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Automated SEM/EDS Analysis of Airbag Residue.* I: Particle Identification

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ABSTRACT: Deployed airbags can be a valuable source of probative forensic materials. During an accident, trace evidence can be deposited on the airbag cover and in addition, the residue produced by the gas generation system is released into the passenger compartment of the vehicle as the airbag deflates. This residue can be used to associate a suspect with the vehicle at the time of the accident. This study identifies particles containing zirconium, strontium, and/or copper–cobalt along with other elements from the gas generation systems and aluminum silicon microfibers from airbag filters as the probative material which may be produced and deposited on a suspect's hands and/or clothing. Scanning electron microscopy can be used to identify this metallic residue. Modification of the search criterion used for gunshot residue analysis allows for automated analysis of the samples. Proper collection of the airbag standard is essential to identify which materials were produced. Prompt collection of suspect samples allows the analysts the ability to make the proper identifications and associations. This analytical technique can be a probative tool in criminal investigations.

KEYWORDS: forensic science, trace evidence, scanning electron microscopy, energy dispersive spectroscopy, airbag, backscattered electron detector

In a crash situation, the airbag is intended to cushion the vehicle's occupants and protect them from hard contact with the interior of the car, known as the "second collision." The occupants decelerate as the front of the vehicle compresses and the airbag inflates. The occupants "ride down" the airbags as the vehicle continues to decelerate (1).

The typical airbag system consists of crash sensors that identify when a collision has occurred, a gas generator to inflate the airbag, and the airbag itself. Filters may be used to screen particles produced during the chemical reaction and to stop these particles from entering the airbag and possibly damaging it. Even with the filters in place, the presence of particles in the airbag indicates the inability of the filters to screen all byproducts from the generated gas.

The airbags themselves are constructed of nylon fibers, with the older models having an inner neoprene lining. Some airbags have vent holes facing forward to direct the escaping gas away from the passenger. The newer technology has coated, nonvented airbags, with the amount of gas escape calculated by the porosity of the airbag. The nonvented airbags may stay inflated longer for multiple collisions without interfering with the occupants as they attempt to exit the vehicle (2).

Gas generation systems are divided into three categories: pyrotechnic, compressed or cold gas, and hybrid models. The pyrotechnic group includes both the early sodium azide-based systems and the more recently developed nonazide sources.

Pyrotechnic systems rely on the precise consumption of a solid propellant that generates a predetermined volume of gas in a predetermined period of time. This inflator type is typically used in driver side systems. Sodium azide was the initial choice for the solid propellant, even though the nonreacted propellant is toxic. The main combustion product is harmless nitrogen gas (1). Additives neutralize the caustic secondary byproducts (3). These disadvantages are offset by the small, lightweight design, and low costs (4). These systems rely on various compounds as oxidizers.

Current research is identifying nonazide-based propellants to eliminate some of the inherent problems. These formulations use alternate organic fuels with oxidizers. Some formulations produce such low amounts of solid residue that a filter is not necessary (5).

Compressed gas generators are more commonly found in side-curtain airbags. Nonvented airbags with low porosity are used. Keeping the airbag inflated for several seconds is important in rollover-type accidents. Compressed gas cylinders containing inert gasses, like argon or helium, are rigged with a pyrotechnic charge for the release of the gas.

Hybrid models work with both a chemical reaction and the release of compressed gas. This combination is used in both passenger and side-seat airbag systems. Both parts can be released simultaneously or in stages, if designed in that manner. The current technology of "smart airbags" relies on the dual nature of the generator, allowing the chemical reaction in less severe collisions, but triggering both sources when a severe accident is detected (6).

Methods

Automated scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) analysis allows the analyst to quickly identify and categorize large populations of particles without having to manually operate the instrument. This study was done using several such instruments (Aspex Instruments, Delmont, PA). The SEM is equipped with a backscattered electron (BSE) detector which is used to screen particles based on their brightness, selecting high average atomic numbers and bypassing those with lower average atomic numbers. The detector is calibrated by the

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brightness of copper versus the darkness of the adhesive tape used on the sample stubs. If the brightness of the particle surpasses the threshold, the EDS detector begins to collect and segregate the characteristic X-rays emitted by the elements in the sample particle. This identifies the elements in the particle and the software attempts to sort the particle into preset categories. The software purchased with the instruments was written for automated gunshot residue (GSR) analysis and is a reasonable way to sort most of the detected particles. The samples are scanned at magnification of 180× with a maximum integration time of 4 sec. The ½ inch sample surface is divided into *c.* 140 equal sized fields, with each field being divided into 25 electronic fields. The instrument scans across the electronic field and will analyze the first 25 particles which surpass the brightness threshold before going on to the next electronic field.

The airbags sampled in the study were recovered from both foreign and domestic vehicles from the 1991, 1994, and the 1996 to 2006 model years. Most of the airbags in the study were collected from four automotive repair shops that participated in the study, although a few were recovered from a State of Illinois junkyard. The majority of airbags were either driver or passenger side, although some side seat or side curtains were submitted. Samples were taken from the interiors of the airbags. The SEM stub was inserted through the vent holes and the sticky tape sample surface was dabbed against the inner surface. If the airbag was nonvented, a cut in the fabric was made to facilitate sampling.

Results

The residue formed from an exothermic chemical reaction is the byproduct of the reactants and any unconsumed reactants. Trace amounts of other materials may be detected in the byproducts if they are present when the reaction occurred. Particles with different elemental compositions are formed as the materials cool.

The airbag residue samples showed diversity in their compositions. The presence of large particle populations along with trace amounts of elements give an indication of the possible formulation used in the airbag system. The following particle types were commonly found in airbag residue standards. These particles are not intended to be the complete listing of all possible particle populations found in every airbag standard.

Zirconium has many uses, including a heat source for "squibs," a pyrotechnic device used to ignite a charge, and airbag inflators (7). The signal from the crash sensor ignites the squib, which is attached to an initial charge. The high pressure and temperature of the initial charge ignites the gas generant. Zirconium, when mixed with potassium perchlorate as an oxidizer, forms ZPP. This initiator charge is used in numerous patents for airbag inflators. Titanium may be added to this mixture in a powdered form which either acts as an additional metallic fuel or as an additional metal oxide.

