TECHNICAL NOTE

Carlo Torre,[1] *M.D. and Grazia Mattutino*[1]

Application of True Color X-Ray Vision for Electron Microscopy in Fired Bullets and Gunshot Residue Investigation

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ABSTRACT: An X-ray color imaging system was used to study primer particles and fired bullets from different .22 rimfire ammunition. The technique proved to be very useful, allowing a ready, concomitant analysis of the morphology of primer particles and their elemental composition. The investigation of the bottom of fired bullets showed that antimony present in the bullet alloy is not evenly distributed, but organized in plates made up of almost pure antimony. Moreover, particles and other traces adherent to lead bottomed bullets containing elements different from lead, therefore, useful to the understanding of primer composition are readily and easily detected.

KEYWORDS: forensic science, criminalistics, fired bullets, gunshot residue, SEM, X-ray, color imaging

Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) is a well-established technique for detection and classification of gunshot residue (GSR) particles (1–4). This method is especially valuable for detection of GSR on anatomical surfaces and clothes of suspect shooters. In cases of shooting from short distances, it allows the detection of GSR on the target area. Traces characteristic of ammunition can also be detected around the bullet entrance hole in cases of long range firing distances (5).

In these circumstances, knowledge of the primer composition is useful for the identification of the type of ammunition fired and to verify its compatibility with particles found on the suspect or at the crime scene. This kind of investigation is particularly helpful in cases of unusually primed ammunitions with mercury fulminate or lead free primers, or in .22 rimfire cartridges whose primers usually lack antimony. Obviously, attention must be paid to the possibility of carry-over of primer materials from previous firings (6–10).

Detection of GSR on the bottom of fired bullets (11) can be used for understanding primer composition, especially when cartridge cases are not found. This analysis is easily performed on brass, copper or steel bottomed bullets. In the backscattered image, GSR are readily recognized because of their brightness. On the other hand, detection of GSR and other primer smears in lead bottomed bullets is often challenging, since in backscattered imaging, there is no way to distinguish lead belonging to the bullet from heavy metals contained in the primers only by brightness.

In this work, the Cameo™ program for X-ray color imaging was tested for the study of GSR on fired bullets. Theoretical bases and general applications of color X-ray vision for electron microscopy and microanalysis are described by Statham (12). Briefly, the specific X-ray energy spectrum emitted in the SEM by excited atoms from different elements is detected by EDX and used to reconstruct a true color image which, still retaining the topographical and morphological features of the sample under study, also portrays its underlying elemental composition. This can be achieved by means of an energy dispersive X-ray detector and analysis system. Just as the visual pigments in the cones of the retina, the system assigns color to photons based on their effective wavelengths using a rainbow color scale. The result is similar to offsetting the human visual response to the electromagnetic spectrum into an X-ray wavelength region of choice. Unlike the other SEM color enhancement techniques, the color obtained is "natural"; that is to say, changes in topography and shadowing affect the color in the same way as visible objects.

Methods

Shooting experiments were carried out with several .22LR rimfire ammunitions (Eley, RWS, Federal, Winchester, Fiocchi, Orbea, Remington). Samples were collected from cartridge cases using wooden toothpicks and transferred to double-sided adhesive tape coated stubs. Stubs were then carbon-coated. In order to obtain the removal of any "loose" GSR particles and prevent possible contamination of the SEM sample chamber, bullets were washed in distilled water, then dried and attached on stubs that were not carbon-coated. Each sample was examined and analyzed using a Cambridge 110 scanning electron microscope with a Link ISIS 300 microanalysis system equipped with the Cameo program for X-ray color. After an initial survey conducted at approximately 350X, traces of interest were further examined at a higher magnification.

Results

The analysis of residues found in cartridge cases with an X-ray color imaging system provides the operator with a preliminary visual cue for the definition of the elemental composition of primers (Figs. 1 and 2) to be further investigated by microanalysis.

By examining lead bullets, it was observed that antimony is not evenly distributed, but concentrated in small plates with sharp contours, clearly standing out against the lead background. Figure 3

[1] Associate professor and student respectively, Dipartimento di Anatomia, Farmacologia e Medicina Legale, Lab. di Scienze Criminalistiche, Università degli Studi di Torino, Torino, Italia.

