Errors in gunshot residue assessment by scanning electron microscopy/elemental analysis in criminal cases: II. Missed tin (Sn) and antimony (Sb) in an unusual, non-gunshot residue population of particles containing phosphorus (P).

By Bryan Burnett

Automated scanning electron microscope analyses of gunshot residue samplers are being performed by many crime laboratories all over the world. Often the criminalist-technician operators of these instruments do not have adequate training for the interpretation of spectra generated by energy dispersive X-ray spectroscopy. In this case, a number of unusual particles, most of which contain phosphorus (P) and tin (Sn) were assigned by the criminalist as being "unique" to gunshot residue. Unfortunately, none of the particle spectra recorded in this case can even be assigned as "consistent," much less "unique" to gunshot residue. Errors made by this technician are 1) missed elements, tin (Sn) and antimony (Sb), and 2) including phosphorus (P) as a gunshot element. The origin of these particles is unknown.

Introduction

The defendant, while wearing gloves, was alleged to have shot the victim with a 9 mm pistol (this is case 2 in part I of this article series). The ammunition was Winchester 9mm with copper-jacketed bullets. Gloves were found in the defendant's trashcan. Scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) were performed on these gloves as well as an additional pair of gloves of the same type that was found associated with the defendant's brother. The gloves and the defendant's hands were sampled for gunshot residue. The criminalist-technician testified that he found eight particles out nine recorded in these six samples that he described as "unique" gunshot residue particles (Table 1) .

Table 1. *Listing of elements present in each of the nine gunshot residue spectra submitted by the technician in this case. The boxes filled with yellow identify elements present in the spectra, but the technician had failed to identify in his report. For the spectra of particles 7, 8 and 9, the technician did not suggest element compositions, but assigned a "unique" status to each.*

		PRIMARY GSR ELEMENTS			SECONDARY GSR ELEMENTS				PROHIBITED GSR ELEMENTS				
#	PARTICLE SOURCE	Pb	Sb	Ba	K	Fe	Cu	Zn	P	Sn	PARTICLE SHAPE	NOTES	FIG. #
1	DEFENDANT'S HAND, L.	●	●	●	●	●	●	◦	●	—	irr	Pb//SbBa FeCu "UNIQUE"	1D
2	DEFENDANT'S HAND, L.	●	◦	●	●	◦	—	—	●	—	sph	Pb//Ba K Ca	2
3	DEFENDANT'S HAND, R.	●	●	●	—	◦	◦	—	—	●	sph	PbSbSnBa//FeCu "UNIQUE"	1A
4	DEFENDANT'S HAND, R.	●	●	●	●	—	◦	◦	—	—	sph	PbBa//SbCu "UNIQUE"	
5	DEFENDANT'S GLOVE	●	●	●	●	◦	◦	—	●	●	irr	PbSbBa//FeCu "UNIQUE"	1C
6	DEFENDANT'S GLOVE	●	●	●	—	—	●	—	—	●	irr	Pb/SbSnBa//Cu "UNIQUE"	
7	BROTHER'S GLOVE	●	◦	◦	◦	—	◦	—	●	—	sph	"UNIQUE"	
8	BROTHER'S GLOVE	◦	●	●	●	●	—	—	●	◦	irr	"UNIQUE"	
9	BROTHER'S GLOVE	?	●	●	●	◦	◦	—	◦	●	irr	As? "UNIQUE"	1B

● MAJOR OR INTERMEDIATE-SIZED PEAK
◦ MINOR PEAK
— ELEMENT NOT PRESENT
? ELEMENT'S PRESENCE UNCERTAIN

Tin (Sn)

Particles, whose spectra were alleged to be "unique" to gunshot residue, were found on the six samplers examined in the SEM for this case (Table 1). Of the nine alleged gunshot residue particles, five had spectra that showed the presence of tin. The technician indicated that only two of those particles contained tin (e.g., Fig. 1A), yet he still claimed these two particles "unique" to gunshot residue. Tin is not known to be used in Winchester center-fire primers (1). He also appeared to have missed the other tin-containing particles.

How could tin be missed in some of these spectra? The presence of a relatively large amount of potassium (K) as well as antimony (Sb) in many of these spectra masks tin (e.g., Figs. 1B & 1C). For the spectrum of a particle without tin (e.g., Fig. 1D), note the deep gap in the spectral area between potassium K-α and the antimony L-α peaks. The gap is not present in the same region for the spectra of tin-containing particles shown in Figs. 1B and 1C.

There are no reports of tin as being a component of Winchester cartridges, indeed, the Winchester Material Safety Data Sheet (1) for their cartridges does not contain any reference to tin, either as metal or a compound.