Large populations of zirconium rich particles were commonly detected (Figs. 1–5). The automated search software does a poor job of classifying the zirconium particles it has detected. Zirconium is only present in the preset category "zircon." In order for the software to sort the particle into the zircon category, it must also detect the presence of silicon in the sample. Lacking silicon, these particles were erroneously classified as aluminum, miscellaneous silicates, miscellaneous metals, "other," iron rich, stainless steel, sintox, zinc, and various GSR particle types, with or without trace amounts of the classification material present. An additional search criterion of "zirconium rich" is essential to the GSR search criterion if this software is to be used for airbag residue analysis.

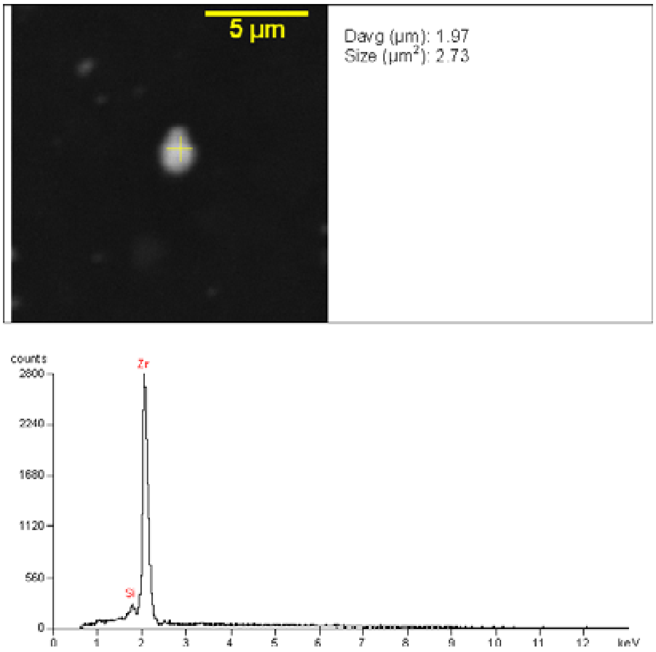

FIG. 1—*This is a typical zirconium particle. Its spherical morphology and BSE image brightness makes it easily detectable in automated analysis. The spectrum for the particle shows a trace of silicon.*

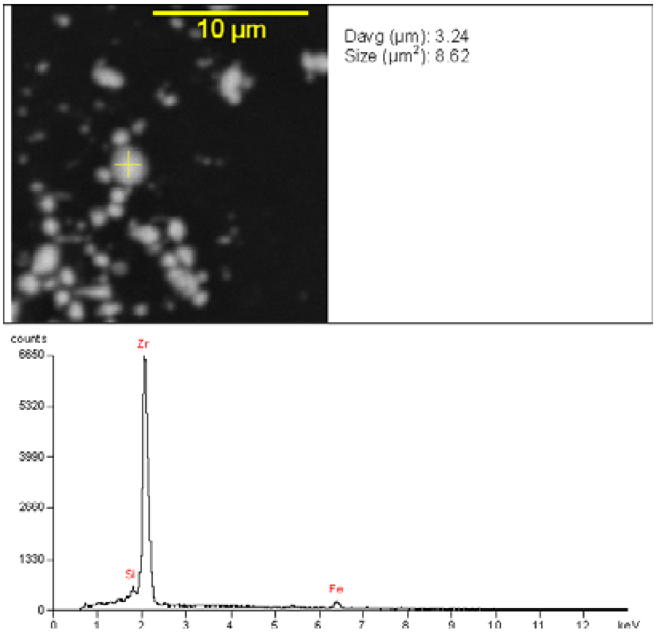

FIG. 2—*This is another typical zirconium particle. Its appearance, along with the other zirconium particles in the field, is spherical. The spectrum shows a trace of silicon and iron.*

Analysis of the passenger side airbag from a 1999 Honda Accord categorized 84% (9,512 out of 11,279 total particles) of the detected particles as "other" along with 52 zircon particles. Analysis of the same standard using a search criterion which contained a "zirconium rich" category identified 74% (6,865 out of 9,275 total particles) of the particles as containing zirconium along with seven zircon particles.

A substitute for sodium azide as a gas generant was developed that contains amine nitrates as the organic fuel plus basic copper

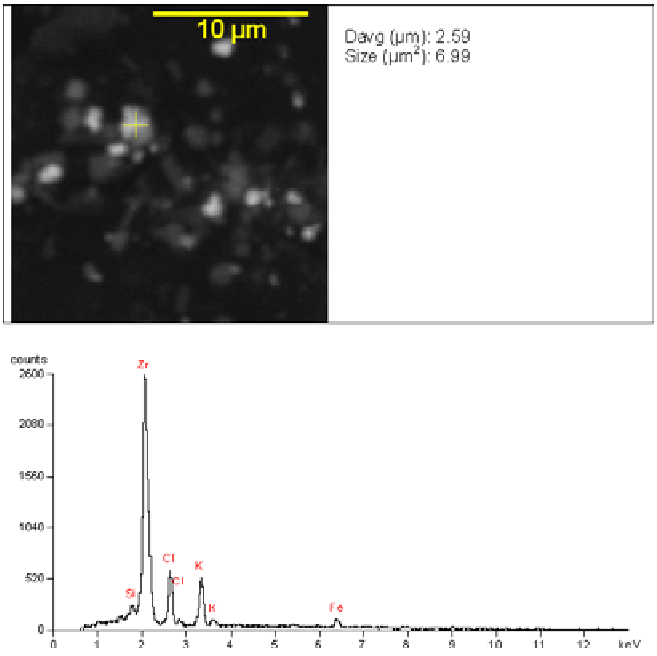

FIG. 3—*Zirconium particles may also be detected with trace amounts of potassium and chlorine. This particle may be residue from the ZPP formulation. Tungsten particles, possibly from the bridge wire in the squib, are sometimes detected.*

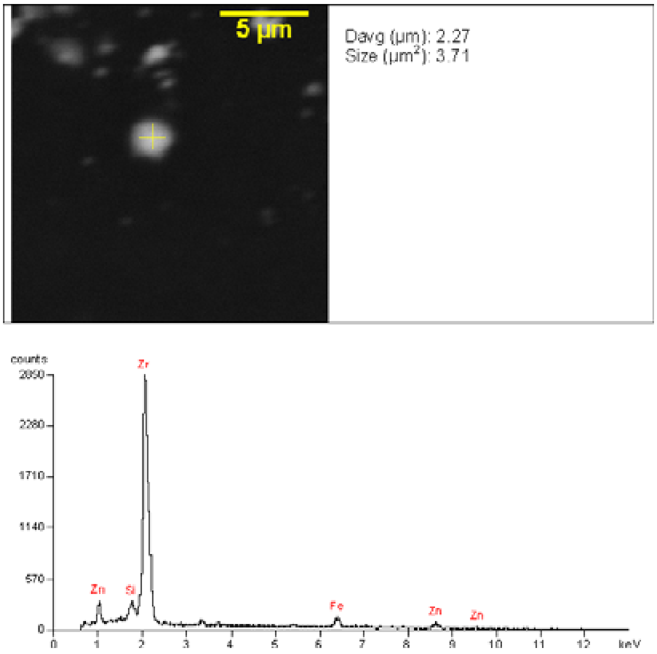

FIG. 4—*Zirconium particles may also contain trace amounts of zinc. This particle has a spherical appearance and its spectrum shows a trace of zinc. Particles with higher zinc levels were also detected and may be categorized as zinc.*