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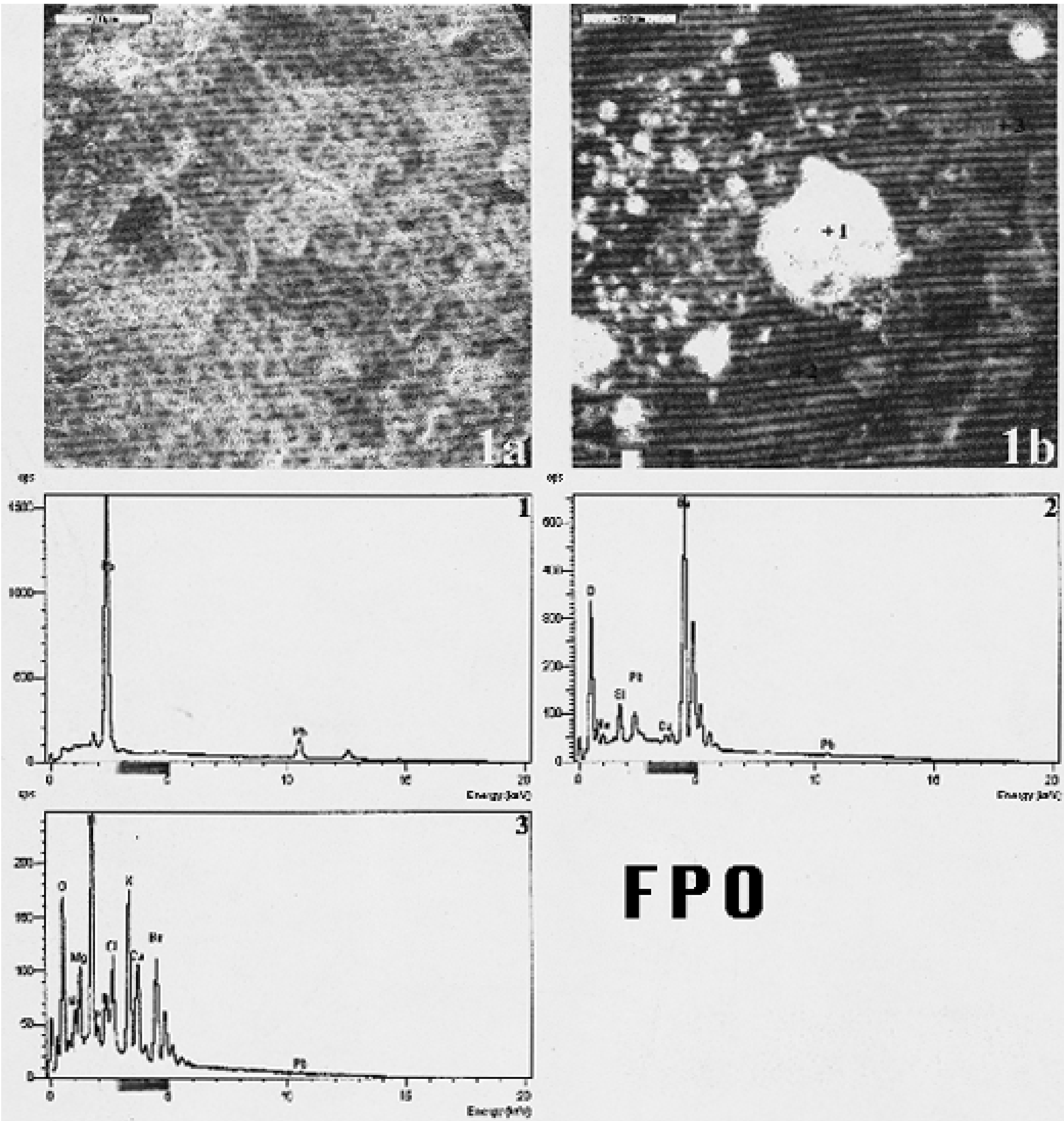


