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Use of Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis (SEM-EDXA) in Identification of Foreign Material on Bullets

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ABSTRACT: The authors report two cases in which examination of foreign material embedded in or adherent to bullets provided critical information in the reconstruction of a crime scene. Analysis of small particles by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDXA) can be accomplished without destruction or injury of the particles. In one case, the detection and identification of mineral fragments embedded near the nose of a bullet provided conclusive evidence that the bullet had ricocheted from a fireplace before striking the victim. In the second case, analysis of particles from two bullets identified them as bone fragments, thus proving which shots fired from a police officer's gun had killed a suspected burglar. SEM-EDXA has not been widely used to identify such material on bullets, but should be considered a potentially powerful tool in forensic science.

KEYWORDS: criminalistics, ballistics, chemical analysis, microscopy, X-ray analysis

Scanning electron microscopy (SEM) has been used in a variety of forensic science applications, including examination of bite and tooth marks, hair analysis, determination of direction of travel of bullets in bone, and examination of seminal stains [*1–6*]. Other real and potential uses of SEM have been discussed in a number of publications [*7–17*], but one of the most important contributions to forensic science investigation comes from the combined use of SEM and energy dispersive X-ray analysis (EDXA) to study gunshot residues, usually from the primer or bullet, on clothing, hands, or in the area of a wound [*18–22*]. In only one of these studies was SEM used to detect material on a bullet and to determine its chemical identity by EDXA. Ravreby mentions that particles were detected by SEM-EDXA on the rear of bullets fired into wooden blocks [*18*]. These particles presumably represented powder and primer residues. We wish to report what may be a new and important use for SEM-EDXA, the examination and identification of material embedded within or adherent to a bullet. In some cases this material may be the pivotal factor in reconstructing a crime scene and determining whether or not an individual will be prosecuted for homicide.

After a bullet is recovered from a body or crime scene, most pathologists wash the blood,

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tissue, and foreign material from it, make a cursory examination to determine its construction and approximate caliber, engrave a number or initials on it, and seal it in a labeled container to be subsequently handed over to a firearms examiner. This process may destroy valuable evidence. If a lead or lead-tipped bullet perforates an intermediary target, fragments of this material may be found embedded in or adherent to the bullet. Foreign material, such as powder, cloth, glass, wood, or paint, is often readily visible if the bullet is examined with a dissecting microscope before cleaning. Once recognized, this material can be subjected to nondestructive tests, including SEM-EDXA. We describe two homicides in which the final determination as to how the deaths occurred was made based on identification of material embedded in the tip of the bullet.

Materials and Methods

Tissue processing for histologic examination was performed by routine methods except that small and delicate specimens were processed by hand to minimize damage. Polarized light microscopy was performed on a Zeiss photomicroscope. SEM was performed on a JEOL Model 35 scanning electron microscope. When necessary, specimens were carbon-coated to reduce charging in the electron beam and to increase resolution, but in other cases the specimens could be viewed directly without coating. EDXA was performed using a Tracor-Northern X-ray detector with an accelerating voltage of 30 kV. EDXA data was analyzed using a Tracor-Northern 2000/4000 computer.

Case 1

The deceased, a 19-year-old white male, was visiting at a friend's apartment when the friend decided to show the deceased his new weapon. The friend left the room and returned twirling a cocked, single-action revolver on his right index finger. As he entered the room, the gun allegedly slipped from his hand and discharged. The friend turned to the victim, who was seated on a couch to the friend's left, and asked if he was all right. The victim appeared dazed, and began to vomit. The emergency medical squad (EMS) was summoned and the victim was transported to the hospital, but he died approximately 1/2 h later.

At autopsy, the deceased had a single ovoid, 10- by 5-mm, gunshot wound of the left forehead, 19 mm (3/4 in.) to the left of the midline (Fig. 1), with no blackening or powder tattooing of the surrounding skin. Just lateral to the entry wound there was a smaller, crescent-shaped wound which was produced by a fragment of lead stripped from the bullet as it entered the skull. This fragment traveled subcutaneously and exited at the crescent-shaped wound. The main part of the bullet perforated the left half of the frontal bone, producing an oval defect with beveling of the inner table of the skull and multiple linear fractures radiating from the entry site. The bullet then passed through the left frontal and temporal lobes and struck the left temporal bone where it produced a small, depressed fracture. The flattened and deformed copper coated .22 caliber bullet was recovered from the occipital lobe. White granular material was adherent to the tip of the bullet. Examination of the weapon revealed a single-action .22 caliber revolver loaded with five live and one spent .22 caliber Long Rifle cartridges.

