J Forensic Sci, May 2006, Vol. 51, No. 3
doi:10.1111/j.1556-4029.2006.00107.x
Available online at: www.blackwell-synergy.com

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Lead-Free Primer Residues: A Qualitative Characterization of Winchester WinClean™, Remington/UMC LeadLess™, Federal BallistiClean™, and Speer Lawman CleanFire™ Handgun Ammunition*

ABSTRACT: The elemental composition and postfiring residues of several "lead-free" or "nontoxic" centerfire handgun ammunition types currently available to the general public were examined. Offerings from Winchester, Remington/UMC, Federal and Speer were obtained from retail sources in both .45 ACP and 9 mm when possible. A total of 112 postfiring residue samples (SEM pins) were collected at varying distances from the muzzle, at two distances from target and from the shooter's hands. An additional 20 samples were collected by direct ignition of primers. Qualitative determinations were carried out using scanning electron microscopy (SEM)/electron dispersive spectrometry (EDS) analysis. All types tested contained aluminum (Al), silicon (Si), copper (Cu), and zinc (Zn). Most contain traces of sulfur and calcium. Winchester WinClean™ and Remington LeadLess™ contained potassium as the principle ingredient. Federal BallistClean™ contained barium, while Speer Lawman CleanFire™ contained strontium. In the main, these compared favorably with manufacturers' MSDS publications and patents granted. The characterizations undertaken here will be of use to the forensic electron microscopist as these formulations gain popularity.

KEYWORDS: forensic science, criminalistics, lead-free primer residue, gunshot residue, scanning electron microscopy, energy dispersive spectrometry

Airborne lead (Pb) encountered while using firearms, especially handguns, is an increasing health concern for those who shoot often or professionally (1). Aerosolized as a result of rapid heating and pressurization during cartridge discharge, lead vapors make their way into the bloodstream, and pose significant health risks. In an effort to combat this problem, most major handgun ammunition manufacturers offer "lead-free" or "nontoxic" options to the shooter. These reduce airborne lead by one of several means: enclosing the base of the projectile; enclosing the entire projectile with brass, copper (Cu), or gilding metal; fabricating the projectile from a sintered metal not containing lead, and removing lead styphnate from the primer mixture.

Traditional primer residue detection methods require lead (Pb), barium (Ba), and antimony (Sb) to assure a "unique" identification (2), be the method particle analysis using scanning electron microscopy/energy dispersive spectrometry (SEM/EDS) (3) or bulk analysis by atomic absorption spectroscopy (4). With lead removed, the forensic trace analyst is left without the tools necessary to achieve positive identification of these new ammunition types. Sintox™ (RAU Gammotec, Thun, Switzerland), introduced in Europe in 1980, has been studied (5) but is not commercially available in the United States. CCI Blazer CleanFire™ (Alliant Techsystems, Anoka, MN) has also been studied (6).

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*Presented at the 57th Annual Meeting of the American Academy of Forensic Sciences, New Orleans, LA, 2005. Preliminary WinClean findings were presented at the 80th Annual Meeting of the Georgia Academy of Sciences, 2003.

Received 23 July 2005; and in revised form 11 Oct. 2005; accepted 23 Oct. 2005; published 5 April 2006.

Analysis of all samples was done by SEM/EDS, consistent with previous work on particle-based analysis (7,8). The current work seeks to give the gunshot residue examiner the remaining tools necessary to achieve identification of new "lead-free" ammunition types currently available on the United States market. Given the health benefits of these formulations, it is likely that the examiner will encounter the need for positive identification in the very near future.

Experimental

Materials

Samples of each ammunition type (Remington/UMC LeadLess™ .45 ACP, 230 Gr. FNEB Part # LL45AP8, Lot # U19UBI and 9 mm 115 Gr. FNEB Part # LL9mm11, Lot # U17NCA; Winchester WinClean™ .45 ACP 185 Gr. BEB Part # WC 451, Lot # SK21 and 9 mm 147 Gr. BEB Part # WC 93, Lot # SH01; Speer Lawman CleanFire™ .45 ACP 230 Gr. TMJ Part # 53885, Lot # B21F21; Federal BallistiClean™ CQT .45 ACP 165 Gr. Compressed Cu, Part # BC45NT3, Lot # 3-38J070 and CQT 9 mm 100 Gr. Compressed Cu, Part # BC9NT3, Lot # 1-23J090) were obtained from commercial retail establishments (Outdoor Superstore.com, LLC, Clemson, SC; American Outdoor Adventures Inc., Albany, GA, and The Sports Authority, Englewood, CO). A 1911-A1 in blued steel with stainless .45 ACP barrel (Springfield Armory, Ganeseo, IL, frame # N41xxxx) and a Beretta model 92 FS with stainless barrel and lightweight aluminum frame in 9 mm (Beretta USA Corp., Ackk, MD, Frame #456xxxZ) were thoroughly field-stripped and cleaned before shooting each type using Hoppe BenchRest #9 Cu solvent (Coatesville, PA) followed by spraying with aerosolized tetrachloroethylene brake cleaner (CRC Brakleen™, part # 05089, CRC

Industries, Warminster, PA) and then a final washing with 90% isopropanol. Barrels were scrubbed with a phosphor–bronze brush and observed to be clean with a flexible fiber light. This was followed by repeated washings with isopropanol using clean cotton patches (Pro-Shot $2\frac{1}{4}''$ flannel, Taylorville, PA). All parts were allowed to air dry, and were re-assembled using a light coating of Boeshield T-9™ PTFE lubricant (PMS Products Inc., Holland, MI) on wear surfaces only. Trigger assemblies were not disassembled, but were sprayed out with brake cleaner and then washed with isopropanol and allowed to air dry.

Live Fire

Live fire was conducted in University forensic labs with the cooperation of campus police. Projectiles were retained in a fiber-filled bullet trap (Arli Spetstechnika Bullet Catcher mod # PU-1Mu, Moscow, Russia), and were recovered every two to four shots. A $\frac{1}{4}''$ thick, $12''$ square piece of paper-faced art board was affixed to the trap with a $1''$ square hole in the center to allow projectiles to pass unhindered. Conductive carbon sticky tabs (Ted Pella prod. # 16084–1) were affixed to Al pin mounts (Ted Pella prod # 16253, Lot # 017012) without touching the exposed surfaces. One of these was placed at 5 cm and another at 10 cm from the center of the board. The shooter's hands were scrubbed with tap water and liquid soap, rinsed with tap water followed by rinsing with de-ionized water. Two shots were taken at each 10, 30, 50, and 70 cm from the surface of the board. After each two-shot sequence, the shooter's hands were sampled within 1 min in an adjacent lab, through a closed door, with the same pin/tab arrangement. Left and right hands were dabbed by another researcher in the thumb, web and index finger areas at least 30 times or until the adhesive had lost its tack. The hands were again scrubbed and rinsed, to be followed by another sequence. Sample stubs were stored on a plastic-covered Styrofoam™ disc, placed in a large zippered freezer bag, and stored in a closed fume hood to await analysis.

Negative controls consisted of SEM pins with carbon tab attached. Handbanks were also collected from the shooter's hands some time after shooting concluded, having been scrubbed using the same protocol as above.