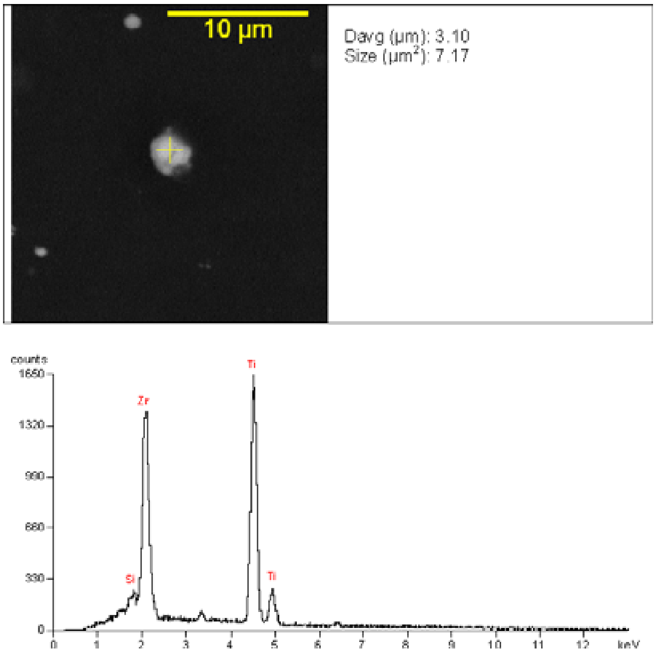

FIG. 5—*This particle could be the result of powdered titanium being added to the zirconium as a metallic fuel or oxidizer.*

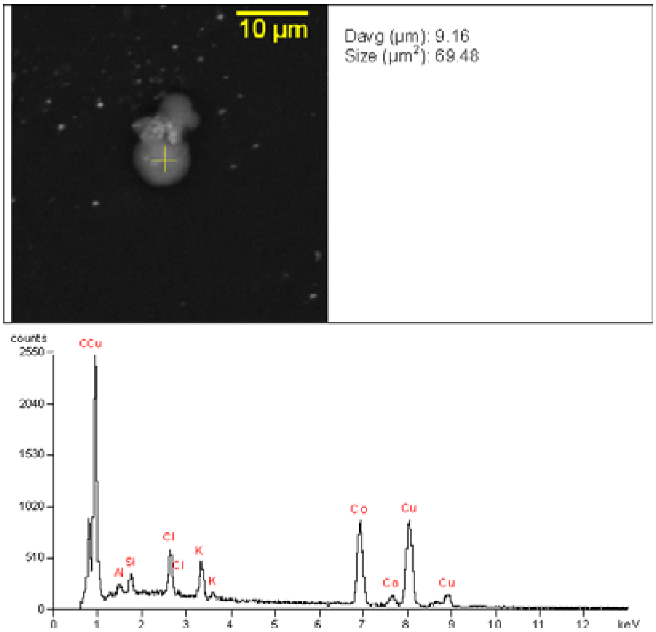

FIG. 6—*This particle has a spherical appearance with moderate BSE brightness. The spectrum shows copper and cobalt with traces of potassium, chlorine, silicon, and aluminum.*

(II) trinitrate and/or cobalt (III) triammine trinitrate as oxidizers. The advantages of this mixture are high gas yield, moderate combustion temperature, and the filterability of the combustion products (8). Autoliv ASP, Inc. (Ogden, UT) called for the use of hexammine colbalt (III) trinitrate (HACN) with copper (III) trinitrate and/or copper (III) trihydroxynitrate as oxidizers in their patent for a hybrid gas inflator (9). The only solid material produced is cobalt and/or copper. The cobalt-based formulations may be phased out in favor of a basic copper formulation.

Despite the claims of filterability, large particle populations suspected of being residue from this formulation have been identified. Trace amounts of potassium and chlorine may be present (Fig. 6). Increased levels of the potassium and chlorine may darken the BSE image brightness and the particle may be classified as a miscellaneous salt. Particles with other trace elements have been detected that may be beneficial in identification of the residue.

Some of the particles produced by the previously mentioned reaction may contain various amounts of copper and cobalt (Figs. 7 and 8). Copper is present in the preset sort categories, but cobalt is not. Some of the particles were identified as copper, but the software is forced to sort many of those particles incorrectly as mercury bearing, tungsten, or various GSR particle types. By adding a "cobalt" category, the software properly sorts most of those particles. Analysis of the previously mentioned 1999 Honda Accord passenger airbag detected 174 copper particles. Reanalyzing the same sample using the modified airbag search criterion detected 78 copper particles and 519 cobalt particles. Many of the detected copper or cobalt particles had trace amounts of the other element present.

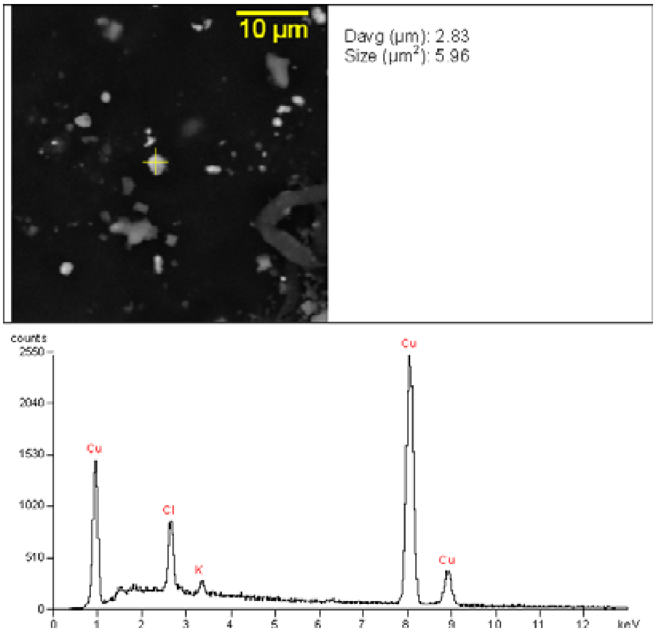

FIG. 7—*This particle is from a standard that has numerous copper-cobalt particles present, although no cobalt is detected. This particle is easily sorted with other copper particles. Chlorine with a trace of potassium is present.*