At autopsy, the prosector was struck by the odd shape of the entrance wound. That, in conjunction with the flattening of the tip of the bullet and the white granular material adherent to the tip in the area of flattening, made her suspect that she was dealing with a ricochet. It was decided that a more intensive investigation was justified; additional statements were obtained from the friend and the scene was revisited and photographed.

The wall opposite both the couch on which the deceased was sitting and the doorway through which the friend entered contained a fireplace inclined inward at an approximate 45° angle. The fireplace consisted of typical native limestone embedded in mortar. Both the

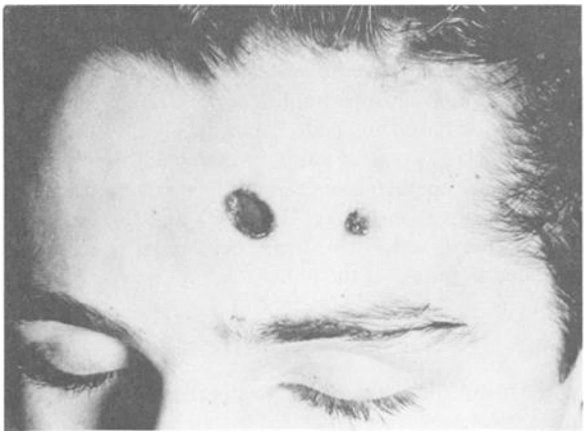

FIG. 1—*Ovoid entrance wound of forehead with small exit wound lateral to entrance.*

stones and mortar were inspected for defects which could have been made by a bullet, but unfortunately, there were multiple defects and irregularities in the stone and the mortar, and no definite defect attributable to a bullet could be found. Random samples of the mortar and stone were taken and submitted along with the bullet for analysis by SEM-EDXA.

Examination of the bullet under SEM revealed two different kinds of materials along the nose and side of the bullet at the point where it was flattened (Figs. 2 and 3). The first type was amorphous and gave EDXA spectra consistent with organic material, probably representing remnants of soft tissue from the victim. The second type of deposit consisted of two particles embedded in the lead nose of the bullet on the flared part of the flattened surface. These particles contained major concentrations of calcium with smaller amounts of silicon and aluminum (Figs. 4 and 5). One of the particles contained trace amounts of titanium and iron (Fig. 5). The spectra of these two particles were not typical of substances normally occurring in the human body or in bone. They were consistent with spectra that might be obtained from minerals (such as limestone) with trace impurities (aluminum silicates) or from conglomerate material (that is, aggregates such as mortar, which consists of sand, limestone, and various impurities). The titanium might represent either an impurity in the mineral itself or a pigment from paint or colored mortar.

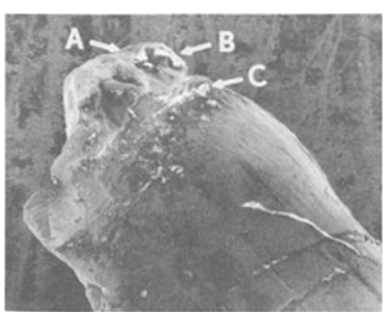

FIG. 2—*This relatively low magnification photograph of the bullet demonstrates disfiguration and flattening of the nose. Embedded within the flared portion of lead on one side are small particles of mineral (*a *and* b*). Material labeled* c *represents amorphous deposits most consistent with organic residue. (Scanning electron photomicrograph; original magnification* ×*100).*

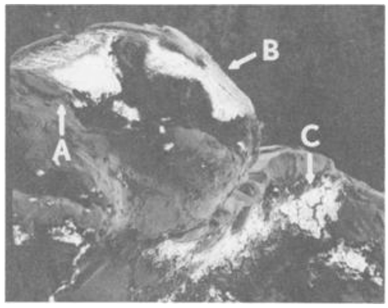

FIG. 3—*This higher magnification of the mineral deposits demonstrated in Fig. 2 shows clearly that particles* a *and* b *are embedded within the soft lead of the bullet. Organic material* c *is still seen. (Scanning electron photomicrograph; original magnification ×390).*