FIG. 1—*Sample taken from a .22LR Eley Standard fired cartridge case. Primer is antimony free. A: secondary electron image, B: color augmented imaging assists spot microanalysis. X-ray spectra relevant to numbered spots of interest in figure B are shown. Using a visual response offset to 2.10 keV 5.00 keV X-ray region, lead (M*α*1 2.3475 keV) is seen as red (spectrum 1), barium (L*α*1 4.4675 keV) as blue (spectrum 2) and intermediate elements (calcium, potassium and chlorine) as green (spectrum 3).*

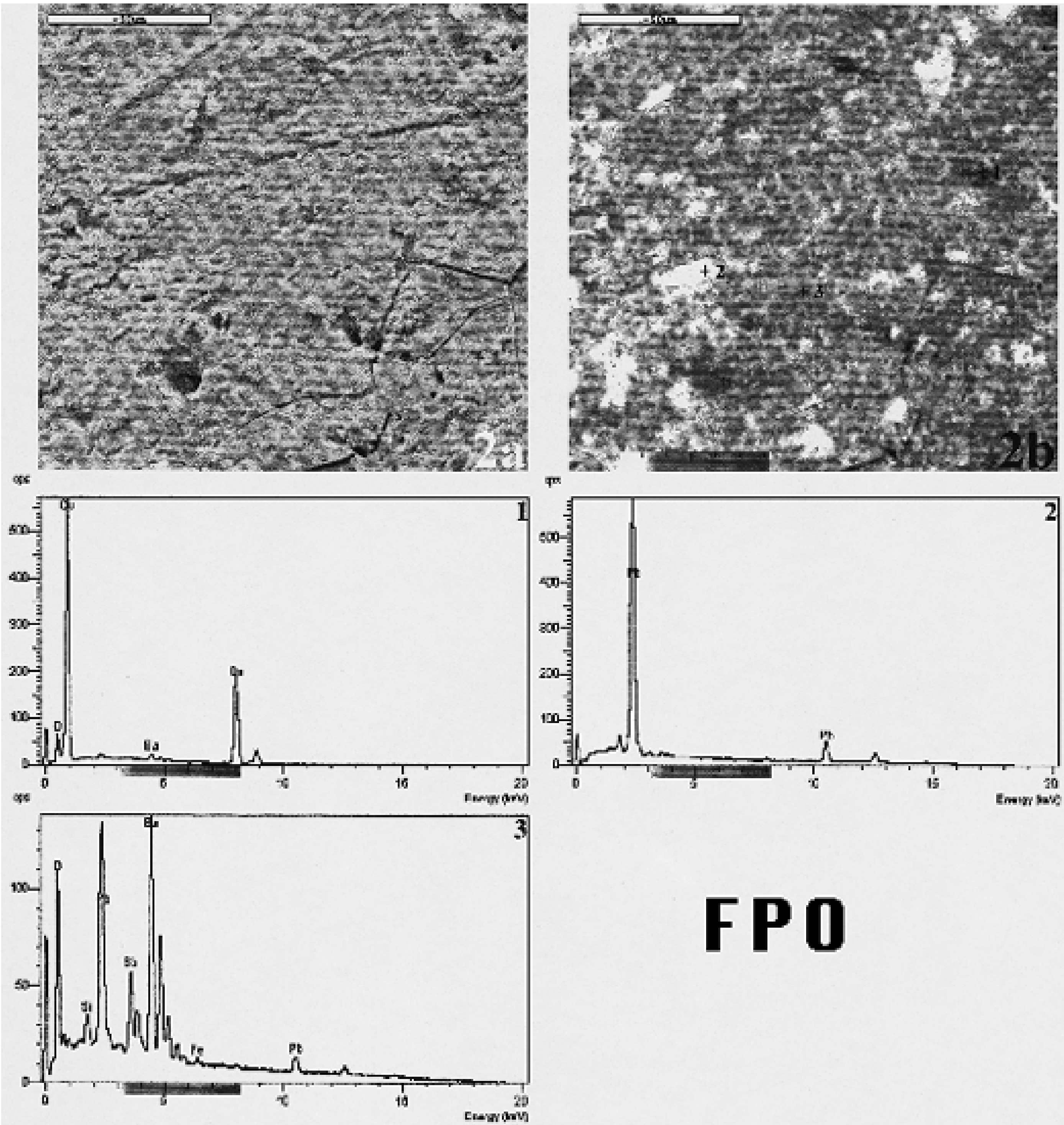


FIG. 2—*Sample taken from an .22LR Federal Expert fired cartridge case. Ammunition is antimony primed. A: secondary electron image, B: color augmented imaging assists spot microanalysis. X-ray spectra relevant to numbered spots of interest in figure B are shown. With a visual response offset to 2.10 keV 8.20 keV X-ray region (copper Kα1 8.0475 keV) traces from the copper coated bullet are seen as blue (spectrum 1); lead is red (spectrum 2), barium and antimony are green (spectrum 3).*