Analysis of a driver side airbag from a 2005 Nissan Ultima detected a particle population of 37% (4,727 particles) strontium, 29% (3,702 particles) aluminum, and 27% (3,545 particles out of 12,702 total particles) iron-rich particles. Strontium nitrate is used as an oxidizer in airbags, along with pyrotechnic and signal flare applications (10), and in some lead-free percussion primer formulations (11). The remaining particles may be due to several sources. The aluminum may be present from its use as an ignition booster when mixed with secondary explosives. Patents for inflator systems call for iron in various forms, including uses as oxidizing agents and as oxidizable inorganic fuels. Particles of these types are common in airbag standards (Figs. 9–13). The probative value of iron is questionable because of its occurrence in everyday particle populations. Therefore, high populations of iron-rich particles with trace elements may be the important identifying feature.

Zinc is used in many patented formulations and shows up as zinc rich particles or as minor component of many airbag residue particles (Fig. 14). Zinc as the peroxide in a hydrated form or as a metal complex is called for in patents for gas producing formulations.

Miscellaneous salts were detected in a number of airbag standards (Fig. 15). This may be the result of byproduct compounds from different oxidizers. They typically have a bright BSE image that would indicate a higher atomic number than the elements detected. The particle shape may give an indication on how the particle was formed.

Filters are used to capture particulates and draw excessive heat away from the generated gasses. Unifrax (Niagara Falls, NY)

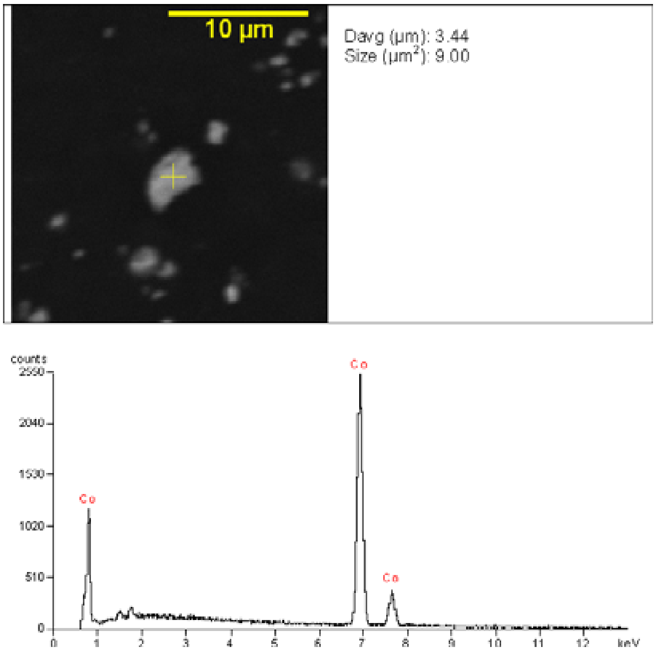

FIG. 8—*This particle is also from a standard that has numerous copper-cobalt particles present, although no copper is detected. Unless the search criterion is modified, this particle will not be properly identified.*

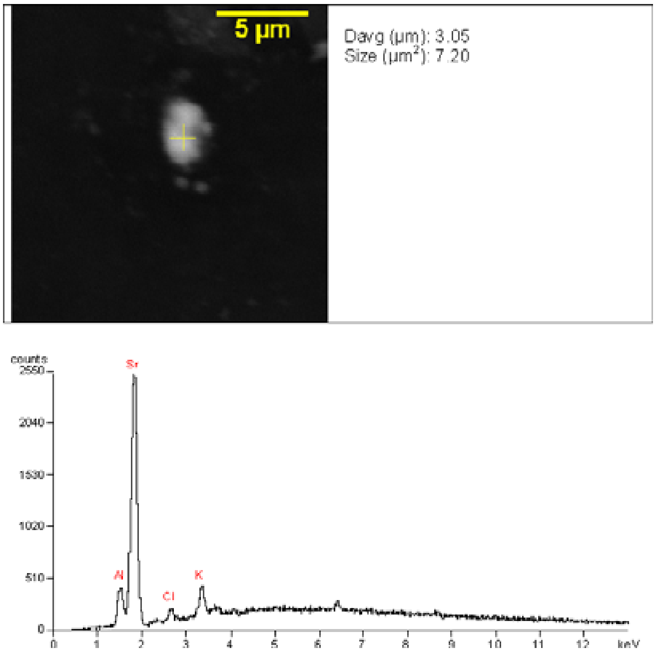

FIG. 9—*This is a typical strontium particle showing a moderate BSE brightness and spherical morphology. Traces of aluminum, chlorine, potassium and iron (not marked) are present.*

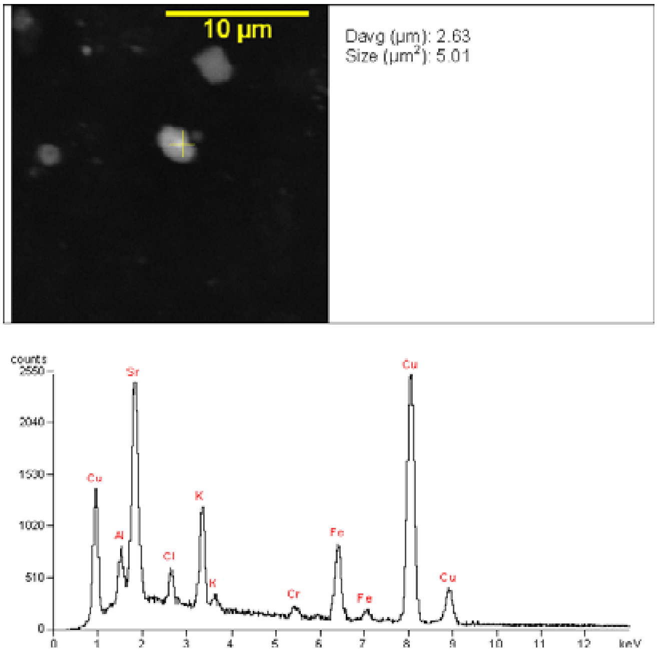

FIG. 10—*This particle was recovered from a standard that had numerous iron, copper, and strontium particles. This spectrum was consistent with numerous particles that were identified in the standard. The identification of a particle type that is frequently found in a standard may be important in determining which particles to look for in the questioned sample.*