Direct Primer Ignition

Three or four cartridges from each type (for a total of 20) were disassembled using a kinetic bullet puller (Quinetics Corp., San Antonio, TX). The live primer was dissociated from the case by judicious use of a Dillon 450 reloading press (Dillon Precision Products, Scottsdale, AZ) and the appropriate die set (Dillon A58-14404 and 14399), having disassembled and cleaned the depriming die with isopropanol. Live primers were stored in cleaned, sealed film canisters. The powder, projectile, and cartridge cases were similarly retained in film canisters, each according to manufacturer and caliber.

Direct ignition of primers was accomplished by retaining an SEM pin/conductive carbon sticky tab in a wooden vice in a fume hood, fan off. The primer cup was held at approximately 10 cm from the stub, and then heated on the back side with a small hand torch fueled by Butane (Benz-O-Matic BF9, Lot # 2030, Newell-Rubbermaid Inc., Atlanta, GA) until ignition occurred. The fan was turned on for 5 min in between each ignition. Direct primer ignition samples were stored and analyzed separately.

Instrumentation and Methods

A CamScan 44 (Cambridgeshire, UK) fitted with a PGT Prism 2000 thin-window 10 mm^2 Si(Li) detector (Princeton Gamma Tech, Princeton, NJ, part # OPG009-1033) was used throughout. The accelerating voltage was held at 20 KeV with a working distance of 23 mm. The takeoff angle was maintained at 30°. Liquid nitrogen was purchased from Airgas (Randor, PA), as was compressed nitrogen for purging and SEM shocks. IMIX/Python software (PGT v. 10.594) drove the EDS.

MSDS reports were obtained either from corporate websites or from direct request from the manufacturer. A patent search for the manufacturers' primer compounds was undertaken through the USPTO Web site.

SEM researchers were not informed of the compositions indicated by the MSDS reports and patents. Having been introduced to primer residue from lead-based ammunition, they were instructed to search manually for spherical particles using backscatter mode at low magnification (× 50–150), and to inspect any particles that provided characteristic morphology and/or sufficient contrast. Direct primer ignition samples were analyzed separately. Of the 136

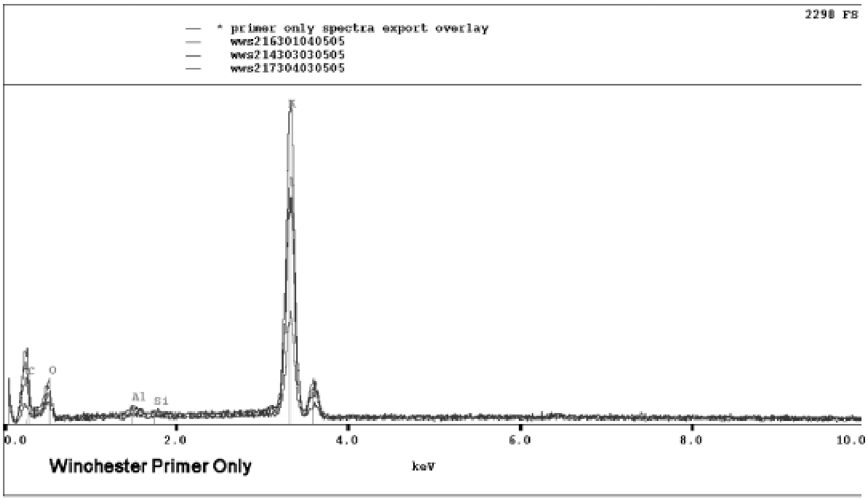

FIG. 1—*Winchester WinClean™ energy dispersive spectrometry overlay of primer-only pyrolysis residues.*

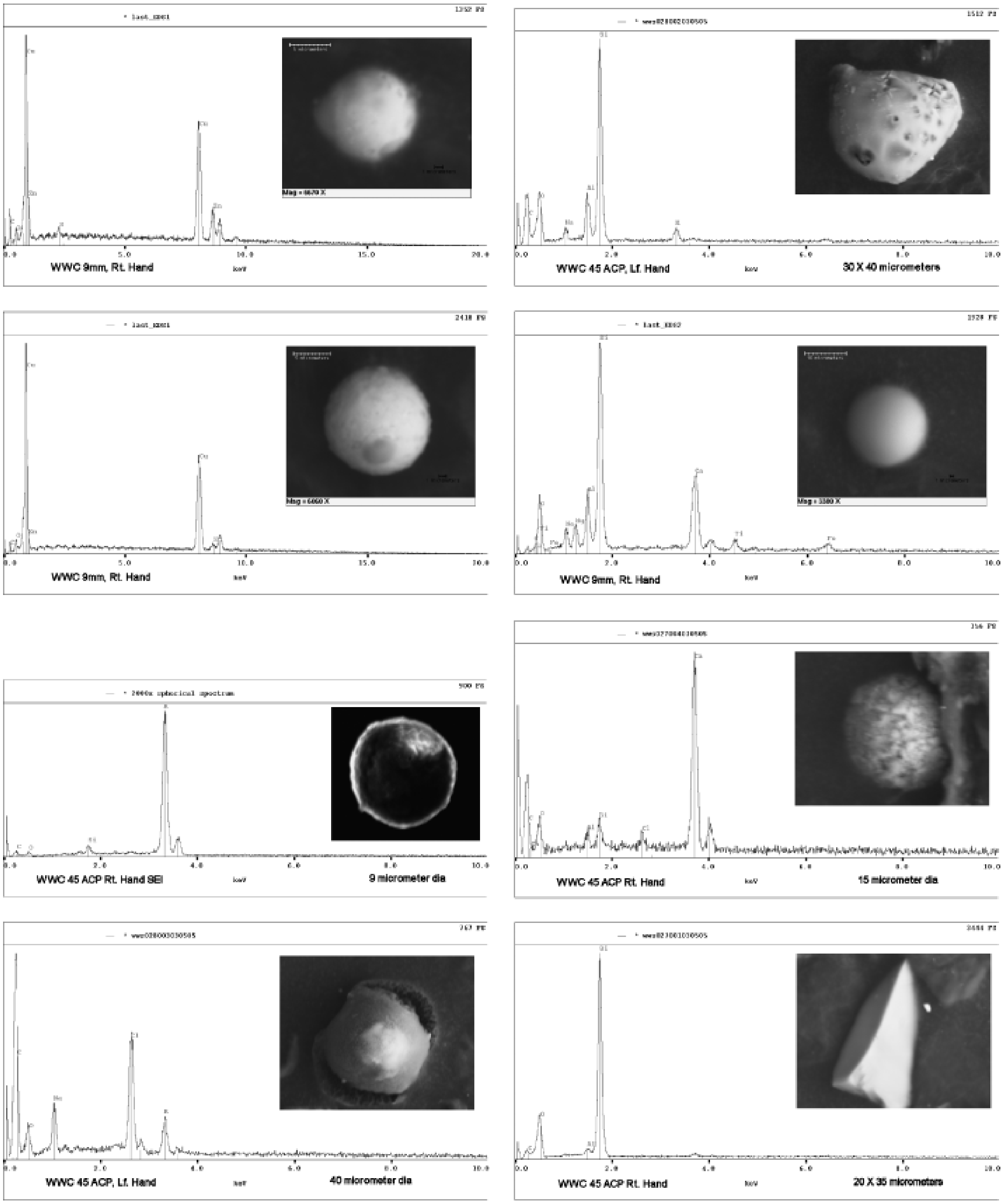

FIG. 2—*Typical Winchester particles recovered from shooter's hands with corresponding spectra.*

samples collected, 62 were analyzed in detail. Each sample so treated yielded at least 100 particles.

