confidence. This does not seem to be the case for TC particles, as seen in the variety of morphologies and compositions in Figs. 1-10. The identification of the TC particles in Table 1 was a subjective judgment and large uncertainties should be assumed in the estimated TC contributions in Table 1. Reasonable arguments could be made that the TC concentration estimates in Table 1 are either underestimates or overestimates. For example, the chemical markers used to identify TC particles (C, S, and Zn) are not unique to tire crumbs and can be found in particles from common non-TC sources, potentially resulting in an overestimate. On the other hand, Zn, being the most critical tracer for TC, is poorly detected by EDS. It is possible that many Zn-bearing TC particles are undetected in the CCSEM analysis because the Zn concentration is just below the minimum detectable level, thus resulting in an underestimate of the TC concentration.

As seen in Fig. 11, most particles 1 µm and smaller are sulfate particles whose X-ray spectra show both C and S (C is contributed by the polycarbonate substrate). Distinguishing TC particles from these sulfate particles based on chemistry alone is difficult, if not impossible. And, as discussed above, identifying TC particles by morphology for particles this small is probably not feasible. However, many, if not most, sulfate particles are damaged by the focused electron beam which leaves a visible hole in the particle. TC particles are not expected to exhibit similar beam sensitivity, so this may provide a means of distinguishing TC particles from most sulfate particles. Isolated Zn-S particles (e.g., Figs. 19 and 20) were observed in most samples and attributed to TC, even though there may be industrial sources of ZnS particles other than TC. It is also possible that TC particles may be generated from traffic tire wear with airborne transportation to and across the field and playground sites.

Selected SEM Images and EDS Spectra of Tire Crumb Source Particles

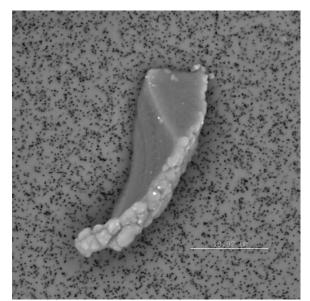


Figure 1. TC particle showing smooth interior and rough exterior surfaces. Image was acquired in BSE mode in which particle brightness increases with atomic number. (Site F1)

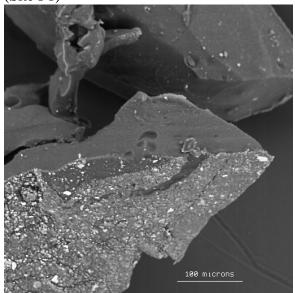


Figure 3a. Rough exterior surface and freshly cleaved interior surface. The bright features on the rough exterior surface are mostly super-micron crustal particles. (Site F4).

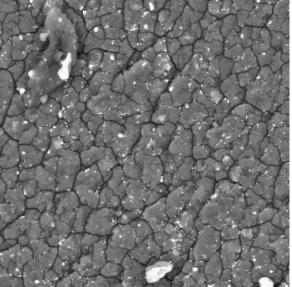


Figure 2. Rough fractured exterior surface of TC particle. (Site F1). The carbonaceous matrix (darker) is sprinkled with bright crustal-like particles of aluminosilicates, quartz, and Fe-rich.

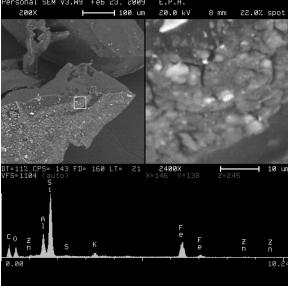


Figure 3b. The magnified image (upper right) is the area shown in the square in the low-mag image at left. The EDS spectrum was acquired at the spot located by the small box in the right-hand image (bright, rectangular particle just below center of image). The particle appears to be an Fe-rich aluminosilicate crustal particle.

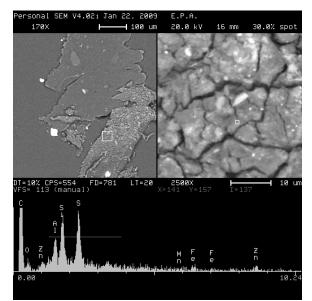


Figure 4a. Two TC particles showing smooth interior surface (upper particle) and rough exterior surface (lower particle) (Site P1). The EDS spectrum acquired in the exterior surface shows a matrix rich in S, Si, and Al with a trace of Fe and Zn.