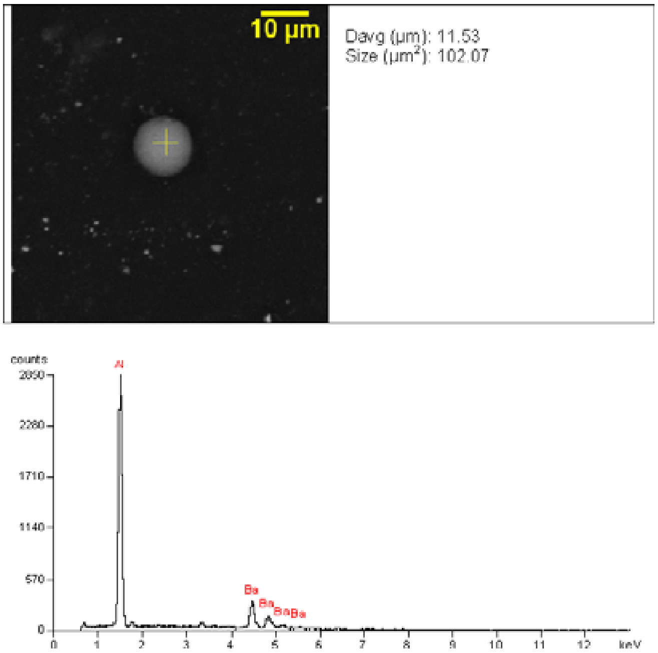

FIG. 11—*This particle is a large spherical aluminum particle with a trace of barium. Aluminum by itself may not have a bright BSE image, but other elements in the particle make it bright enough to be detected.*

manufactures PC Series Papers, a nonwoven filter for pyrotechnic airbag inflators composed of aluminosilicate fibers. The benefits of their product, as advertised, are efficient filtration by condensation, adhesion, and mechanical entrapment, significant cooling of the gas stream, and regulation of the gas flow. A nontoxic latex binder may be added to improve the characteristics of the filter (12).

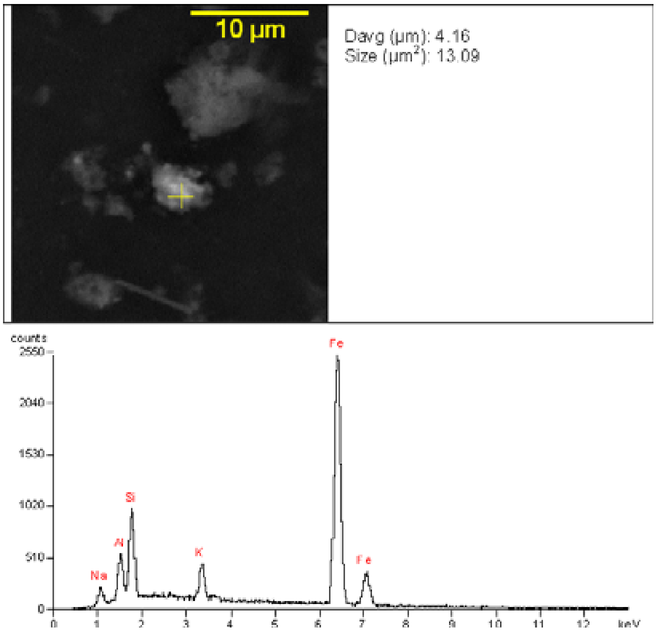

FIG.12—*This particle is a typical iron particle showing trace amounts of silicon, aluminum, potassium, and sodium. Some similar particles show traces of chlorine.*

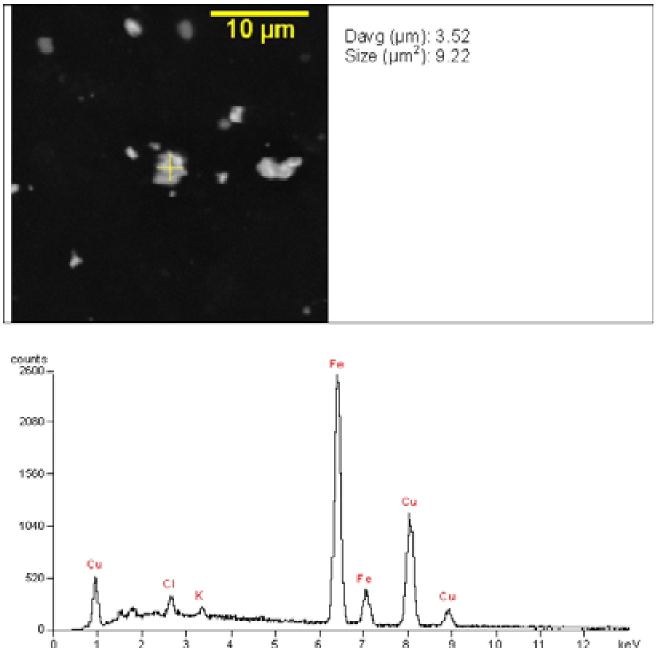

FIG. 13—*This particle shows a combination of major copper and iron peaks along with trace amounts of potassium and chlorine. This particle may have been categorized as either copper or iron.*

Fibers are commonly recovered from airbag standards. These fibers may be shed from the nylon airbag fabric. Nylon has no BSE image brightness because of the low atomic number elements in its composition.

Fibers which are much thinner than the nylon fibers are also commonly recovered from airbag standards (Fig. 16). These microfibers have moderate BSE image brightness. Elemental analysis indicates the presence of aluminum and silicon (Fig. 17). Some may also contain calcium (Fig. 18). They are typically sorted with

