

Appendix I Lithics and Minerals Zachary X. Hruby

Jades

A substantial number of worked jade plaques, beads, celts, earspools, and mosaic tesserae (1.07 kg in total) were found in the El Diablo tomb, underscoring the royal status of its occupant. In the Maya lowlands, this quantity of worked jade is not found in commoner contexts outside of certain workshops. The quality of workmanship indicates a lavish investment, involving hundreds of hours of production and finishing. The features of the jades are consistent with the Early Classic date of the tomb. During the Late Classic period, jades came from varying sources, to be cut from many disparate cores or reworked from older beads, plaques, and celts (Filloy Nadal and Martínez del Campo Lanz 2010:114-117). In contrast, the El Diablo jades appear to have been systematically produced from a few cores or nodules (see below). In addition, Late Classic mosaic masks tend to exhibit tight fits between mosaic tesserae. Early Classic tesserae, including those from Burial 9, offer a contrast, having more rounded and loose fittings. There is also a greater reliance on organic compounds, such as copal, as an adhesive or holding medium (Maynard and Berdan 2010:160).

Most of the jades from Burial 9 were elements of royal regalia, including a belt assemblage with celt tinklers, ear ornaments, and ancestor or deity masks constructed of mosaic tesserae. All of these items were distributed in a fairly small area of the tomb chamber and likely adorned the deceased at the time of interment (see Chapter 3). Two groups of jade artifacts, the mosaic masks and the belt celt assemblages, appear to have been articulated and bound with organic materials. Conservators used a temporary binder, cyclododecane, to lift the mosaic fragments out as a block, which also had the effect of preserving the spatial relationship between tesserae (see Appendix III).

Judging from color, translucency, and crystalline structure, the raw materials for the jade artifacts came from a number of different sources in the Upper and Middle Motagua regions of Guatemala (Rochette 2009; Taube et al. 2011:143-150; Taube and Ishihara-Brito 2012:138-140). The earspools, which are of a light green-blue jade, probably came from the middle Motagua region, perhaps from the modern departments of Jalapa or Zacapa of Guatemala. The jade plaques, of an opaque green to light green color, probably originated in the Upper Motagua region (observations by author, 2008, 2009, 2010). The belt celts are of a dark, forest-green jade, which is common throughout most of the jade-bearing regions of Guatemala. To be sure, all of these general source designations derive from personal impressions of various jade types in jade-bearing regions of Guatemala. They must remain conjectural until chemical and mineralogical sourcing of the jades can be carried out.

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Figure I.1. Plan and side views of each of two large earspools from Burial 9. Drawings: Sarah Newman.

The colors are similar in each class of jade artifact. In large part, this is because the earspools, plaques, and celts were clearly cut from a separate "mother stone" or core. The earspools in particular (Figure I.1) were carved from a single sub-spherical cobble, as shown by their identical color, residual cortex, and form. Each piece had matching fractures, indicating that the jades were cut "facing" one another in the core. The center of each earspool must have been drilled out using a hollow, tubular cane drill. This resulted in the earspool blank and a central piece that might have been transformed into beads not evidenced in the tomb. The surfaces of the earspools were polished with a jade or limestone polishing stone, an artifact common in the Maya area (Kovacevich 2011; Kovacevich and Hruby 2005; Taube and Ishihara-Brito 2012). The jade was affixed to a stick and then spun in the hand or with a bow atop the surface of a polishing stone. Finally, small holes were drilled into the sides of the earspools to stabilize them within a larger assemblage or to affix them to a headdress. These holes could have been

made with a quartz crystal or chert bit: indeed, Kovacevich (2006) has documented large quantities of quartz grain and debitage associated with jade. There may also have been drills that combined mineral and organic materials. No grinding implements, drills, or quartz or jade debitage were found at El Zotz, however, making the location of their production a mystery.