Results

Winchester WinClean™

Direct ignition of primers indicated potassium (K) as the major element. Traces of Al, silicon (Si), calcium, sulfur, zinc (Zn), Cu, nickel, chromium, magnesium, and iron were detected. Figure 1 is a representation of several spectra, overlayed for comparison.

Spherical particles were observed from shooter's hand samples, but were not in the majority. Figure 2 represents several particles with characteristic morphologies and spectra. It was usually the case that whole, smooth spheres contained Cu and Zn on the order of 0.5–6 μm. K-containing particles were generally not spherical, and were never smooth spheres. At best these appeared as "orange peel" spheroids or "teardrops" in the range 6–10 μm, but were usually larger, globular or rounded/angular in appearance. Large "sheet" particles in the 30–100 μm range containing only K were observed from downrange samples taken at close distances (5–20 cm). K- and S-containing particles were common, and were quite often accompanied by Al and Si as jagged, angular, 20 μm

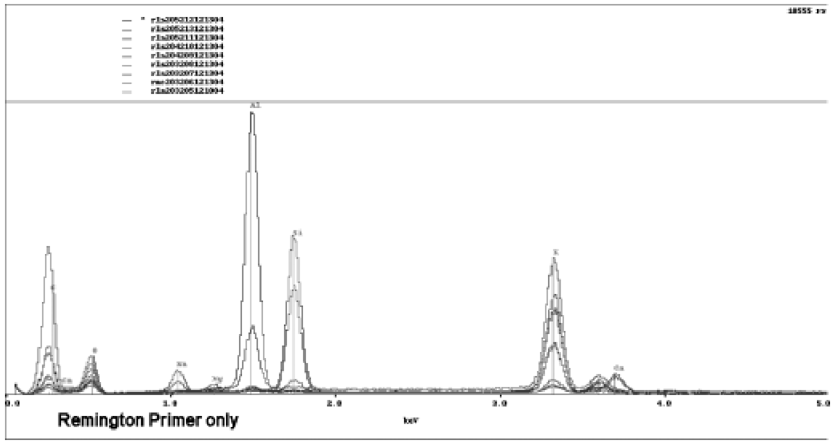

FIG. 3—*Remington/UMC LeadLess*^{TM} *energy dispersive spectrometry overlay of primer-only pyrolysis residues.*

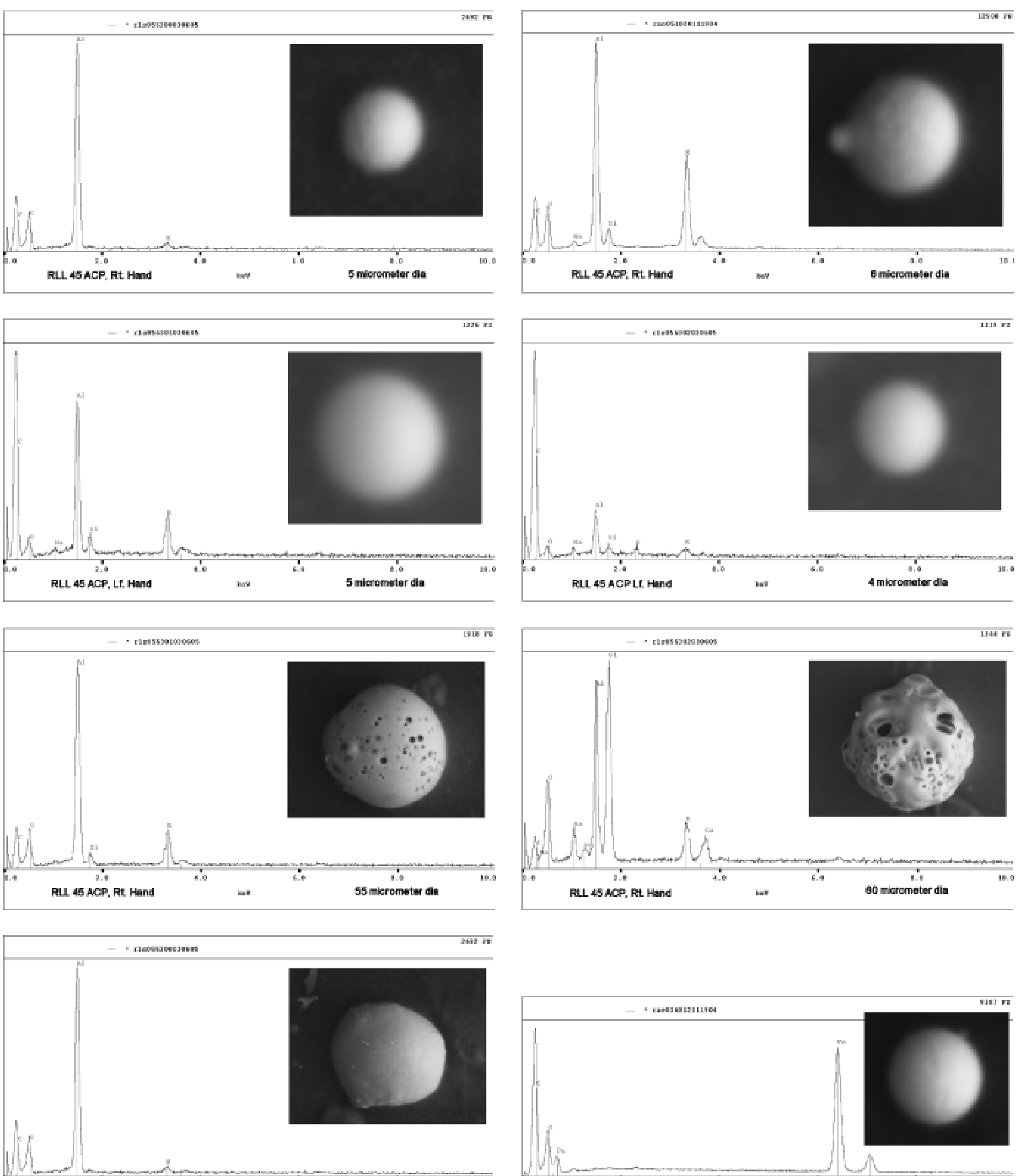

FIG. 4—*Typical Remington particles recovered from shooter's hands with corresponding spectra.*