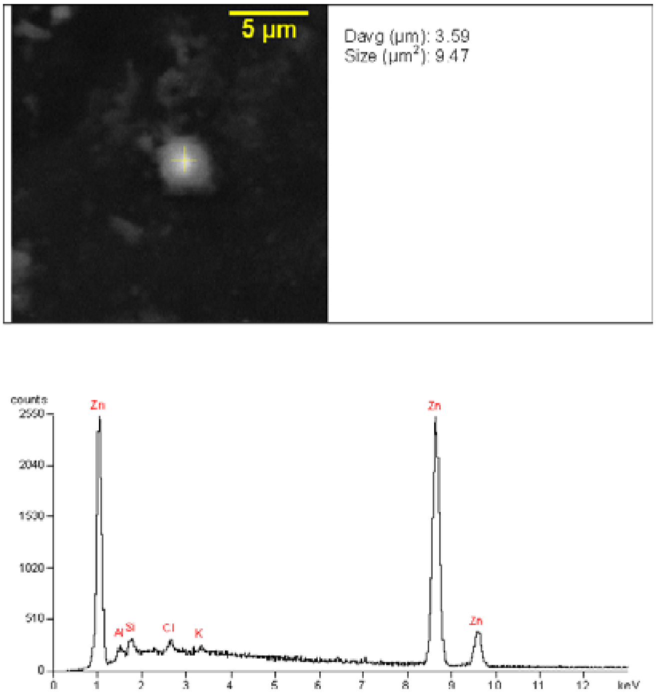

FIG. 14—*This particle shows zinc with trace amounts of aluminum, silicon, potassium, and chlorine.*

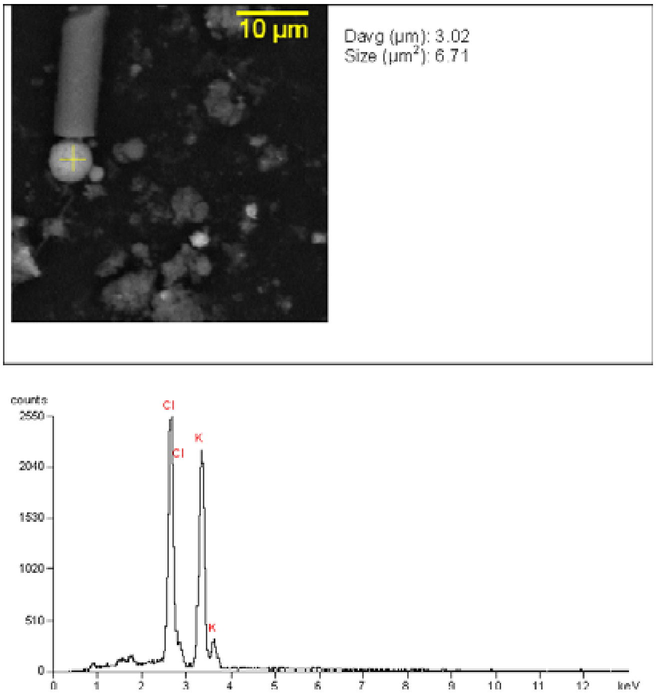

FIG. 15—*This particle is a miscellaneous salt particle that was detected due to its bright BSE image. Its spherical shape may be due to its formation by cooling as opposed to the typical crystalline shape.*

miscellaneous silicates, aluminum, or tungsten or they may be detected by the analyst when simply viewing the SEM sample stub at moderate magnification. These fibers may show heat damage, taking on the appearance of brittle, glassy spheres (Fig. 19). Particles adhering to these fibers may indicate the materials that were being filtered from the gas stream (Figs. 20 and 21).

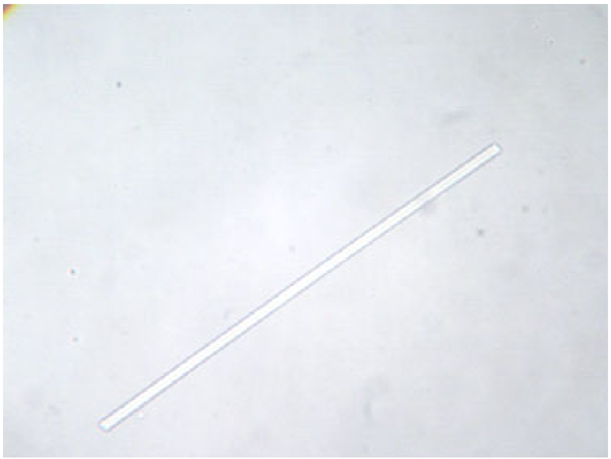

FIG. 16—*This is a photomicrograph of an aluminum/silicon microfiber in permount. It is isotropic with a relative refractive index greater than 1.525. The fiber is 15 μm wide.*

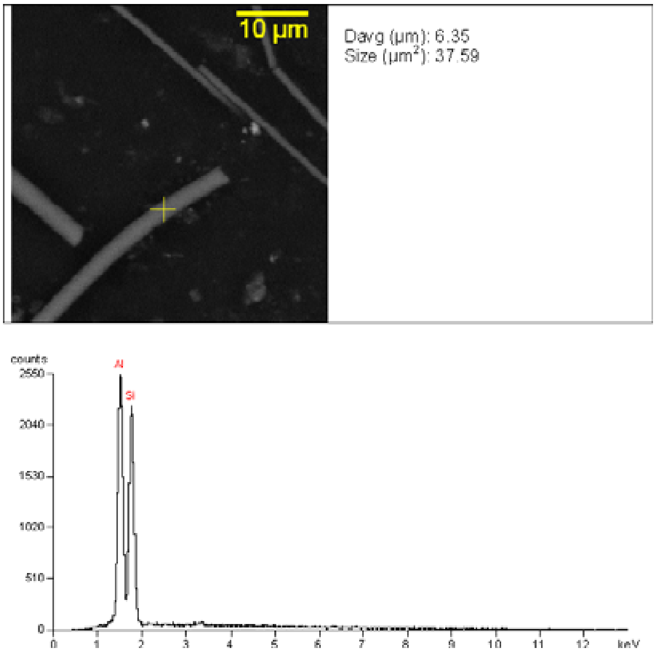

FIG. 17—*This microfiber is typical of microfibers recovered from airbag standards. The microfibers shown have a composition of aluminum and silicon. No heat damage was observed.*

Large masses of the melted material can be recovered (Fig. 22). This filter material may be confused with melted nylon fibers that may be present in the airbag. This material is easily distinguished from the nylon by the difference in the BSE image brightness and the spectrum. As their sizes increase, they become large enough to be detected by light microscopy.

A standard of Unifrax 200 series PC paper showed fibers of similar size and elemental composition as those recovered in some airbag standards. The optical properties of these fibers can be used for identification and comparison. Large masses of melted material, and brittle, glassy spheres, some with remnants of their "tails" were also observed (Fig. 23). This indicates that the heat damage is part of the manufacturing process and not due to the heat of the chemical reaction during deployment.