The plaques (Figure I.2), part of a belt assemblage, were string-sawn from a cobble-sized block. All of the plaques are the same or similar color, showing the matching fault lines that occur naturally in Guatemala jadeite; two plaques display corresponding string-saw scars. The plaques were crafted from one "mother" core. The original cobble may have been collected from a river, since one plaque retained a small portion of river-rolled cortex. The pieces themselves fall into two nearly identical groups of two, a shorter group of roughly 10.4 centimeters, and a longer pair of 12.4 centimeters. The width and thickness of all the pieces are strikingly similar, suggesting a similar origin of production and joint

use in a single assemblage of jewelry. Moreover, the plaques all appear to have been made or planned by the same individual. The strategy and technique of manufacture is heretofore unknown to the author. The plaques feature a centrally carved trench, probably formed by a wedge saw to judge from its v-shaped sidewalls. Although the wedge was likely sawn back and forth, it resulted in a roughly circular incision, opening the possibility that a circular saw was employed. No such saw is attested anciently, and, in fact, this type of incision has been used by some to identify modern forgeries (Walsh 2009; see also Sax et al. 2000). Future microscopic analyses are necessary to understand such scarring.

Each plaque was conically drilled four times: once centrally at each end of the plaque, and once at each end of the trench. In each case the holes were drilled to connect at a roughly perpendicular angle and then smoothed out, probably with a grit-covered cord or string. The stone worker probably devised this process because the plaques are quite long and thin. Normal biconical drilling—making a long hole at each end to meet in the middle—would have been extremely difficult. It would have risked a high probability of either drilling through a face or missing the connecting drill hole entirely. Since replicative experiments have not been conducted, it is unclear whether this process or biconical drilling would have been more efficient. To minimize the effect of these obtrusive incisions and drill holes, inlays were cut to fill in the uppermost portions of the trenches and divots. It appears that string-saw debitage, derived from the same core as the plaques, was used to maintain color consistency between the repair and the surrounding jade.

In all cases the plaques were heavily polished on each long side, less so on the ends, suggesting they may not have been mounted to display those facets. It is uncertain if the faces of the plaques were polished with the inlays in place. Because they were affixed with an organic compound of some kind, it can be assumed that the inlays were cut and polished separately. Interestingly, the shorter plaques were sawn on the



wide face, while the longer plaques were sawn on the edge. It is puzzling why the lapidary would choose to mar such a flawless surface, although it may have to do with the circumference of the saw and the length of the plaque. In any case, and perhaps counter-intuitively, all four of the plaques were perfectly centered. Placed on a piece of cord, each plaque rotates freely, with no side more heavily weighted; in other words, none have any clear "up" or "down" side. How the plaques were hung on the belt is unknown at this time. The centered quality of the pieces suggests that intended free rotation is not out of the question.

The belt celts were all string-sawn

from a single celtiform axe into three equally thick sections (Figure I.3). The original celt was a strong, dark green color, but the jade is not translucent and may have begun its use-life as a utilitarian axe or chisel. Each of the celt plaques have perfectly matching string-saw scars, as well as matching fractures, one of which detached and created a divot that the lapidary polished later. The three celts were found in association with a roughly tubular bead, vertically drilled with tripartite, biconical holes that likely supported each of the three celts (Figure I.4). The bead was drilled biconically from the ends, then incised and polished, and finally drilled vertically. The piece is oval in cross-section,

and was decorated with chamfering at either end, a style common to some ceramic types. The massive plaques, one short and one long, could have flanked each side of the belt celt assemblage. According to the iconography of royal belt regalia, one of the mosaic masks recovered from the tomb may have been placed above the belt celt assemblage (see Chapter 3).

Mosaic tesserae in the tomb may compose one or more masks, but at present only one has been partially reconstructed, the other proving too fragmentary. Although the mask clearly featured two small imperial green earspools, little else is known about the iconography of the piece. Given its



Figure I.3. Plan and side views of the belt celts. Drawings: Sarah Newman.

exaggerated cheeks and large eye sockets, it probably represents a nonanthropomorphic deity. It also included shell tesserae and obsidian bits to accentuate the lips, eyes, and breath-scrolls. The jades that make up the bulk of the mask were shaped to fit tightly in lateral joins, but the bulbous nature of their polished, exterior surfaces conforms to Early Classic practice (see above). Most of the facial tesserae are of a similar color and may have come from the same core, perhaps derived from debitage produced during the manufacture of some other, larger piece. With continued reconstruction efforts, the identity of this deity will assuredly come to light.