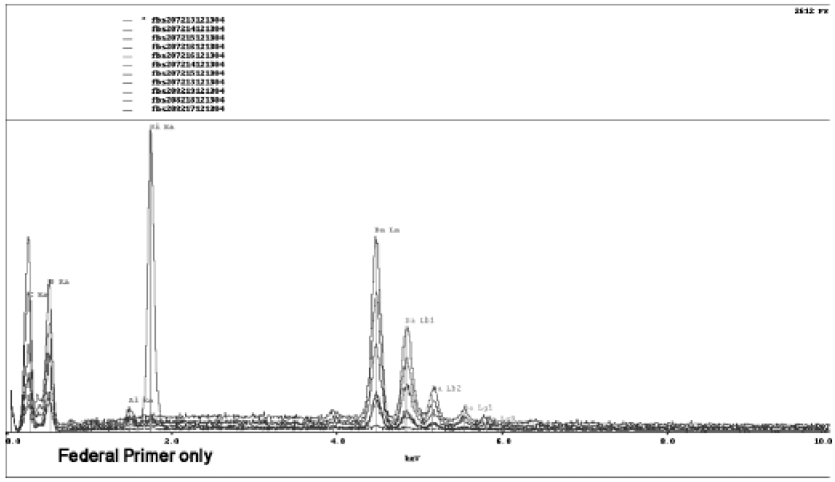

FIG. 5—*Federal BallistiClean™ energy dispersive spectrometry overlay of primer-only pyrolysis residues.*

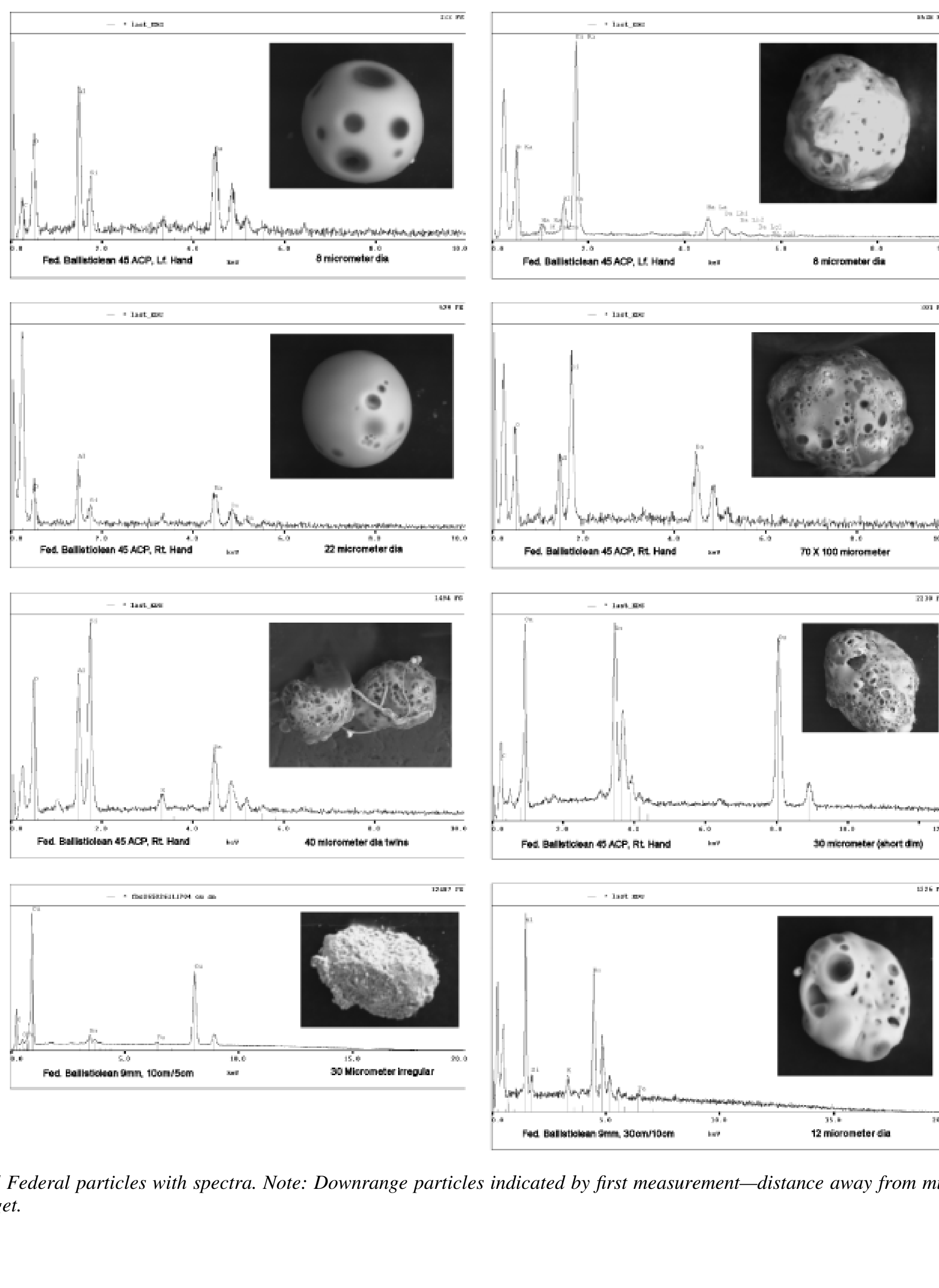

FIG. 6—*Typical Federal particles with spectra. Note: Downrange particles indicated by first measurement—distance away from muzzle, 2nd—distance away from center of target.*

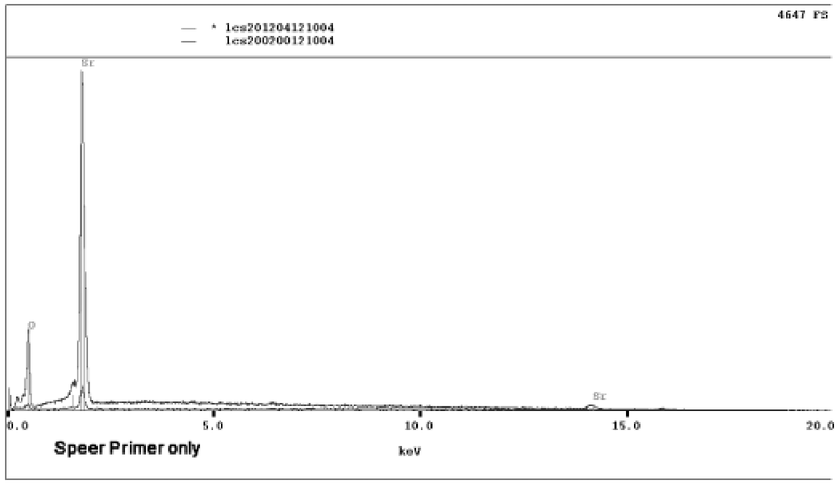

FIG. 7—*Speer Lawman CleanFire™ energy dispersive spectrometry overlay of primer-only pyrolysis residues.*

and larger particles. The vast majority of the particles contained varying proportions of K, Al, Si, and sodium (Na). These usually also contained some trace amounts of sulfur, calcium, and magnesium, especially if the size exceeded 10 µm. Though morphologies varied widely, the characteristic K, Al, Si, Na composition was remarkably uniform throughout.

This composition grouping offered little in the way of BSE contrast. Anything that afforded backscatter electron contrast such as the microscopist is familiar with from lead-based work was usually Cu+Zn, with the occasional Ni trace, and quite small (0.5–6 µm). Particles lacking K, i.e., Al plus some light metal or salt, were spherical. These appear as whole, smooth spheres in the 6–10 µm range, and piled smaller spheres or "globs" in the 20 µm and up range.

Boron, listed in the MSDS and patent, was detected with this equipment from the unexploded raw priming mixture and from a boric acid control as a shouldered carbon peak at approximately 185 eV (Kα1). It was not observed in primer-only pyrolysis residues, perhaps due to low concentrations in the sample, or to instrumental operating parameters. Thus, boron detection through single-particle analysis from live fire samples was neither expected nor observed with this equipment under these operating conditions.