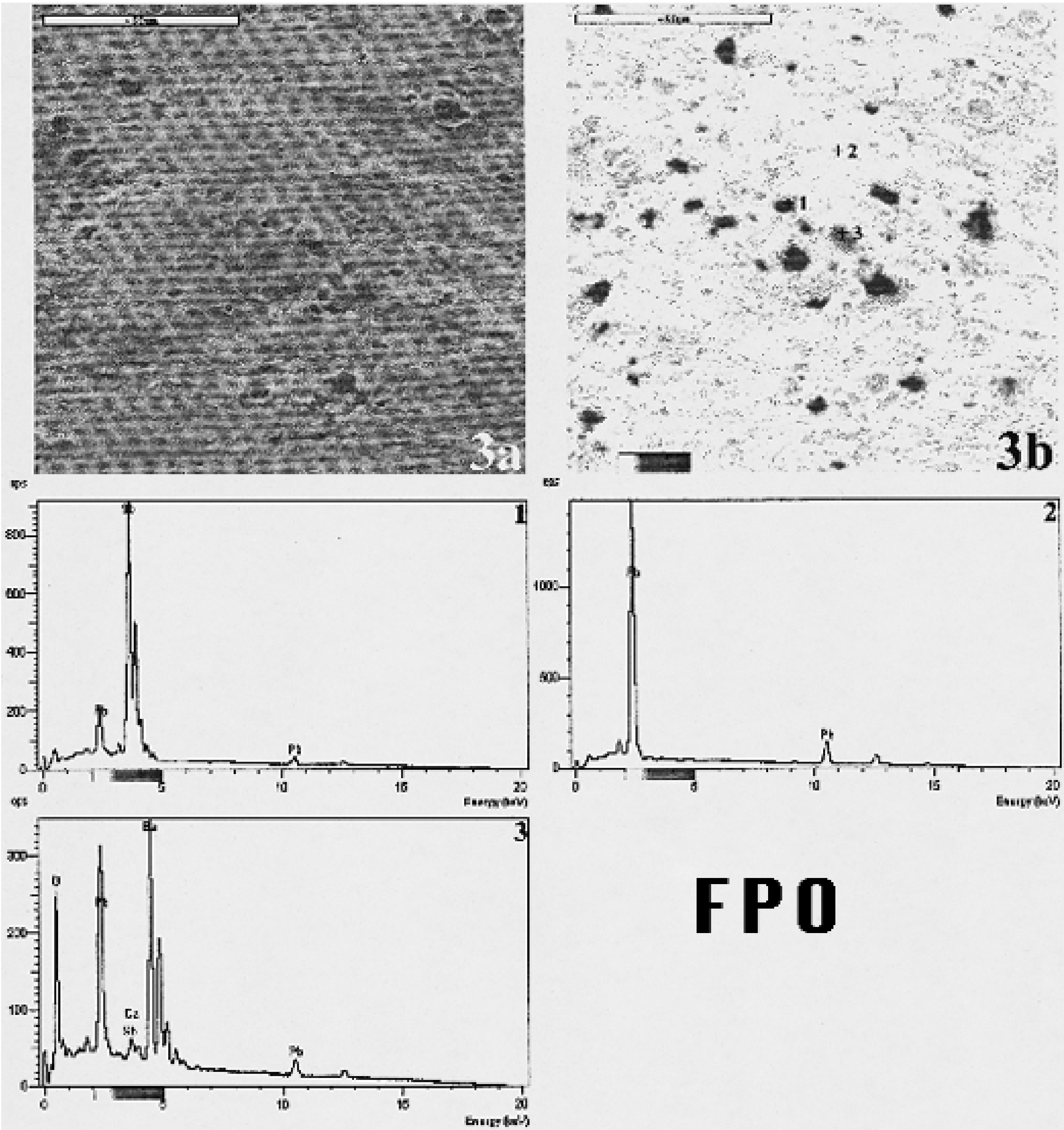


FIG. 3—*Bottom of .22LR Winchester EZXS fired bullet. Primer is antimony free. A: secondary electron image, B: color augmented image using a visual response offset to 2.10 keV 5.00 keV X-ray region shows that antimony of the bullet (green, spectrum 1) is not evenly distributed, but concentrated in small plates with sharp contours, clearly standing out against the lead background (red, spectrum 2). These plates could not be distinguished in conventional SEM imaging. A GSR (blue, spectrum 3), composed by barium and lead, with calcium and antimony in traces, is present.*

shows that antimony plates can not be distinguished by plain morphology (Fig. 3*a*), without the aid of an X-ray color imaging system (Fig. 3*b*).

Bullet bottoms present several craters sometimes filled with GSR. Crater walls are composed of lead that may include an antimony plate (Fig. 4). Even at low magnification traces of interest are easily recognized by their color, so that their morphology and elemental composition can be further investigated at higher magnification. Color assists the spot microanalysis in determining the exact distribution of the different elements (Fig. 5). In addition to typical GSR, smears with an elemental composition characteristic of the primer are seen (Fig. 6).

Discussion

It was possible to demonstrate that antimony contained in the bullet alloy is not evenly distributed, but condensed in plates of almost a pure composition. Such a distribution was never previously described in forensic literature and must be kept in mind whenever passing judgment on the elemental composition of the primer in a fired bullet. If this composition is unknown, there's a risk of mistaking normally present antimony plates for primer traces and misjudging the primer composition.

X-ray color imaging is of great help especially in the detection of GSR on the bottom of fired bullets. The difficulty of GSR detection in lead bottomed bullets has been reported (11) and is well known to those performing this kind of research in forensic laboratories. This color technique allows spotting trace of interest, even at low magnification and before morphological recognition. Furthermore, it enables speeding up procedures, allowing immediate discrimination (i.e., for good imaging, acquisition time is about 5 min) of spherical particles of interest from the many particles of pure lead, which do not provide the operator with any information on the primer composition. Primer traces, which would easily pass unnoticed or require a considerable amount of time for detection with traditional systems, can be readily detected and analyzed.