The final jade of note recovered from the tomb was a semi-spherical bead of the highest quality imperial green color (Figure I.5). In view of its location close to cranial remains, the bead was surely placed in the mouth of the interred individual, a practice common in many parts of the Precolumbian Maya world (see Chapters 2 and 3). Aside from its high-gloss polish, a striking aspect of the bead is its large-diameter drill hole. Instead of the typical biconical drill pattern found in most Precolumbian beads, this example features straight sidewalls. Although the remnant ridges that accompany biconical drilling could have been ground away, it is more likely that the center of the bead was cut with a cane drill. A small central core-portion of this high-quality material could then have been saved for some other use.

Small fragments of jade were also found close to the cranium and near a corroded hematite mirror. At present, they do not appear to be easily related to any distinct object or item of dress.

Obsidian

An obsidian knife was found within the tomb, near the right side of the royal body on its bier—it lay atop other debris, suggesting



Figure I.4. Plan and side views of the tripartite tubular bead associated with the belt celts. Drawings: Sarah Newman.

Figure I.5. Plan and side views of the semi-spherical bead. Drawing: Sarah Newman.





an original location that was somewhat off the floor (Chapters 2 and 3). The knife is made from a large overshot blade removed from a polyhedral core of obsidian that can be sourced to El Chaval in highland Guatemala (Figure I.6). The overshot portion of the blade was thinned by percussion; the distal end of the blade was used as a handle. The knife appears to be encrusted with organic material and cinnabar, with heavy use wear. The platform was removed via pressure flaking, leading to an extremely sharp cutting tool that was effective on all edges. The use wear has been examined microscopically (see Appendix II), confirming initial suspicions that the knife was used to cut flesh and bone. It may be that the knife itself was used in the six human sacrifices included in the tomb and in slicing off the phalanges placed in cache bowls outside the tomb.

Specular Hematite Pigment Cakes

The tomb also contained the remains of a neatly stacked heap of fifteen brightly colored red balls of mineral pigment. These rested on a sedge or straw mat,



Figure I.6. Photograph and line drawing of obsidian knife with textural detail. Photo: Arturo Godoy; drawing: Zachary Hruby.

now visible only as a brown powder in the vicinity, leaving an impression on the bottom of several balls. Presumably moisture had entered the tomb after, or perhaps during, interment, softening the pigment—this may have been linked to water damage in the nearby wall or the explosion of a liquid-containing olla to southwest. Just to the west of the clustered balls lay a bone needle, raising the possibility that the tightly clustered lumps had been covered or sealed by a textile, perhaps to carry them into the tomb.

The term pigment cake, rather than ball, more accurately describes the objects. Most feature a roughly cuboidal shape, and the term has been used elsewhere to describe portable packages of raw pigment (e.g., Scott et al. 1996). The hematite was mixed with an unknown binder, perhaps with a light or nearly white-colored clay to increase consistency and avoid dilution or softening of its bright red color (Appendix IV). Despite this binder, the cakes are quite friable. As Scott and his colleagues (1996) have shown, a variety of binders were used in making Chumash pigment

cakes in California. Notably for the Chumash case, gas chromatographymass spectrometry analyses indicate that a mixture of human and antelope blood was employed in forming the cakes. Chemical analyses of the El Diablo samples may also reveal a similar range of binders. One feature at least was certain: an X-ray of several cakes by a laboratory in Antigua, Guatemala, as facilitated by Edwin Román, showed no other foreign material inside. The cakes were originally prepared and organized for long-distance trade but, in the context of this tomb, were likely offered as an overt sign of the king's economic reach and wealth. Shiny silvery specs in the material suggest that the main ingredient in the cakes was specular hematite, one of the most important red pigments for the Maya, especially during the Early Classic period (Houston et al. 2009:64). However, specular hematite was not, and is not, locally occurring in the lowlands. It must have been traded from the highlands or other as-vetunidentified sources. Although a few examples of singular, small hematite nodules have been recorded elsewhere,