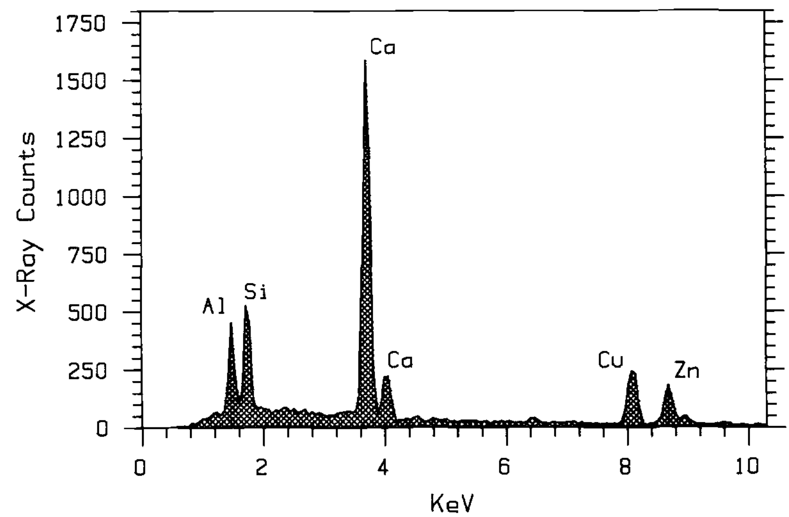

FIG. 4—*This EDXA spectrum of particle* A *(see Figs. 2 and 3) demonstrates major peaks at 3.76 and 4.02 keV typical of the* K*-alpha and* K*-beta transitions of calcium. Lesser peaks correspond to the elements aluminum (*K*-alpha 1.49 keV) and silicon (*K*-alpha 1.74 keV). Smaller peaks of copper and zinc may have been derived from either the bullet coating or from extraneous fluorescence of the brass components of the SEM column, a common artifact. Elements below atomic number 10 are not detected with this particular instrument, thus the oxygen and carbon that are integral components of limestone (calcium carbonate) are not seen in this spectrum.*

Examination of the mortar and multiple samples from the fireplace stones demonstrated considerable variation in the composition and relative peak heights of the component elements from one sample to another and even from one area to another within a single sample (Figs. 6 and 7). This variation is best explained by the heterogeneity of the minerals themselves. All of the elements identified in the mineral fragments from the bullet were identified in the mineral fragments from the fireplace, and although a perfect match was not obtained, there were several very near matches.

It was clear that the nose of the bullet had impacted a mineral material with force sufficient to embed small fragments within the superficial substance of the bullet. Since these

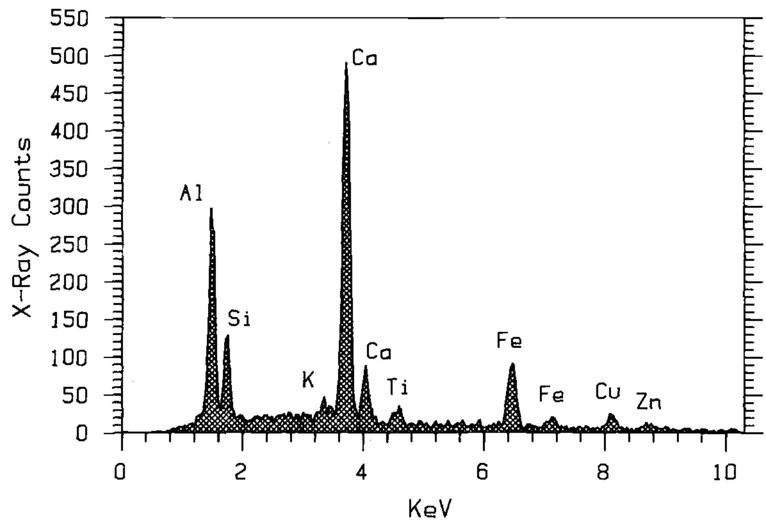

FIG. 5—*This EDXA spectrum of particle* B *again demonstrates major peaks at 3.76 and 4.02 keV typical of calcium, as well as the smaller peaks of aluminum and silicon, and the copper and zinc peaks. There are also trace quantities of iron (6.40 keV) and titanium (4.51 keV) within this fragment of mineral. These last elements not only occur as "impurities" in a variety of minerals, but they are common elements in pigments used by masons or painters.*