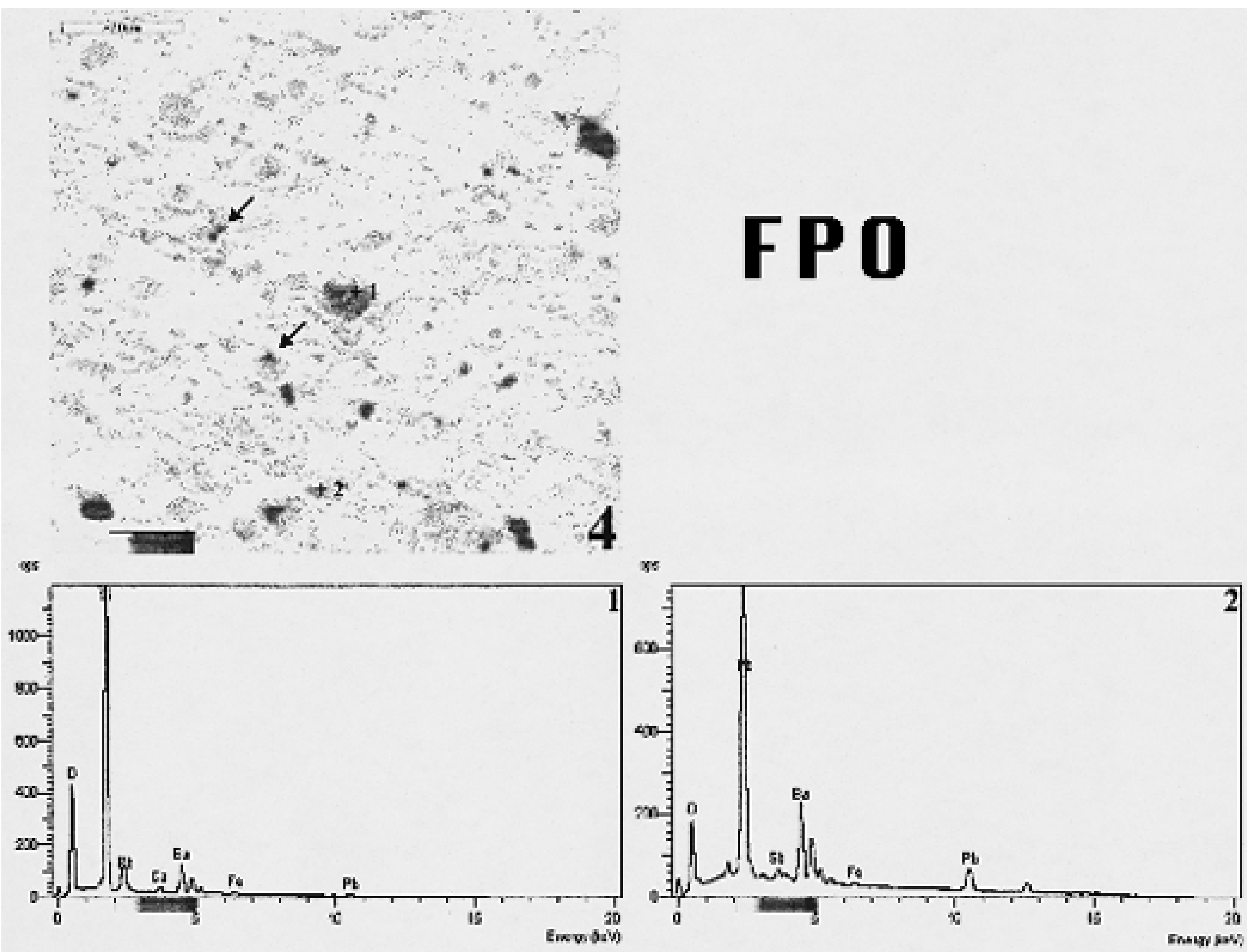


FIG. 4—*Bottom of .22LR Winchester EZXS fired bullet. Color augmented image using a visual response offset to 2.10 keV 5.00 keV X-ray region shows craters containing an antimony plate in their wall (arrows). In one of the craters is enclosed a GSR composed by silicon, barium and lead, with calcium and iron in traces (spectrum 1). A smear characterized by lead and barium, with antimony and iron in traces, is also seen (spectrum 2).*