Remington/UMC LeadLess™

Direct examination of primer ignition revealed Al, Si, K, Na, calcium, and traces of magnesium. Figure 3 represents several spectra overlayed for comparison.

Spherical particles were more frequently found with Remington. Figure 4 characterizes typical particle morphology with accompanying spectra. The smallest, in the 1–6 µm range, consisted of Cu and Zn, as was the case with Winchester. The addition of more Al to the primer mixture appears to have generated more frequent spherical particles in the 5–10 µm range on shooter's hands. Particles evidenced a more spherical, smooth shape concordant with Al content, while more silicon tended to indicate a "pocked" or more angular particle. The vast majority of particles in the 5–15 µm range contained Al, Si, and K in some ratio, usually with traces of Na. Those in the 20+ µm range tended to be less smooth in texture, though composition was similar to the rest. It was not unusual to find Na, calcium and magnesium salts, usually combined with K or Al/Si, or both. Downrange samples varied considerably more in morphology, from spherical to flat and angular. Throughout, the composition was similar to those collected on hands. The vast majority of particles regardless of size and morphology consisted of Al, K, Si, and Na.

Federal BallistiClean™

Direct examination of primer-only pyrolysis revealed Ba, Si, and Al as principle ingredients. Figure 5 represents several spectra overlayed for comparison.

The vast majority of the particles recovered from the hands were of a "pocked," spherical morphology varying in size from 4–10 µm. Figure 6 characterizes typical particle morphology with accompanying spectra. These were usually composed of Al, Si, Ba, and K with traces of Na, regardless of size. Na did not generally appear in sufficient quantity to register definitively until particle size reached 6 µm. Nonspherical, irregular but smooth particles were detected frequently. They appeared as ovular or spheroid in overall shape with rounded angular facets and heavily pocked surfaces. These were predominantly in the 15–40 µm range. Composition of these was unchanged from above. Downrange samples varied considerably more in size and shape, but did not change in characteristic Al, Si, Ba, K composition. This composition was also observed as larger "piled" or "globular" particles in the 20–40 µm range. "Pocked," irregular or "globular" particles containing Al/Si and Ba in some ratio from 10–35 µm were common. Spherical Cu plus tin (Sn) particles from 1–6 µm were observed.

Speer Lawman CleanFire™

Direct examination of primer-only pyrolysis indicated strontium (Sr) and oxygen as the principle ingredients. Figure 7 represents characteristic spectra overlayed for comparison.

Sr-containing spheroids in the 3–20 µm range were recovered consistently from shooter's hands after live fire. Figure 8 is a collection of particles typical of those recovered from the shooter's hands and from downrange. These take the form of spheres with fissured or "crackled" surfaces. The Sr is often accompanied by Al/Si and chlorine. This is consistent with previous work with CCI lead-free ammunition (9). CCI and Speer are owned by the same company, Alliant Techsystems (ATK). Downrange samples evidenced the same tendency toward spherical shape, but larger

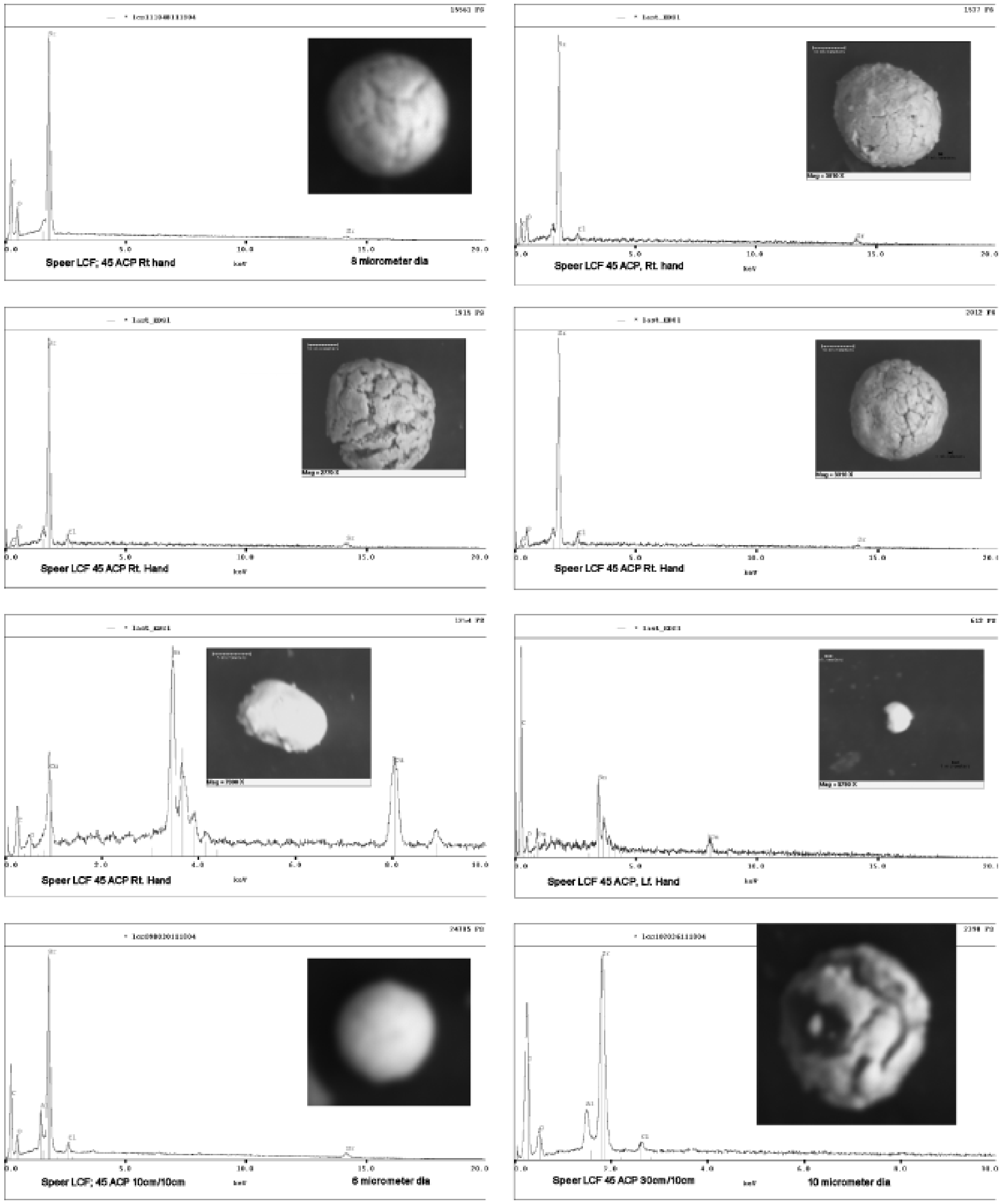

FIG. 8—*Typical Speer particles recovered from shooter's hands with spectra. Note: Downrange particles indicated by 1st measurement—distance away from muzzle, 2nd—distance away from center of target.*

particles were more common. Though still composed almost exclusively of Sr+oxygen, Sr+chlorine, or Sr+Al/Si, a more marked crackling of the surface was observed. Spheroids often exceeded 20 µm and were heavily fissured, especially at close range (5–20 cm). Cu only or Cu+Zn particles in the 2–5 µm range were common both on shooter's hands and downrange.