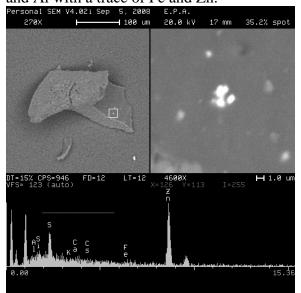


Figure 5. EDS spectrum was acquired from the bright, micron-sized Zn inclusion (zoomed image on right) in the interior surface of the tire crumb. (Site F1)

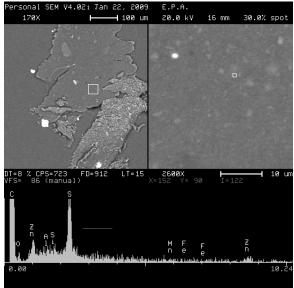


Figure 4b. Interior surface is much smoother and is not decorated with supermicron crustal particles. Spectrum shows S and trace of Zn.

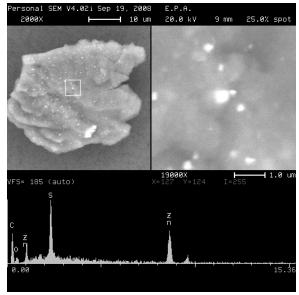


Figure 6. EDS spectrum collected from the bright, micron-sized Zn inclusion (zoomed image on right) shows C, S, and Zn.

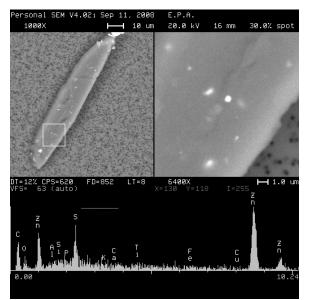


Figure 7. EDS spectra of TC particles typically show C, S, and Zn. The spectrum was acquired from the bright inclusion at center of right-hand image. (Site F1)

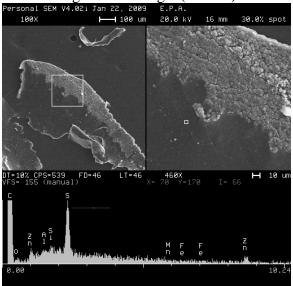


Figure 9. EDS spectrum shows C, S and trace Zn in the interior surface of this TC particle. (Site P1)

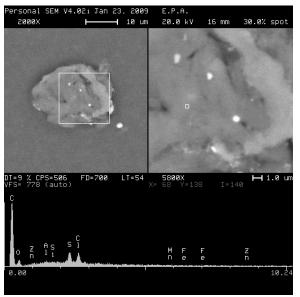


Figure 8. EDS spectrum from this TC particle showed no Zn in the TC matrix. (Site P1)

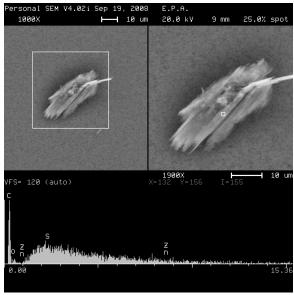


Figure 10. TC particle from shredded tire sample (Site P1) showing fibrous morphology. EDS spectrum shows only C.

Selected SEM Images and EDS Spectra of Air PM₁₀ Samples.

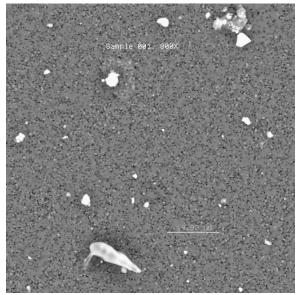


Figure 11. Representative field image for Site F1 Location B sample. Magnification = 600x. Tiny sub-micron black dots are pores in the polycarbonate filter. Sub-micron gray dots are sulfate particles.

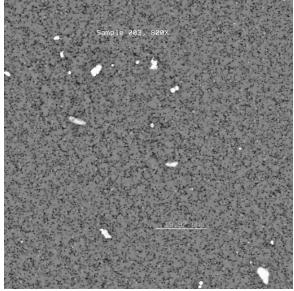


Figure 12. Representative field image for sample Site F1 Location A Duplicate sample. Magnification = 600x.

Site F1 Air PM_{10} Samples: Postulated TC Particles

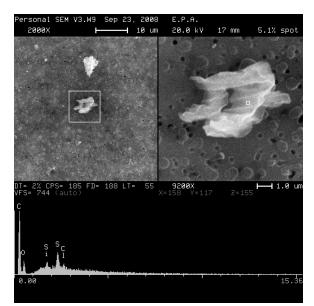


Figure 13. C-S-rich particle. Site F1 Location B sample.