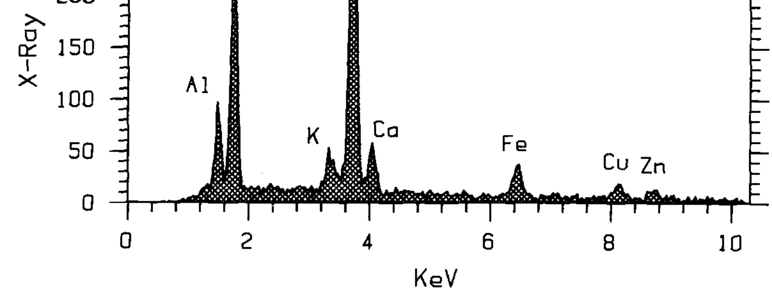

FIG. 6—*This spectrum was obtained from one of the many samples of limestone rock. Note the major peaks corresponding to calcium as well as the lesser peaks of aluminum and silicon. Trace quantities of other elements are present.*

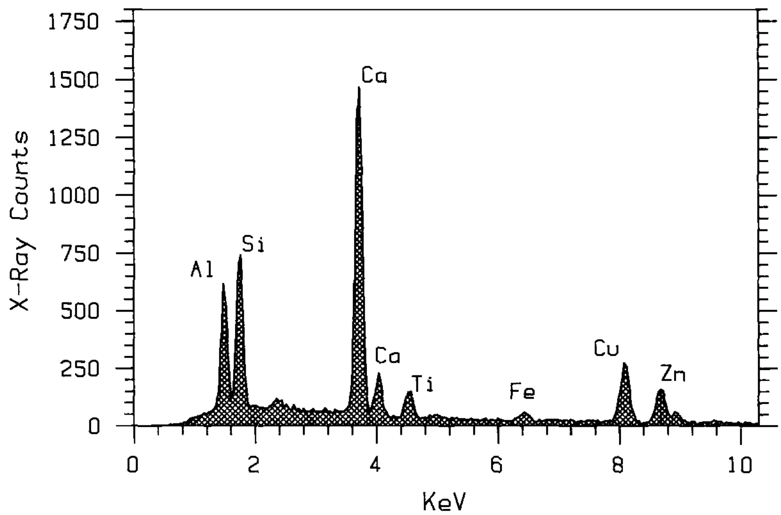

FIG. 7—*This spectrum was obtained from a sample of firebrick. Note the major peak corresponding to calcium, and lesser peaks of aluminum, silicon, and iron as well as trace quantities of other elements. Although the same elements that are seen in other spectra are also demonstrated in this spectrum, note that the relative peak heights, which correspond to concentration, vary considerably. This variation is best explained by heterogeneity in composition of these particular materials.*

mineral materials are not normally found in human tissues, including bone, they must have originated outside the body of the deceased. The elemental composition of the mineral particles was consistent with the kinds of minerals and material found in the fireplace. This information confirmed the medical examiner's opinion that the entry wound was consistent with that produced by a deformed bullet, and we concluded that the deceased was struck and killed by a ricochet. Because of this opinion, all charges were dismissed against the alleged perpetrator.

Case 2

A police car on routine patrol received a call concerning a possible burglary in progress at a row of nearby townhouses. While inspecting one of the townhouses, an officer noticed a broken window next to a sliding glass door and saw a suspect inside with a flashlight. The officer did not attempt to enter but called for assistance and waited behind a fence. Four to five minutes later, the glass door opened and the suspect walked out onto the patio. The officer stepped from behind the fence and shouted "Freeze! Police!" The suspect turned in the direction of the officer, raised his left hand in which he clenched a long slender instrument which resembled a large knife [later discovered to be a 40-cm (16-in.) snowmobile cleat], and charged towards the officer yelling loudly. The officer fired his revolver four times, and the suspect collapsed on his left side, landing approximately a foot from the officer. The officers attempted first aid, but the suspect was dead.