MSDS, Patent, and Experimental Result Comparison

Table 1 provides a quick reference guide comparing MSDS-indicated composition, patent-indicated composition, and results reported herein. It can be seen that these compare quite favorably. It should be noted that Ba is indicated in Remington's formulations while none appeared in experimental ignition.

Case and Projectile Composition

The interiors of the Remington and Speer cartridge casings revealed a black, semiflexible polymer coating (presumably a sealant), consisting primarily of sulfur. Excess polymer could be seen inside the cartridge casing as a ring around the area where the base of the projectile had been seated. That portion of the projectile in contact with this sealant retained some of the compound after pulling from the assembled case.

Federal uses the same primer size for both the 9 mm and 45ACP cartridges tested. This was accomplished by means of a ring of casing material in the larger primer cup, used as a spacer.

Remington and Speer used a Cu-only jacketing material for their lead projectiles. The Speer projectile is a completely

TABLE 1—*MSDS, patent, and experimental result comparison.*

	Experimental Results	MSDS* Contents	Patent Claim†
W/WinClean™	K, Al, Cu, Zn, S	Cu, Zn(cup) DDNP Potassium nitrate Boron Nitrocellulose	Cu, Zn (cup) DDNP Potassium nitrate Boron Nitrocellulose or nitroglycerine calcium silicide or tetracene
R/UMC LeadLess™	Al, Si, K, trace Ca, Na, Mg	Cu, Zn (cup) DDNP Barium Tetracene Nitrocellulose Nitroglycerine	(mix only) DDNP Tetracene Pentaerythrityl tetranitrate (PETN) or Aluminum or Antimony sulfide Nitrocellulose Barium nitrate Binders
F/BallistiClean™	Ba, Si, Al	Cu, Zn (case) DDNP Tetracene Barium nitrate Aluminum Nitrocellulose Nitroglycerine	(mix only) DDNP Tetracene Barium nitrate Aluminum Nitrocellulose Nitroglycerine Ground glass Gum tragacanth
SL CleanFire™	Sr, O, Al, Cl	Cu, Zn, Ni DDNP Tetracene Strontium nitrate Nitrocellulose Nitroglycerine	(mix only) DDNP Tetracene Strontium nitrate Nitrocellulose Nitroglycerine

*WinChester WinClean™ MSDS: Olin Corporation # 00062.0001, revision #7 Remington LeadLess™ MSDS: Remington Arms Centerfire Rifle, pistol & revolver loaded round (LeadLess), revised October, 2001. Federal BallistiClean™ MSDS: Federal Cartridge Company, small arms ammunition BallistiClean centerfire pistol ammunition, issued January 2, 2004. Speer Lawman CleanFire™ MSDS: CCI-Blount Inc. Sporting equipment group, Rifle-pistol primers, CleanFire, revised June 11, 1998.

†WinChester WinClean™ U.S. patent #5,417,160: Mei, George C. (St. Louis, MO); Pickett, James W. (Gillespie, IL); Olin Corporation, May 23, 1995.

Remington LeadLess U.S. patent # 5,684,268: Lopata, Frances G. (Little Rock, AR); Remington Arms Company Inc. (Madison, NC), November 4, 1997. Federal BallistiClean™ U.S. patent # 5,831,208: Erickson, Jack A. (Andover, MN); Federal Cartridge Company (Anoka, MN), November 3, 1998.

Speer Lawman CleanFire U.S. patent # 4,963,201: Bjerke, Robert K. (Lewiston, ID); Ward, James P. (Lewiston, ID); Ells, Delbert O. (Clarkston, WA); Kees, Kenneth P. (Lewiston, ID), Blount Inc. (Montgomery, AL), October 16, 1990.

enclosed, ball type. The Remington projectile is a truncated ball with lead core exposed on the forward end. Winchester uses a lead projectile in a truncated cone shape, covered in brass with lead exposed at the nose. Federal uses a continuous sintered bronze projectile in a truncated cone shape. Each uses an uncoated brass cartridge case. Table 2 is a summary of these findings.

Negative Controls

Carbon sticky tab examination revealed nothing save carbon with a small amount of oxygen. Although the occasional discrete particle was observed, it proved to be carbon uniformly. Please see Fig. 9 for a spectral overlay of carbon blanks.

TABLE 2—*Cartridge case and projectile covering comparison.*

Manufacturer	Cartridge case	Projectile	Projectile Covering
Winchester	Cu, Zn	Pb(+traces)	Cu, Zn
Remington	Cu, Zn	Pb(+traces)	Cu, (trace S)
Speer	Cu, Zn	Pb(+traces)	Cu, (trace S)
Federal	Cu, Zn	Cu, Sn	n/a

Handblank samples showed hundreds of particles, all some form of chloride salt. These were uniformly Ca, K, or Na, with Na taking the usual form of a cube or "piled" squares. KCl particles tended to be oblong and angular. Ca+KCl particles appeared as "piles" of smaller crystals. Varying in size from 2 to 30 μm, compositions were uniformly chlorinated salts. No transition metals were detected, nor was Al.

Discussion

The presence of Al in postfire hand samples using the Speer ammunition was a concern. It was not discovered in the primer mixture, and appeared in a firearm known to contain no Al in high-pressure/high-temperature areas. An inspection of the powder flakes under low-power reflected light revealed the presence of small, bright silver-colored particles. A dozen or so flakes were placed on a stub as above, and then in the SEM for elemental analysis. Backscatter electron imaging immediately revealed these particles as being interspersed throughout the flakes, not simply adhered to the surface by seating pressure against the projectile or casing. Figure 10 illustrates these particles distributed throughout the powder. Closer examination yielded several compositions of metal embedded in the powder; including Al, iron, titanium, and Cu. Figure 11 represents four of these particles with accompanying spectra.

The presence of Al and other metals in the powder charge is not unexpected. Although this sort of particle-within-particle analysis has not been performed on powder flakes to the authors' knowledge, others have reported the presence of trace metals from powder pyrolysis (10). It is unclear why these metals are present. It may be the case that the manufacturer added them deliberately, as a method of increasing flame temperature. Or, these may be an unintended artifact of powder manufacturing.

Most primer residue examination is done via screening using an automated computer program that observes characteristic contrast, composition, and morphology in backscatter electron mode, followed by detailed examination by a trained microscopist (11). These software applications will have to be recalibrated to include these new ammunition types. Careful, trained examination will prove much more important, as differentiating these residues from pyrotechnic products (12) is not straightforward.