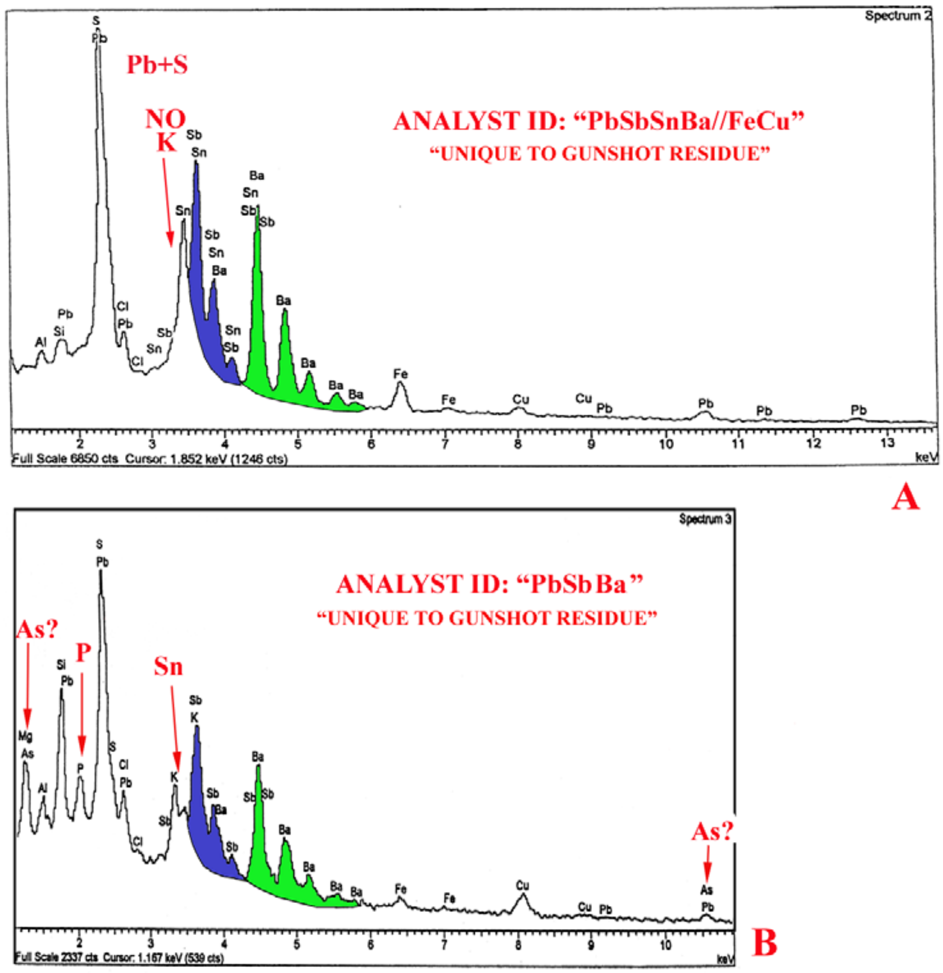

__Figure 1.__ Spectra of four particles identified by the technician analyst as "unique" to gunshot residue. The Sb and Ba peak areas are colored in these spectra to assist in the comparisons. technician assigned elements are noted in each spectrum. A: Particle spectrum (Table 1, #3) with lead (Pb), antimony (Sb) and barium (Ba). Tin (Sn) is present without potassium (K). B: Spectrum of a particle (Table 1, # 9) that has a tin (Sn) component apparently missed by the technician. This spectrum was also used in the previous article in this series. The spectrum above 11 keV was cut off by the technician.

Antimony (Sb) & Calcium (Ca)

Spectra that have both calcium and antimony under some circumstances can present a challenge where it is difficult to separate these peaks without careful scrutiny (by an experienced analyst) or by computer deconvolution. In this case, the technician obviously did not perform either for the interpretation of one of the spectra (Fig. 2) from this case. The region of interest in this spectrum is enlarged (Fig. 2, insert) and the K lines are drawn in for potassium (K) and calcium (Ca) and the L-α and L-β lines for antimony (Sb). The questioned peak is offset slightly to the right of the antimony L-α line. The calcium K-α line is offset (does not align) to the right of that peak. A calcium K-β peak is not discernable. However, a small peak that corresponds to the antimony L-β line is present.