At autopsy, 3 through-and-through gunshot wounds were present in the body of the 17-year-old white male. Gunshot Wound A, the fatal wound, was present in the right chest. The bullet perforated the right third rib, the right lung, the heart, and the left fifth rib, and exited the left side of the chest. Gunshot Wound B was in the right inguinal region. This bullet traveled posteriorly through the soft tissue of the pelvis, striking and fracturing the right superior ramus, and exited the right buttock. Gunshot Wound C was present in the right flank, in the midaxillary line. The bullet travelled posteriorly through the soft tissue and also exited the right buttock, but at a separate site. There was no evidence of close range firing around the entrance wounds in the skin, nor evidence of either soot or powder on the overlying clothing of any of the 3 wounds.

The officer's weapon was a Smith & Wesson .357 Magnum loaded with Remington 158 gr .357 Magnum semijacketed soft-point ammunition. Four rounds had been fired.

A search of the scene revealed two bullets lying on the surface of the ground in close proximity to the body. They were labeled Bullets 1 and 2. Bullet 1 consisted of only the copper jacket of a semi-jacketed bullet; Bullet 2 was complete with both jacket and core. Additionally, a bullet hole was found in the wood siding of the house, adjacent to the sliding glass patio door, close to the ground. The siding was cut away and Bullet 3 was recovered within the insulating material in the wall between the inner and outer layers.

Although the grand jury ruled that the shooting was justified and no criminal charges were pressed against the officer, the family of the deceased hired a forensic pathologist and planned civil litigation against the officer. During an investigation of the scene, the pathologist recovered what appeared to be a fourth bullet from the ground, in the area where the deceased had collapsed. This projectile was buried a few inches into the dirt, and was subsequently identified as the lead core from Bullet 1.

It was the contention of the family that the deceased was shot in the chest with Bullet 1 while lying on the ground helpless after receiving two nonfatal gunshot wounds. Presumably this bullet then struck the ground, depositing its jacket on the surface and its core several inches beneath.

One of the authors (VJMD) was consulted by the defendant's attorneys to see if he could either deny or confirm the plaintiff's contentions. The autopsy report, police reports of the incident, police laboratory reports, diagrams of the scene, photographs of the body and scene, the three bullets (in the case of Bullet 1, both the jacket and core), and the piece of wood siding perforated by Bullet 3 were submitted for review.

When placed on its side, Bullet 3 fit snugly in the rectangular hole in the wood siding, suggesting that the bullet had been tumbling as it struck the wood. The failure of this high velocity round to penetrate the entire wall also suggested that the bullet had lost considerable energy before striking the wall. Both findings could be explained if Bullet 3 had passed through the deceased's body before impacting the wall. Under a dissecting microscope, shiny, white, apparently crystalline material was found adherent to the lead core of this bullet at its junction with the jacket. Similar material was also found on Bullet 2. Although the particles could not be identified with certainty, they resembled small fragments of bone. Decalcification and histological study of the particles might determine if they were bone, but it was feared that such handling could also cause the loss or disintegration of the extremely small particles. For this reason, nondestructive examination by SEM-EDXA seemed to be a preferable procedure.

A total of five particles were submitted for examination by SEM-EDXA. Two particles removed from Bullet 2 were essentially identical, and consisted of calcium and phosphorus in quantities proportionate to that seen in bone (Fig. 8). One of these particles was then fixed, rehydrated, and decalcified for routine histologic examination. The small fragment demonstrated the typical lamellar pattern of bone, complete with lacunae, in both ordinary light microscopy and by polarized light microscopy (Figs. 9 and 10). Three particles recovered from Bullet 3 also consisted of calcium and phosphorus in quantities proportionate to that seen in bone. Two of these particles were then rehydrated and decalcified for histological examination. One particle fragmented during processing and was lost. The other particle was successfully prepared, and upon examination revealed the typical lamellar pattern of bone, complete with lacunae.