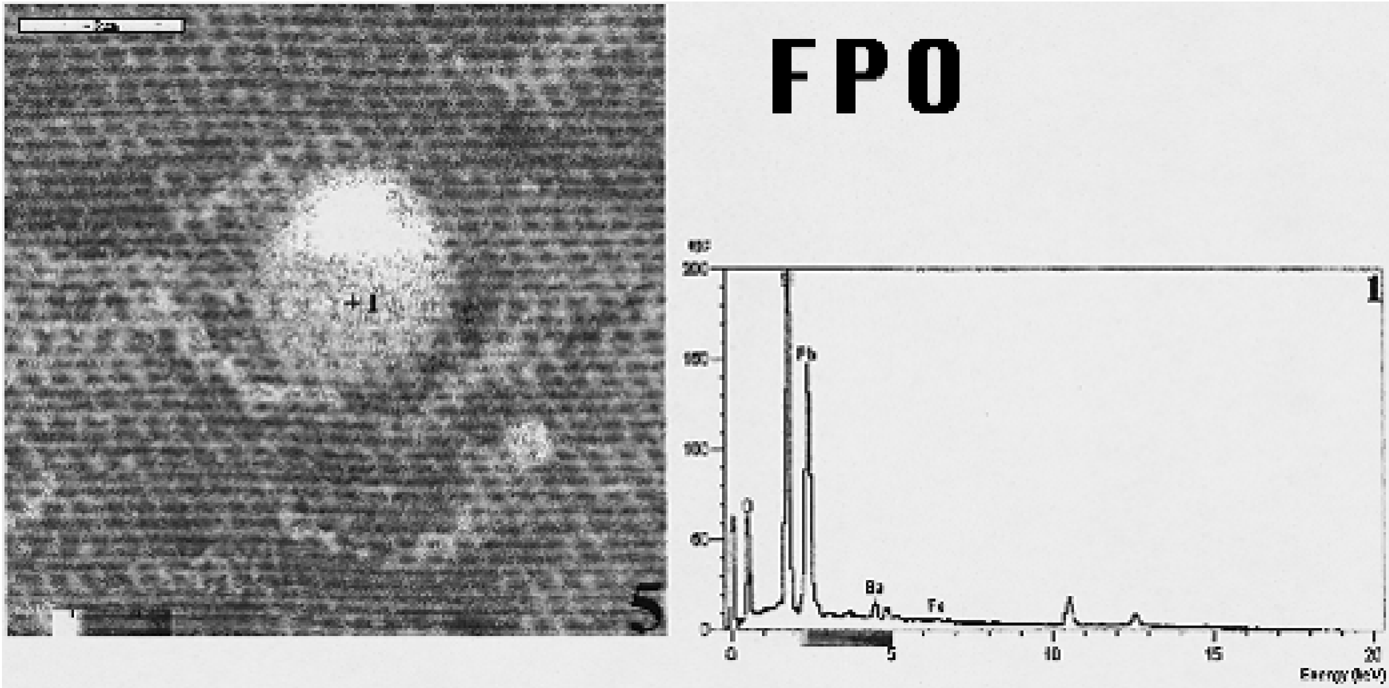