It would, of course, be preferable to run a computer deconvolution routine to do a separation of the possible calcium and/or antimony elements that are possibly present in this particle. Since a computer deconvolution is not possible here, the manual placing K- and L-lines for calcium and antinomy (Fig. 2) suggest a relatively small amount of calcium in relation to antimony that has set the questioned peak nearer to the antimony L-α line than the calcium K-α line. The lack of a discernable calcium K-β peak and the presence of a peak at the antimony L-β line support this assessment.

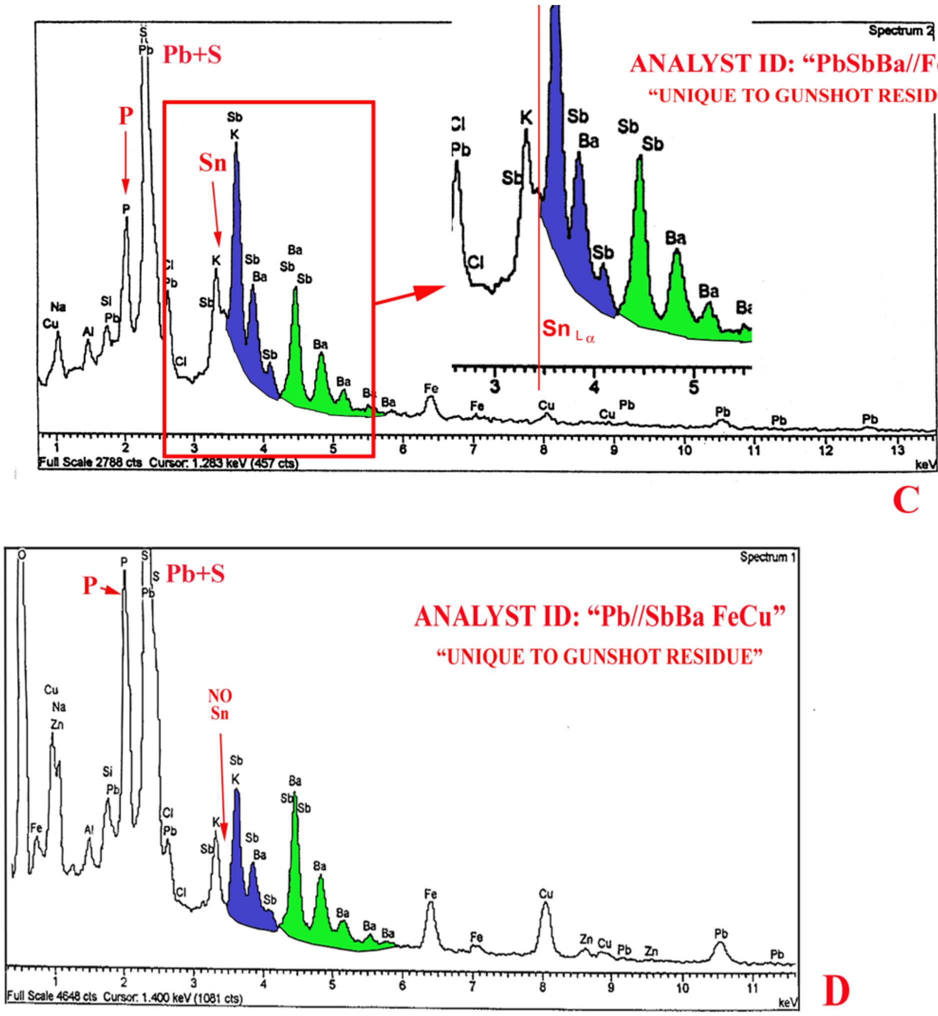

Figure 1 (continued). *C: Spectrum particle #5 (Table 1) that also has tin (Sn) apparently missed by the technician. Insert: portion of the spectrum with the tin L-α line drawn in. D: Spectrum particle #1 (Table 1) identified by the technician as lead-antimony-barium with iron and copper. Tin is not present.*

Phosphorus (P)

Of the nine spectra submitted by the technician in this case, seven particles have phosphorus present (Table 1), six of which have phosphorus in relatively large concentrations (e.g., Figs. 1C & 1D).

The ASTM (3) notes that phosphorus may be found in gunshot residue, but does not provide guidance as to frequency or relative concentration of phosphorus (when present) in gunshot residue particles. Wolten et al. (3) note that "occasional phosphorous [is]… found in residue particles." Romolo (4) says that the presence of phosphorus in gunshot residue is "at trace level only." Giacalone (5) has found occasional lead-phosphorous particles in case work, but does not ascribe a firearms source for these particles..

The criminalist-technician in this case has described most of these phosphorus-bearing particles as "unique" to gunshot residue . Winchester primer material does not contain phosphorus (1) nor are there any reports of American-manufactured small-caliber primers/ammunition containing phosphorus. The origin of these phosphorus-containing particles is not from the discharge of a 9 mm cartridge.