Confusion with sparkler pyrolysis residues, perhaps resulting in a false positive, is possible if automated systems are heavily relied upon. Figure 12 is an image/spectra pair from sparkler analysis carried out in 2003. This particle grouping could be mistaken by computer software for Federal BallistiClean™. As a practical matter, the large (30 μm) particle is a hollow sphere largely composed of iron, with smaller (2–4 μm) spheres of Ba, Al, and Si attached, and thus can be distinguished from the continuous single-particle composition of the Federal residues. It should thus pose no serious problem for the trained electron

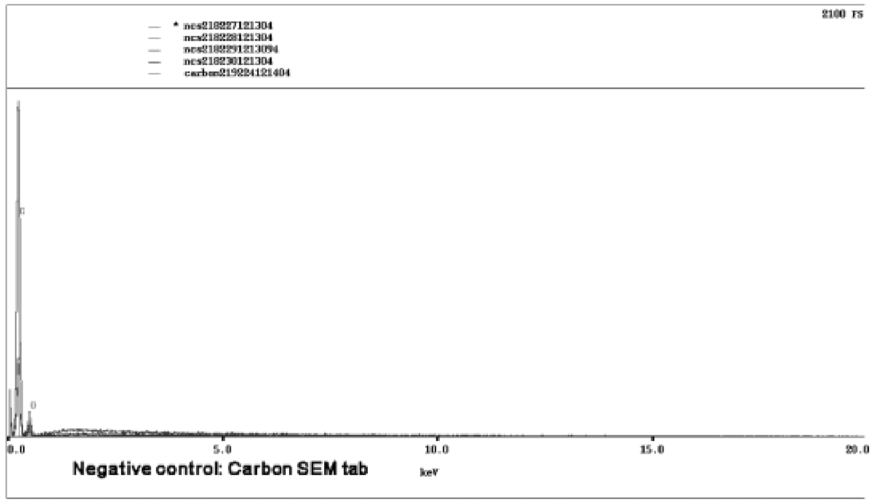

FIG. 9—*Negative control spectral overlay.*

microscopist, but may provide a false "hit" from automated searches.

Primer residues containing K without Al may not provide sufficient BSE contrast for automated identification, which may lead to a false-negative result. Additionally, K, Ca, Mg, and Na salts are easily soluble in water (and thus human sweat), and will not be expected to persist on shooters' hands as long as those that are not soluble.

It is our opinion that the Winchester and Remington "lead-free" ammunitions are indistinguishable on a single-particle basis. Though the Remington contains more Al in the primer mix and evidences a greater number of spherical particles, these are not exclusive to this ammunition type. Sufficient miscibility in the gaseous state exists such that any number of distributions from the elements present could result upon cooling, and thus deposition. Detecting a particle with 30% Al will not distinguish Remington from Winchester. If a large number of particles are available, this type of analysis may be possible.

One must also think of this study as a per-lot-number analysis. Though each manufacturer seeks to provide a unique or better product, it is often the case that a single manufacturer may have several patents to their name for priming mixtures, or may indeed have several versions of a single primer mixture in a single patent. It may also be the case that in times of expediency, one ammunition manufacturer may purchase components from several suppliers to meet demand or schedule. That said, the vast majority of what each manufacturer provides for sale must meet MSDS restrictions, and thus can be thought of as consistent, but in no way unique, to a single manufacturer.

Finally, environmental cross-identification from lead-based primer residue has been well studied (13). This type of examina-

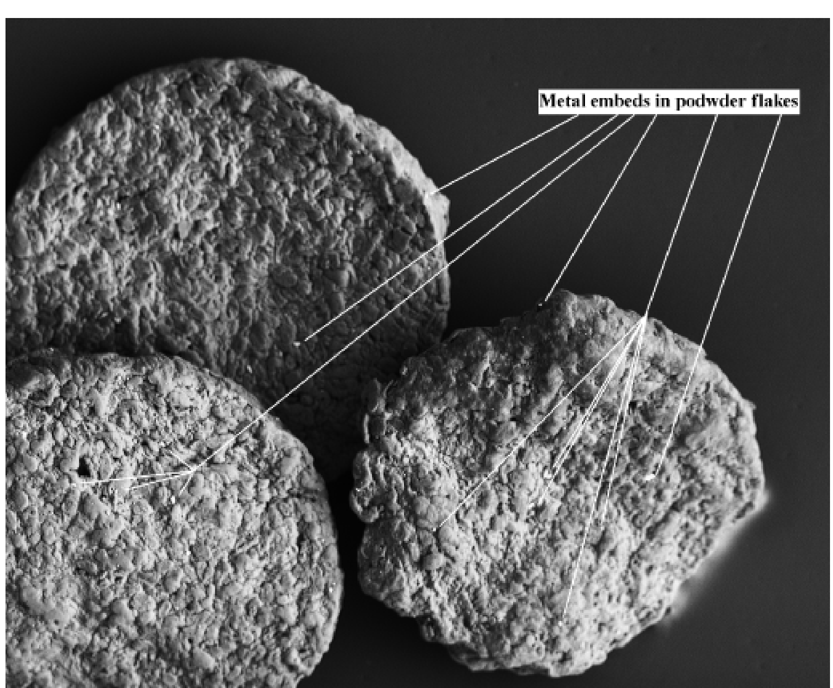

FIG. 10—*Speer Lawman CleanFire*^{TM} *powder flakes with metal inclusions indicated.*