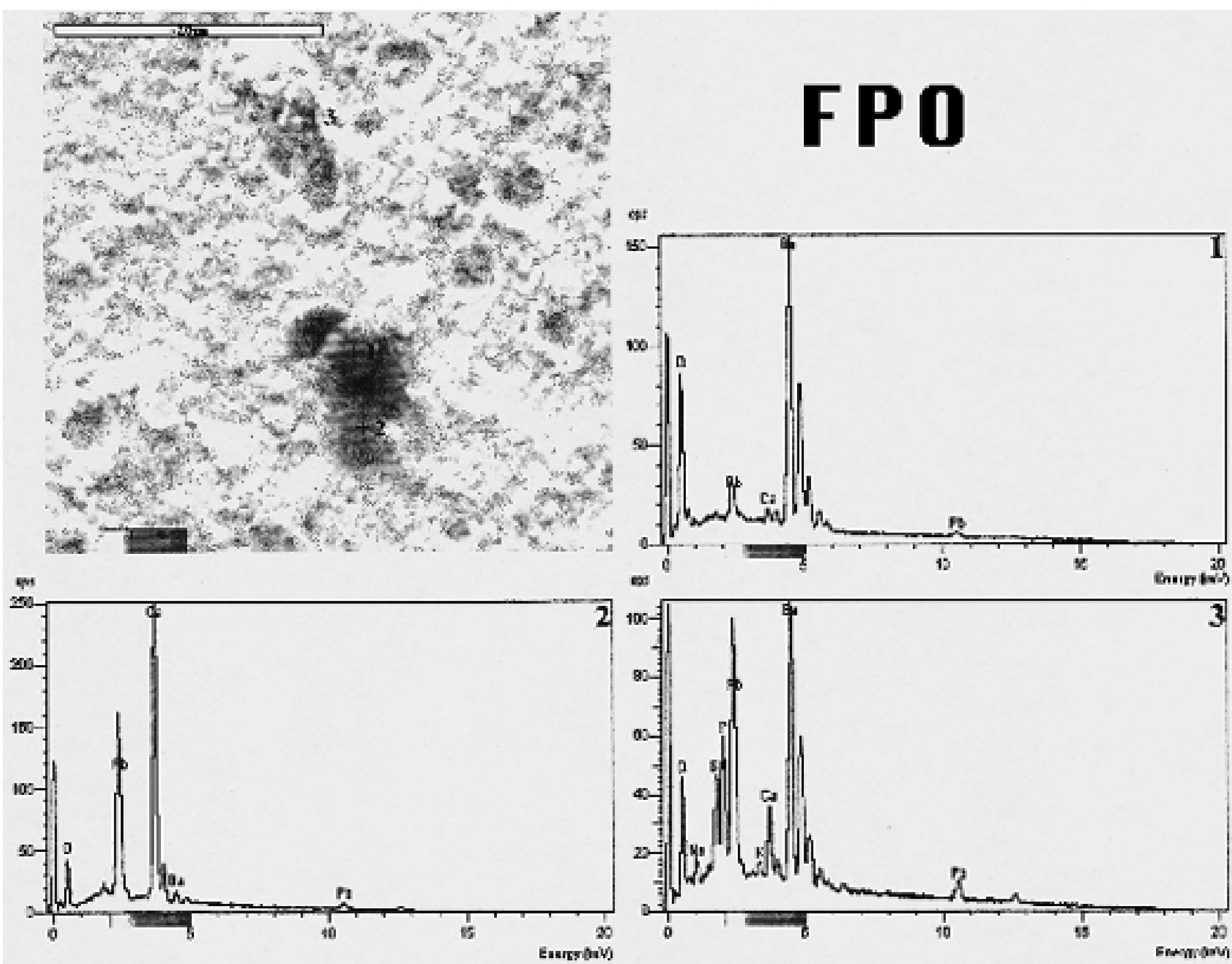