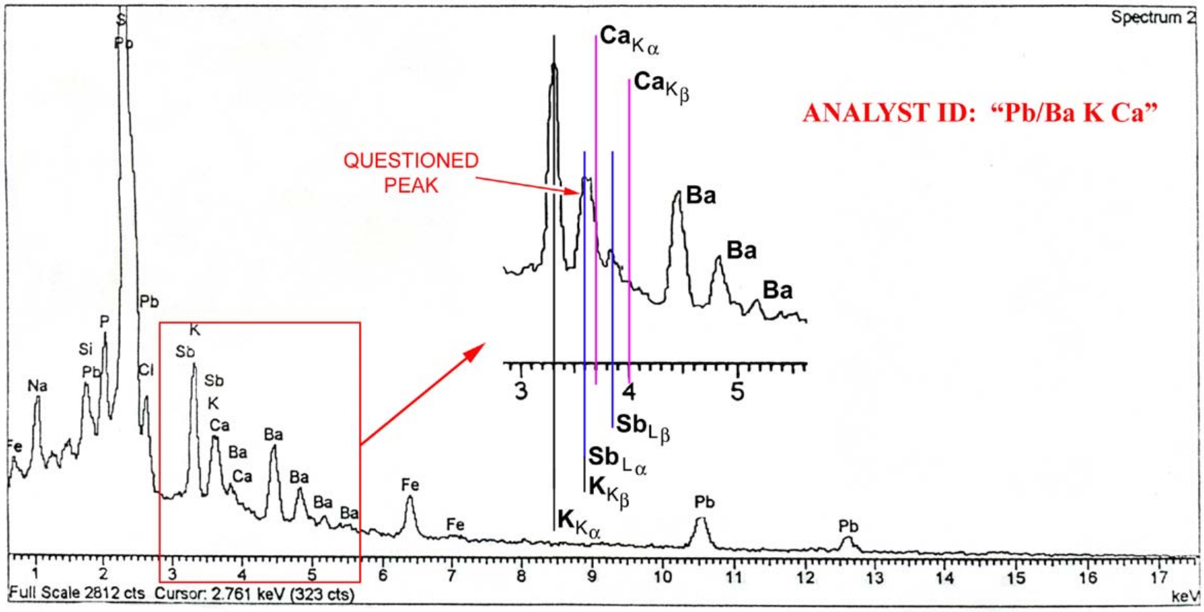

Figure 2. *Spectrum of particle 2 (Table 1) where the technician missed antimony (Sb) The technician has also ignored sodium (Na) as well as phosphorus (P) in this particle. Insert: enlargement of the area of interest with K– and L– lines of potassium (K), calcium (Ca) and antimony (Sb) shown. The* $K_{K\text{-}\beta}$ *and* $Sb_{L\text{-}\alpha}$ *peaks occupy essentially the same position, 3.589 and 3.605, respectively. The peak at 3.6 keV is too large to be from potassium alone.*

Potassium (K)

Six of the nine alleged GSR particles have major potassium (K) content (Table 1) Potassium may be found as a component in varying amount in some GSR samples (3). The technician in this case chose not to report potassium association for almost all of these particles (Table 1).

Conclusions

Torre et al. (6) have documented particles composed of lead, antimony and barium originating from automotive friction-brake products from a number of manufactures. The particles at issue may have been generated from automotive brakes. Indeed, iron (Fe) and copper (Cu) were present in most of these particles which are consistent with a brake origin. However, due mainly to the phosphorus association, these particles differ from those described by Torre et al (6) as originating from brakes. Thus, the source of these particles remains unknown.

The use of the terms "unique" (4) or "highly specific" (7) to describe lead-antimony-barium or any other element combination of gunshot residue-like particles is inappropriate. Torre et al. (6) suggest that it is more appropriate to compare particles sampled from the firearm or casing at issue to the suspected gunshot residue-similar particles from the defendant or crime scene.

Wolten et al. (3, p.58) state, "The presence of substantial numbers of inconsistent particles overrules the evidentiary significance of particles consistent with gunshot residue." Virtually all the particle spectra submitted by the technician in this case are inconsistent with gunshot residue and, thus, all the samplers in this case are negative for gunshot residue.

Part III of this series will examine a case where "highly specific" gunshot residue particles were incorrectly identified. These particles were likely from automotive brakes.

Part IV of this series will examine a case where "consistent" gunshot residue particles were assigned to particles that were likely from a population of partially-geomodified soil lead .

References

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Comments Appreciated!