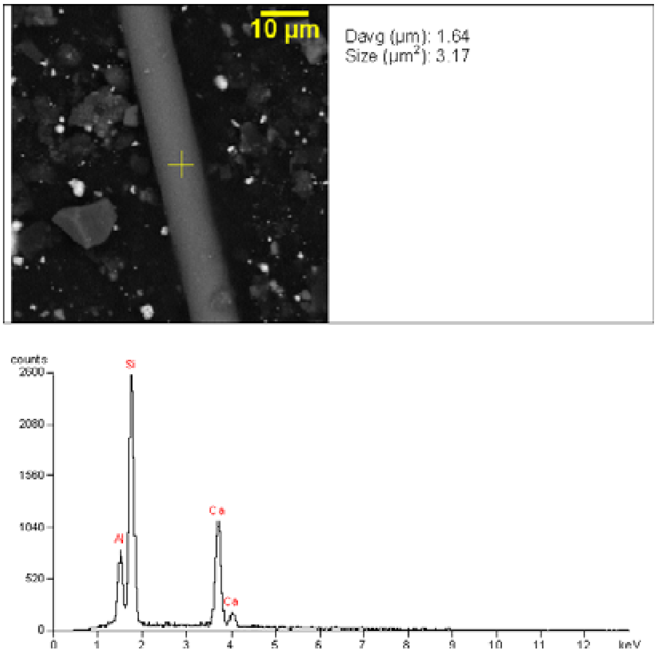

FIG. 18—*This microfiber is also typical of those recovered from airbag standards. Calcium is also detected with the aluminum and silicon.*

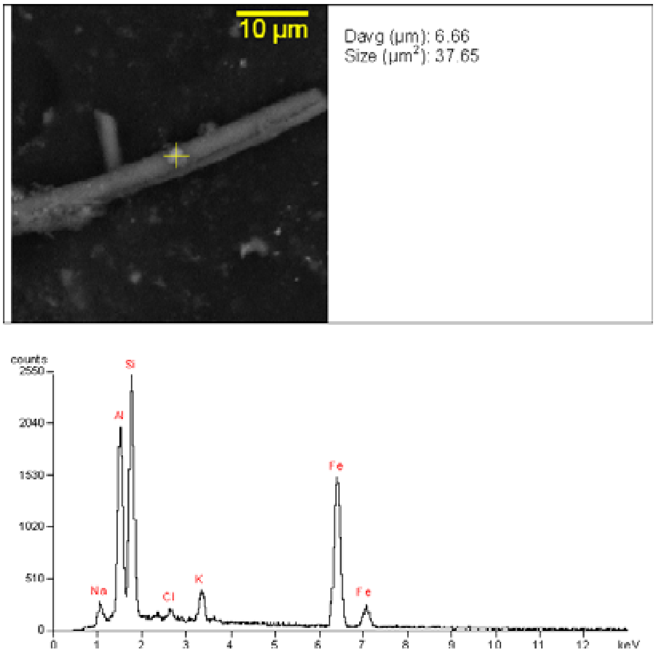

FIG. 20—*This is an image of an aluminum/silicon microfiber with a particle adhering to the fiber. The main particle type from this standard was iron rich particles with traces of sodium, aluminum, silicon, chlorine, and potassium.*

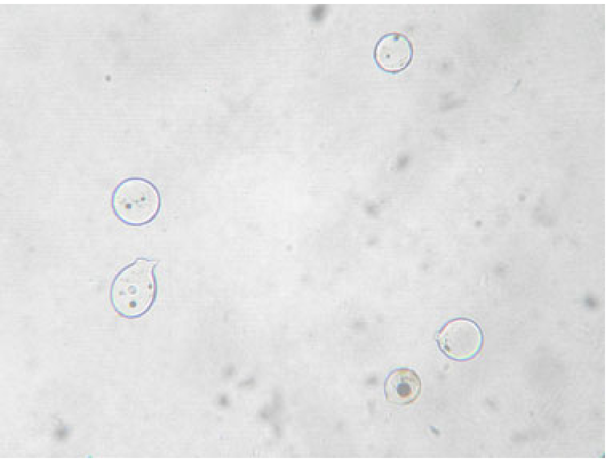

FIG. 19—*This is a photomicrograph of a sample of the brittle, glassy spheres, c. 100 µm wide, in permount. The particles tend to have air pocket inclusions and some of them show remnants of their "tails." The particles from this standard are isotropic and have a refractive index of 1.5468.*

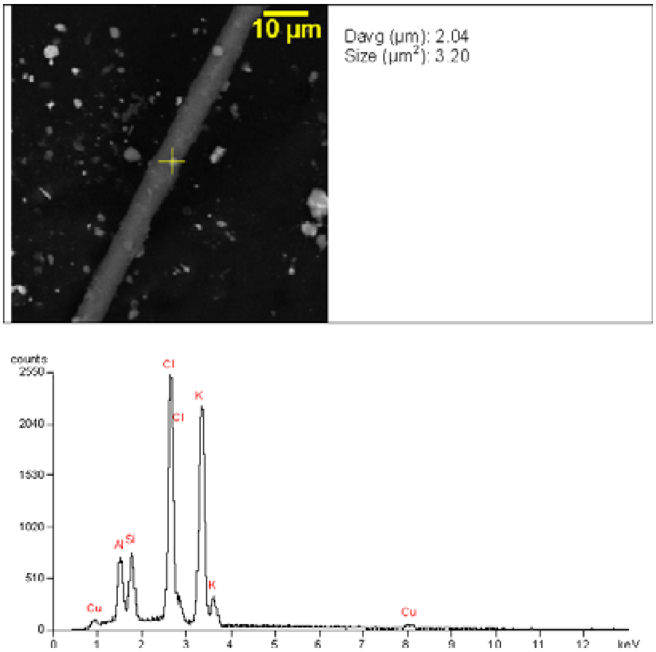

FIG. 21—*This image is an aluminum/silicon microfiber from a standard that had a high copper population. The particle adhering to the fiber shows potassium and chlorine and a trace of the copper.*

Case Report

Shortly before midnight on April 9, 2005 a traffic accident occurred in Naperville, Illinois. While the police who arrived at the scene tended to one of the drivers who was injured, the other driver simply left the scene and walked to her house which was a few blocks away. She was later arrested and charged with aggravated driving under the influence (DUI), leaving the scene of an accident, disobeying a traffic signal, and speeding.

In an effort to associate the suspect with being in the car at the time of the accident, her clothing and the deployed airbag from her vehicle were submitted for analysis. Analysis of her clothing included SEM samples being taken from the cuffs of her shirt and the thigh areas of her blue jeans. After the SEM samples were taken, her clothing was scraped with a metallic spatula to recover any additional trace evidence. Analysis of the airbag included stereoscopic examination of the airbag cover for transferred fibers, SEM samples being taken from the interior of the airbag, and finally the airbag was scraped with a metallic spatula to recover any additional trace evidence.