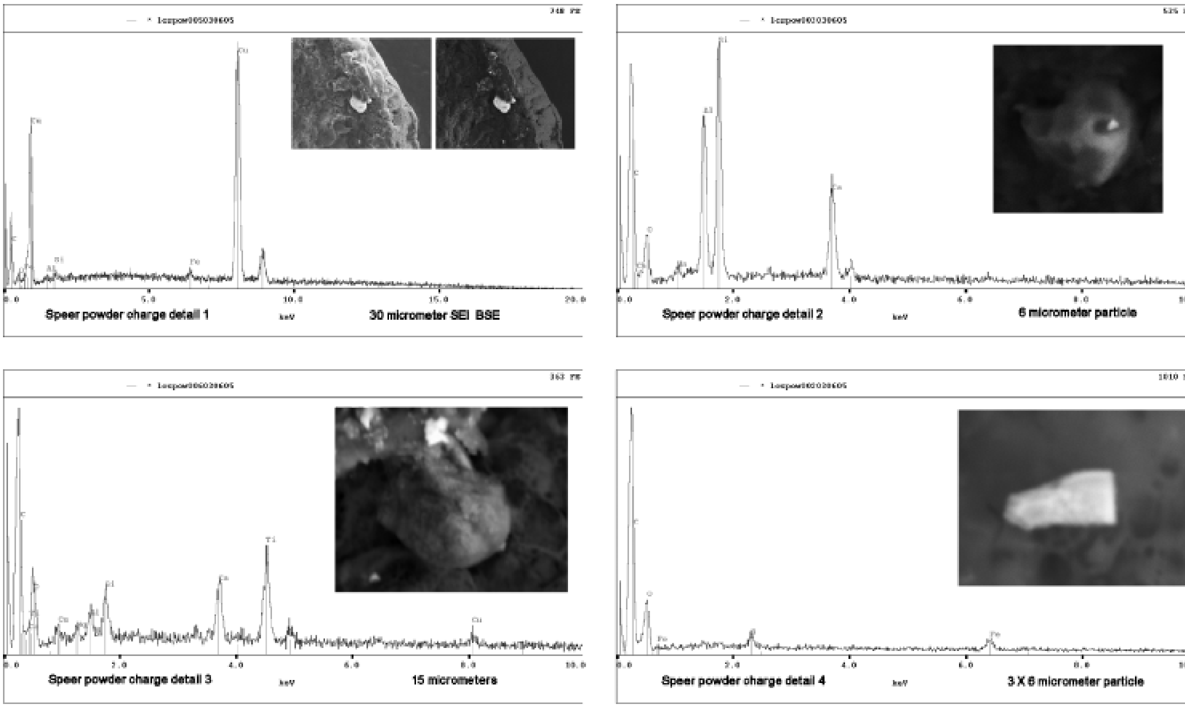

FIG. 11—*Speer Lawman powder charge metal embeds with spectra.*

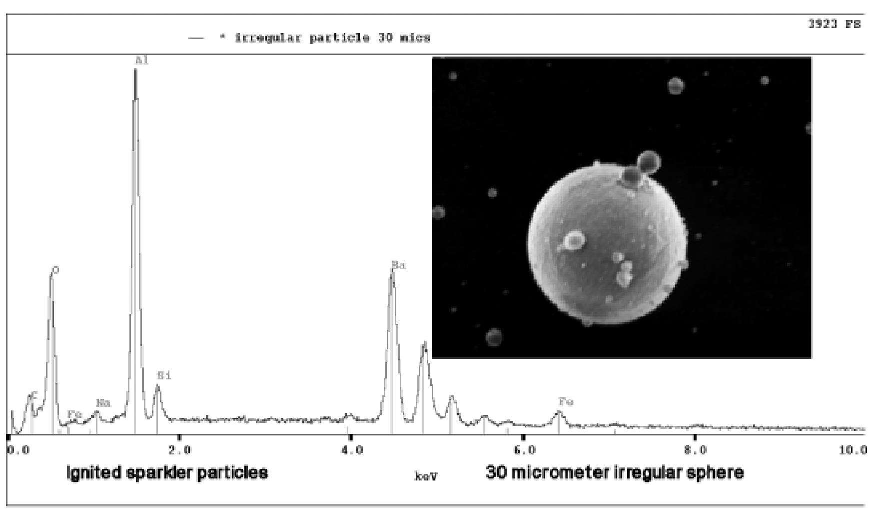

FIG. 12—*Ignited sparkler image/spectral pair, 30-μm iron sphere with Ba/Al/Si spheres attached.*

TABLE 3—*Summary of results.*

			Morphology	
	Particle Elements	Shape	Size (μm)	Texture
Winchester	Cu+Zn	Spherical	0.5–6	Smooth
WinClean™	K+Al+Si+Na	Spheroid/"teardrop"	6–10	Orange peel
	Al+Na, Ca, or Mg	Spherical	6–10	Smooth
Remington	Cu+Zn	Spherical	1–6	Smooth
LeadLess™	Al, Si, K (trace Na)	Spherical	5–15	Smooth/pocked
	Al, Si, K (Na or Ca)	Varied	20+	Smooth to angular
Federal	Cu+Sn	Spherical	1–6	Smooth
BallistiClean™	Al, Si, Ba, K (Na)	Spherical	4–10	Heavily pocked
	Al, Si, Ba, K (Na)	Irregular (globular)	10–35	"Rockpile"
Speer Lawman	Cu or Cu+Zn	Spherical	2–5	Spherical
CleanFire™	Sr, Al/Si, or Cl	Spherical	3–20	Crackled
	Sr, Al/Si, or Cl	Spheroid	20+	Heavily fissured

tion must now be completed for these new ammunition formulations.

Conclusions

SEM/EDS analysis of commercially available "Lead-free" handgun ammunition reveals the elemental compositions of Winchester WinClean™, Federal BallistiClean™, Remington Lead-Less™, and Speer Lawman CleanFire™. Table 3 reflects a summary of these results. All contain some combination of Al, Si, Cu, and Zn. Most contain traces of sulfur and calcium. All are expected to have 1–6 μm Cu+Zn spherical particles from cartridge casings, primer cup, and/or projectile jacketing. Cu+Sn spherical particles in the same size range can be anticipated from Federal, originating from the projectile composition.

Winchester and Remington primer residue particles contain K as a differentiating ingredient while offering few spherical particles containing all of the above. Spherical particles tend to contain more Al, and thus more would be more likely with Remington. Characteristic Federal particles contain Ba as well as Al, Si, Cu, and Zn. The morphology of these is distinct, as all are heavily pocked or cratered. Those in the normal primer residue size range (1–10 μm) are smooth in texture while retaining the pocked surface characteristics. Speer contains Sr as the principle metal and is easily recognized by spherical, "crackled," or "fissured" morphology.

Further study is indicated. Environmental cross-identification, e.g., Sr sources from fireworks, tracers, flares, etc., must be examined in detail, as was the case with the seminal Aerospace report on lead-based primer compositions. A study of the persistence of these new types of residues on the hands should be conducted, especially those that contain K. A per-manufacturer analysis of powder charge metal contents should also be undertaken.

Acknowledgments

The authors would like to thank Albany State University and the Department of Criminal Justice and Forensic Science for the use of their equipment and their kind considerations during our research. Separately, we would like to thank the undergraduate students in the Forensic Science program for their assistance and enthusiasm. Funding for consumables was provided by a Title III grant from the department of planning and research, Albany State University.

References

1. Abudhaise BA, Alzoubi MA, Rabi AZ, Alwash RM. Lead exposure in indoor firing ranges: environmental impact and health risk to the range users. Int J Occup Med Environ Health 1996;9(4):323.
2. Schwoeble AJ, Exline DL. Current methods in forensic gunshot residue analysis. Boca Raton, FL, USA: CRC Press; 2000.
3. Wolten GM, Nesbitt RS, Calloway GL, Loper GI, Jones PF. Particle analysis for the detection of gunshot residue. 1: scanning electron microscopy/energy dispersive x-ray characterization of hand deposits from firing. J Forensic Sci 1979;24(2):409–22.
4. Heard BJ. Handbook of firearms and ballistics: examining and interpreting forensic evidence. New York, USA: John Wiley and Sons; 1997.
5. Gunaratnam L, Himberg K. The identification of gunshot residue from lead-free Sintox ammunition. J Forensic Sci 1994;39(2):532–6.
6. Charpentier B, Desrochers C. Analysis of primer residue from lead free ammunition by X-ray microfluorescence. J Forensic Sci 2000;45(2):447–52.
7. Basu S. Formation of gunshot residues. J Forensic Sci 1982;27(1):72–91.
8. Wolten GM, Nesbitt RS, Calloway AR, Loper GL, Jones PF. Final report on particle analysis for gunshot residue detection. ATR-77(7915)-3. El Segundo, CA, USA: The Aerospace Corporation; 1977.
9. Harris A. Analysis of primer residue from CCI blazer lead-free ammunition by scanning electron microscopy/energy dispersive X-ray. J Forensic Sci 1995;40(1):27–30.
10. Miyauchi H, Kumihashi M, Shibayama T. The contribution of trace elements from smokeless powder in post firing residues. J Forensic Sci 1998;43(1):90–6.
11. Germani MS. Evaluation of instrumental parameters for automated scanning electron microscopy/gunshot residue particle analysis. J Forensic Sci 1991;36(2):331–42.
12. Kosanke KL, Dujay RC, Kosanke B. Characterization of pyrotechnic reaction residue particles by SEM/EDS. J Forensic Sci 2003;48(3):531–7.
13. Wolten GM, Nesbitt RS, Calloway AR, Loper GL. Particle analysis for the detection of gunshot residue. II: occupational and environmental particles. J Forensic Sci 1979;24(2):423–30.

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