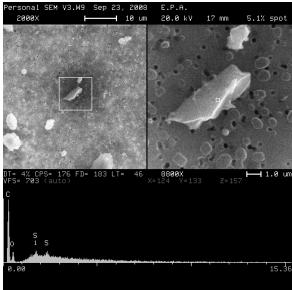


Figure 14. C-S-Si-rich particle. Site F1 Location B sample. Note the dull-gray, micron-sized sulfate particles.

Site F1 Air PM₁₀ Samples: Postulated TC Particles

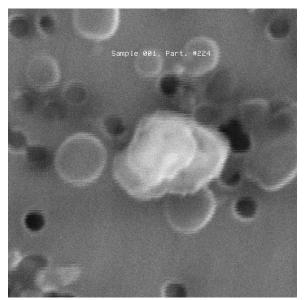


Figure 15. C-Zn-S-rich particle. Site F1 Location B sample, #224

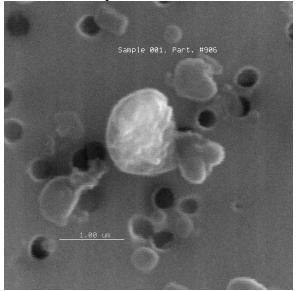


Figure 17. C-S-Si-Zn-rich. Site F1 Location B sample, #906

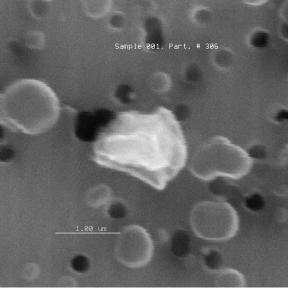


Figure 16. C-Zn-S-Si-rich. Site F1 Location B sample, #306

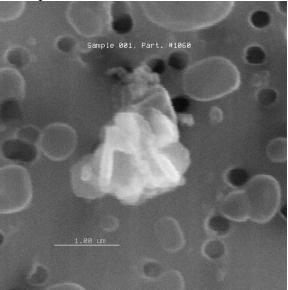


Figure 18. C-S-Zn-Si-rich Site F1 Location B sample, #1060

Site F1 Air PM₁₀ Samples: Postulated TC Particles

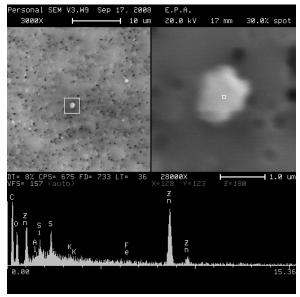


Figure 19. C-Zn-S-rich particle. Site F1 Location A Duplicate sample, #179

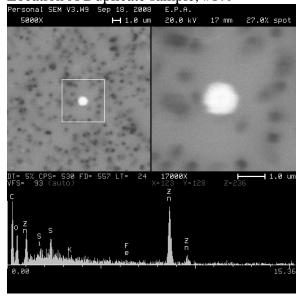


Figure 21. C-Zn-S-rich particle. Site F1 Location A Duplicate sample, #1148

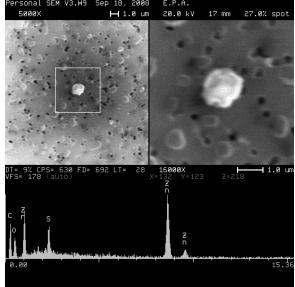


Figure 20. C-Zn-S-rich particle. Site F1 Location A Duplicate sample #794

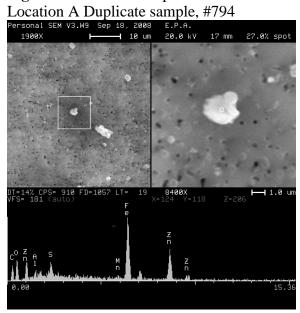


Figure 22. C-Fe-Zn-S-rich particle. Site F1 Location A Duplicate sample, #1241

Site F1 Air PM₁₀ Samples: Postulated TC Particles

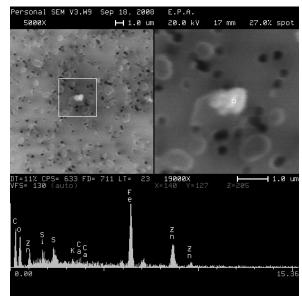