FIG. 5—*Bottom of .22LR Winchester EZXS fired bullet. Color augmented image shows the same GSR as depicted in Fig. 4, at higher magnification. Since visual response is now offset to 1.40 keV 5.00 keV X-ray region (silicon Kα1 1.7475 keV), the GSR is seen as red, while lead is green. Lead nodules (spectrum 1) on the GSR surface are easily detected.*



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References

1. Wolten GM, Nesbitt RS, Calloway AR, Loper GL, Jones PF. Final report on particle analysis for gunshot residue detection. Report ATR-77:7915-3; The Aerospace Corporation, Washington, DC, 1977.
2. Wolten GM, Nesbitt RS. On the mechanism of gunshot residue particle formation. J Forensic Sci 1980;25(3):533–45.
3. Basu S. Formation of gunshot residue. J Forensic Sci 1982;27(1):72–91.
4. Wallace JS, McQuillan J. Discharge residues from cartridge-operated industrial tools. J Forensic Sci Soc 1984;24(5):495–508.
5. Ravreby M. Analysis of long-range bullet entrance holes by atomic absorption spectrophotometry and scanning electron microscopy. J Forensic Sci 1982;27(1):92–112.
6. Zeichner A, Levin N, Springer E. Gunshot residue particles formed by using different types of ammunition in the same firearm. J Forensic Sci 1991;36(4):1020–6.
7. Zeichner A, Levin N, Dvorachek M. Gunshot residue particles formed by using ammunitions that have mercury fulminate based primers. J Forensic Sci 1992;37(6):1567–73.
8. Gunaratnam L, Himberg K. The identification of gunshot residue particles from lead-free Sintox ammunition. J Forensic Sci 1994;39(2):532–6.
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. Wrobel HA, Millar JJ, Kijek M. Identification of ammunition from gunshot residues and other cartridge related materials—a preliminary model using .22 caliber rimfire ammunition. J Forensic Sci 1998;43(2):324–8.
11. Bergman P, Enzel P, Springer E. The detection of gunshot residue (GSR) particles on the bottom of discharged bullets. J Forensic Sci 1988;33(4):960–8.
12. Statham PJ. True color X-ray vision for electron microscopy and microanalysis. Mikrochim Acta [Suppl.] 1996;13:573–9.

Additional information and reprint requests:
Carlo Torre, M.D.
Dipartimento di Anatomia, Farmacologia
Medicina Legale
Lab. di Scienze Criminalistiche
Università degli Studi di Torino
C.so M. D'Azeglio 52, 10126
Torino, Italy

FIG. 6—*Bottom of .22LR Eley Tenex fired bullet. Primer is antimony free. Color augmented image using a visual response offset to 2.10 keV 5.00 keV X-ray region shows a GSR mostly composed by barium (blue, spectrum 1), laying on a calcium smear (green, spectrum 2). A trace typical of Eley cartridges, characterized by barium, lead, phosphorus, calcium, and silicon (spectrum 3) is seen.*