Thus, based on SEM-EDXA, the elemental content of all five particles removed from Bullets 2 and 3 were typical of bone. Histologic examination confirmed that at least one

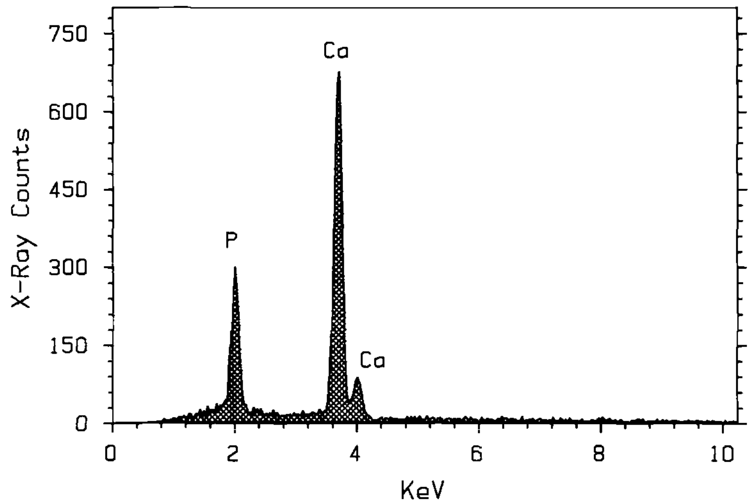

FIG. 8—*EDXA spectrum typical of all five of the particles removed from Bullets 2 and 3. Note the peaks at 3.76 and 4.02 keV corresponding to the K-alpha and K-beta transitions of calcium, and the peak at 2.02 keV which represents the K-alpha transition of phosphorus. Spectra obtained from known specimens of bone demonstrated the same elements in essentially identical proportions.*

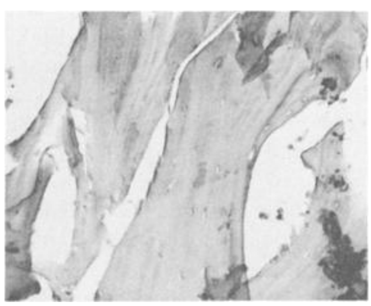

FIG. 9—*This photomicrograph of one of the particles that was fixed, rehydrated, and processed for routine light microscopy demonstrates the typical lamellar pattern of bone. Lacunae are easily seen, although the cells are not apparent.*

particle recovered from each bullet was unequivocally bone. One would have to conclude that both Bullets 2 and 3 passed through bone at some time during their flight. Since the autopsy revealed that only two bullets went through bone, Gunshot Wound A through the deceased's chest, and Gunshot Wound B through the right groin, these wounds must have been made by Bullets 2 and 3. By exclusion, Bullet 1, whose lead core was found embedded in the ground near where the suspect fell, must have either produced Gunshot Wound C (the nonfatal soft tissue wound of the right flank) or missed the deceased entirely.

Thus, the hypothesis that the deceased was fatally shot (Gunshot Wound A) with Bullet 1 while lying on the ground was clearly not tenable. No further action was considered against the officer.

FIG. 10—*This is the same particle of bone demonstrated in Fig. 9 now viewed in polarized light to confirm the typical birefringent nature of lamellar bone.*

Summary

Both cases illustrate the necessity of meticulous examination of bullets removed from bodies or crime scenes before they are cleaned or otherwise altered. In addition to visual examination aided by use of a dissecting microscope, it may be appropriate to subject the bullet or material removed from it to nondestructive examination by scanning electron microscopy and energy dispersive X-ray analysis before destructive or potentially destructive tests are used. In the two cases cited above, important evidence obtained by SEM-EDXA resulted in major legal decisions, one eliminating the possibility of murder, the other confirming a police officer's account of a shooting death. We are aware of one other case in which examination of a bullet for foreign material by SEM-EDXA provided critical information: The presence of aluminum embedded within a bullet proved that it had passed through an aluminum wire screen before striking the occupant of a house.[3]

SEM-EDXA should be considered a valuable tool in the medical examiner's armamentarium. It is possible that much valuable information can be lost by "routine" cleaning of bullets, or other objects, if the examiner is not aware that in many cases small fragments of material can be analyzed by nondestructive methods with rather dramatic results. This technique is most suitable for demonstration of non-organic materials (that is, metal, minerals, various pigments, etc.), but in some cases can provide presumptive identification of organic materials, especially if they are mineralized tissues, such as bone. Both morphologic information (SEM) and elemental analysis (EDXA) can be obtained simultaneously, and once these findings are documented, the specimens are available for other forms of examination.

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