Figure 23. Site F1 Location A Duplicate

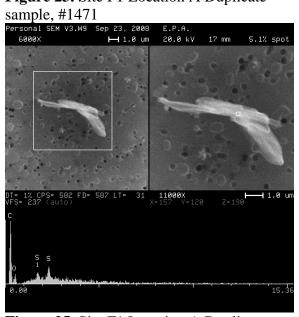


Figure 25. Site F1 Location A Duplicate sample, #519

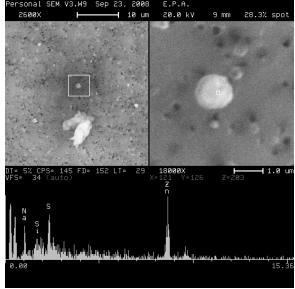


Figure 24. Site F1 Location A Duplicate

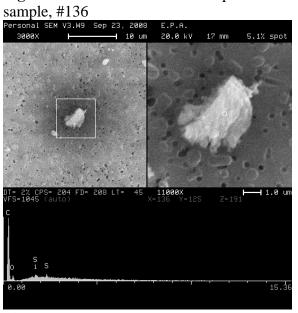


Figure 26. Site F1 Location A Duplicate sample, #153

Site P1 Air PM₁₀ Samples: Postulated TC Particles

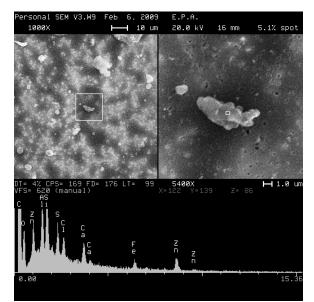


Figure 27. Site P1 Location A sample, # 515

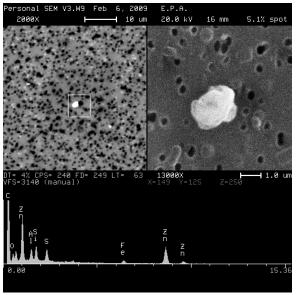


Figure 29. Site P1 Location A sample, #1712

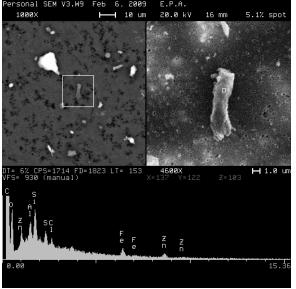


Figure 28. Site P1 Location A sample, #1546

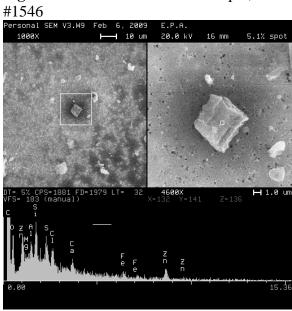


Figure 30. Site P1 Location A sample, #162

Site P1 Air PM₁₀ Samples: Postulated TC Particles

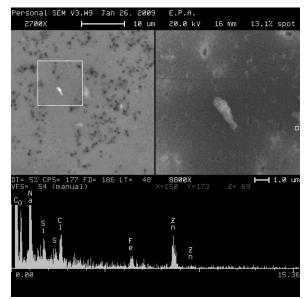


Figure 31. Site P1 Location A sample, #1500

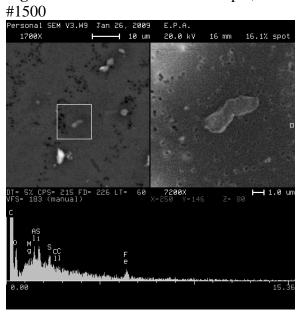


Figure 33. Site P1 Location A sample, #1115

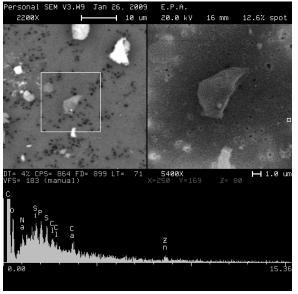


Figure 32. Site P1 Location A sample, #49

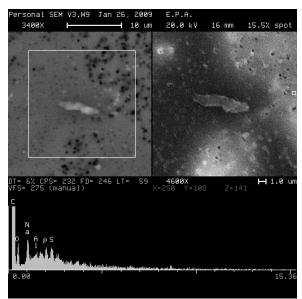


Figure 34. Site P1 Location A sample, #1537

Site P1 Air PM₁₀ Samples: Postulated TC Particles

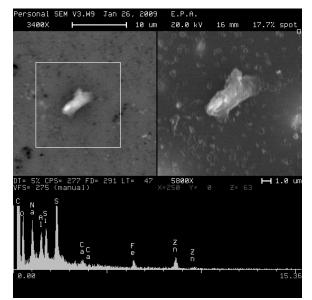