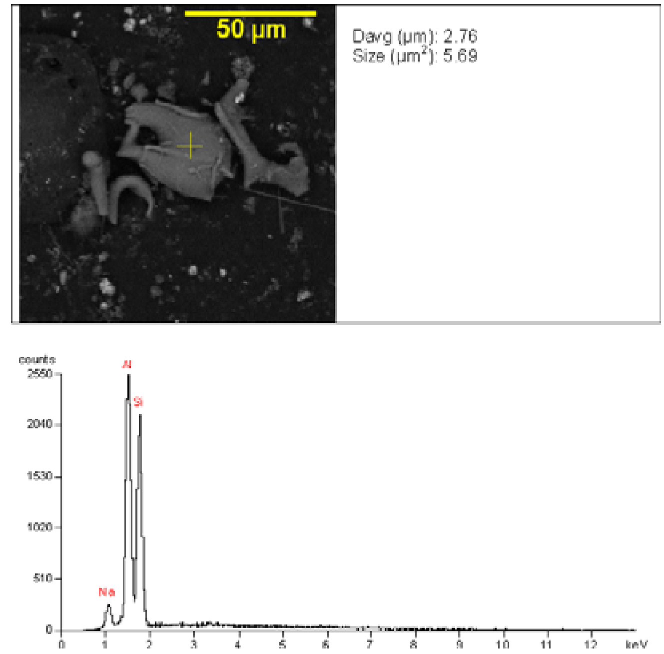

FIG. 22—*This mass of melted fibers is typical of those found in airbag residue standards. It has a spectrum similar to the aluminum/silicon microfiber spectrum with a trace of sodium.*

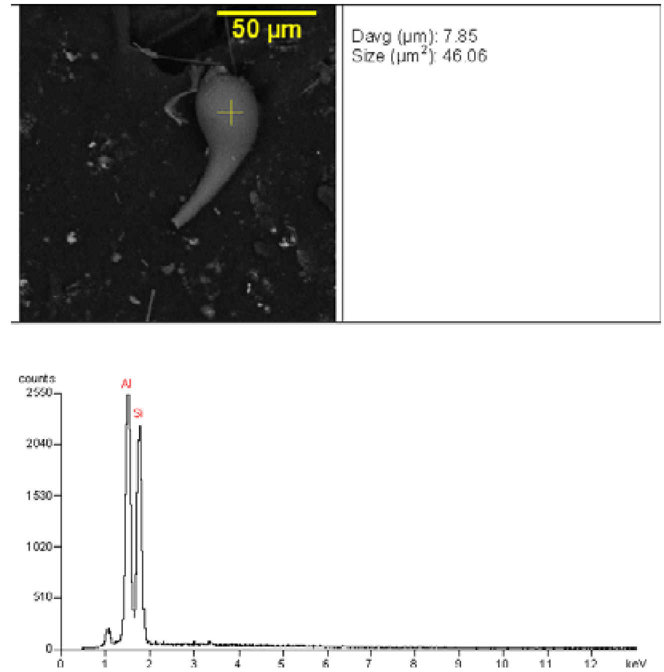

FIG. 23—*This particle has the typical melted appearance and the remnant of a "tail" that has broken off. The spectrum is aluminum and silicon with a trace of sodium (not marked). Particles of this size are visible using a stereomicroscope.*

Zirconium particles and aluminum silicon microfibers were identified in the SEM standard taken from the airbag and the SEM samples taken from both of the suspect's garments. Brittle, glassy spheres with elemental spectra of aluminum and silicon were recovered from the trace materials scraped from the airbag and both of the suspect's garments. Clearly, the materials produced by the airbag deployment were released into the passenger compartment of the vehicle and deposited onto the suspect's clothing. That trace material remained in place long enough for the clothing to be collected and the samples taken in the laboratory.

Discussion

The collection of evidence samples for airbag residue can be easily done using commercially available kits intended for collecting GSR evidence. Because of the particle nature of airbag residue, any research into GSR is also applicable. Positive test results for GSR indicate exposure to GSR, not necessarily who fired the weapon. Airbag residue on a suspect's clothing would indicate that they were present in the car during the accident, but not necessarily the driver. Likewise, excessive handling of the suspect's clothes or excessive amounts of time between the accident and the collections of samples may remove enough residues to affect the test results. Contamination issues should be considered during the collection of evidence samples and standards.

Airbag residue analysis is not as straightforward as GSR analysis. The "unique" airbag residue particle does not exist. No one particle type has been consistently identified in all airbag residue standards. Any particle that was detected could possibly be found in some other environmental sample. However, some particles identified in airbag standards have limited sources outside of airbag construction. Alloys of copper and cobalt have limited uses. The detection of high populations of copper-cobalt particles is likely from a nitrate source, such as the HACN formulation from an airbag. Other particles formed with trace elements in their compositions indicate the presence of these materials at the time of the reaction and may be probative. Zirconium particles that contain potassium and chlorine are likely from the ZPP formulation. Microfibers from airbag filters may yield valuable information. The presence of these fibers, and the identification of embedded particles, gives insight into their function. The proper collection of the airbag standard is essential for proper analysis.

In an effort to validate our procedure, background samples were taken from 30 test subjects who had recently traveled in vehicles. The only restriction was that the airbags in the vehicles were known not to have deployed. SEM samples were taken from the cuffs of the subjects' shirts or jackets and thigh areas of the subjects' pants. The samples were analyzed to determine the normal amounts of the particles in question which are typically found in airbag residue (Table 1). High populations of zirconium, strontium, aluminum, copper–cobalt, and cobalt are atypical for the clothing that was sampled. Background levels of copper, zinc, and iron make particles which contain those elements less probative. No aluminum silicon microfibers were detected.

TABLE 1—*Particles detected in validation study.*

Element	Average Number of Particles per Sample
Copper and cobalt	0.07
Cobalt	0.14
Strontium bearing	0.11
Strontium with titanium	0.45
Strontium with sulfur	1.28
Zirconium rich	0.63
Zircon (zirconium with silicon)	1.40
Aluminum	0.45
Copper	48.33
Zinc	278.19
Iron rich	1236.46

Particle populations identified in quantity in the airbag standard and also identified in samples taken from the hands or clothing of a suspect, along with trace and biological materials, may be the circumstantial evidence that indicates exposure to the known airbag source.

Acknowledgments

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