Figure 35. Site P1 Location D sample, #834

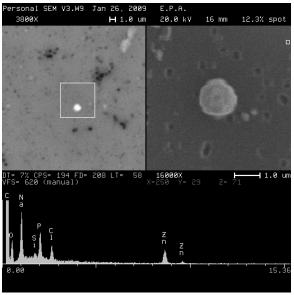


Figure 37. Site P1 Location D sample, #1328

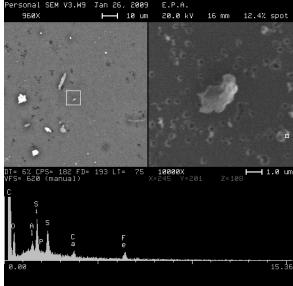


Figure 36. Site P1 Location D sample, #1065

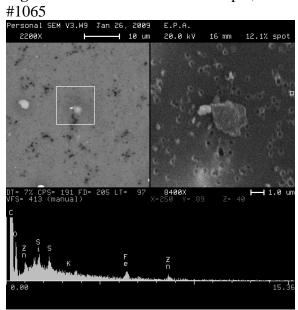


Figure 38. Site P1 Location D sample, #1370

Site F4 Air PM₁₀ Samples: Postulated TC Particles

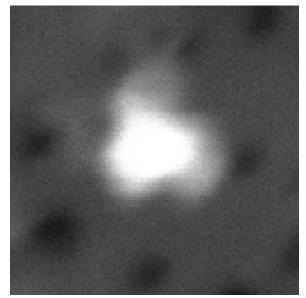


Figure 39. Fe-Zn-S-rich. Site F4 Location B sample, #146

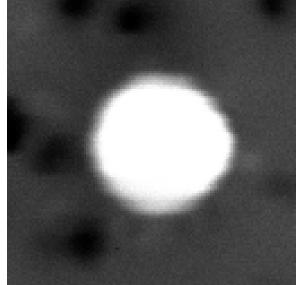


Figure 40. Si-Al-Zn-Fe. Site F4 Location B sample, #761

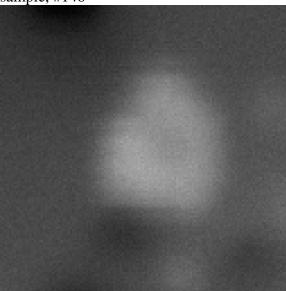


Figure 41. S-K-Zn-Ca. Site F4 Location B sample, #1038

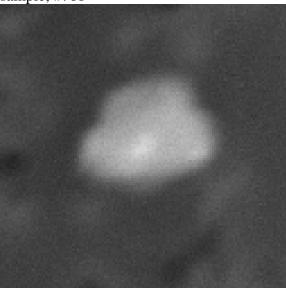
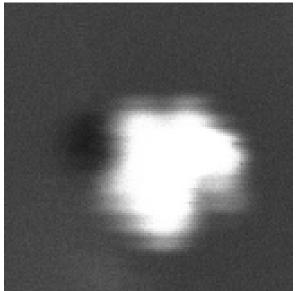


Figure 42. Si-Al-Zn-S-Fe. Site F4 Location B sample, #1561



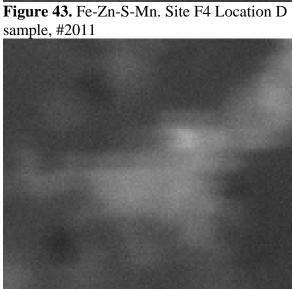


Figure 45. C-S. Site F4 Location D sample, #1480

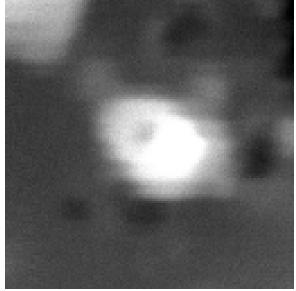


Figure 44. Zn-Fe-S-Si. Site F4 Location D sample, #2118

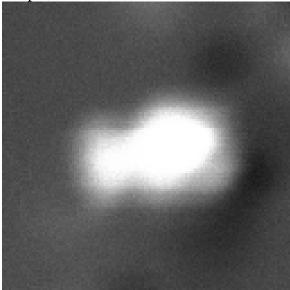
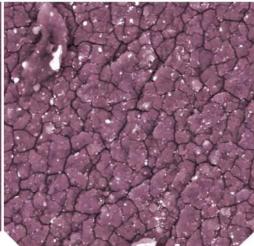
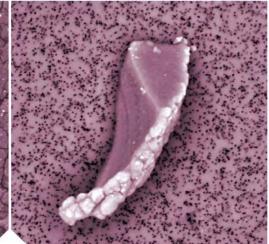


Figure 46. S-Ti-Fe-Zn. Site F4 Location D sample, #2262







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