Squar	e Ball #	Weights	Top length	Top width	Length	Width	Height	Notes
D9	3	644.72	7.9	6.2	9.5	8	7.3	<i>petate</i> print; weighed w/ plastic, w/o fragments
E9	4	556.02	5.5	5.2	8.5	7.3	5.4	malformed; part taken or squashed?
D10	3	577.35	5.6	5.3	8.1	7.5	6	
D10	1	598.95	5.35	5.1	8	7.8	6.3	most regular; flattened after pinched on two divots
E10	3	582.13	5.4	5.4	8	7.7	6.2	<i>petate</i> imprint; weighed with plastic; disintegration
E10	5	608.82	5.5	4.5	8	7.5	6.5	probably dry molded; possibly eroded on top; poss. shaped w/ leather
E10	2	662.69	6.2	5.75	8	7.6	6.2	measuring cube from center point of sides
E10	1	563.36	5	4.9	7.8	7.4	6.6	cube form
D10	2	603.11	6.05	5.4	7.8	7.3	6.3	very light <i>petate</i> imprint; disintegration
E9	1	639.56	5.9	5.5	7.7	7.5	6.5	good symmetrical example; rounded bottom
E10	6	661.85	5.9	5.2	7.5	7.2	6.5	most regular w/ knuckle imprint, palm imprint on top
E10	4	686.51	5.9	5.8	7.5	7.1	6.5	symmetrical
D9	1	666.95	6.2	5.2	7.5	6.9	6.6	
E9	2	610.08	5.8	5.8	7.4	7	6.1	petate imprint; fell on side; fragments missing
D9	2	591.46	6.6	5.85	7.3	6.8	6.5	<i>petate</i> print; weighed and measured w/o fragments; possibly fell over
Total		9253.56	88.8	81.1	118.6	110.6	95.5	into water upon wan conapse; natiened after pinched
A		(1(00	F 00	E 41	7.01	F 0 F	()7	
Average		616.90	5.92	5.41	7.91	1.37	6.37	

 Table I.1. Specular hematite measurements.

especially a single nodule in a royal tomb at Calakmul, Mexico (Pincemin et al. 1998:322, Fig. 11), the El Diablo examples represent the first systematically recorded samples from the Maya area. The archaeological rarity of these objects attests to their easily degradable nature, but also to the likelihood that they were highly valuable trade items.

Each cake in Burial 9 was molded by hand in an apparently expedient but controlled and duplicated manner. A slab of pigment colloid was placed on a flat surface, and then the palm and side of the hand were used to flatten opposite sides simultaneously. Once the plan view was roughly square, the top was flattened or dimpled, probably with the side of the hand or thumb. Measurements recorded and shown here in Table I.1 reveal that this last maneuver was important, since of all measurements the height, or thickness, of each cake was markedly regular (~6.4 cm). Nevertheless, except for a few examples that had come into contact with liquid anciently and subsequently lost integrity, most of the fifteen cakes were consistent in all dimensions. The regularity of size suggests some kind of measurement system was used to standardize the amount of pigment being produced and exchanged, and perhaps to make long-distance transport easier.

Each cake weighed roughly 0.6 kilograms, with a total weight of 9.25 kilograms for the group. Decay of organic materials in the cakes as well

as contact with foreign substances may have affected the original weight of these objects over the centuries. Given the relative irregularity of the weights compared to the regularity of the physical dimensions of the cakes, form and shape appear to have been the dominant measures. As mentioned before, the cakes lay on a mat, but the lack of an impression on the majority of the artifacts suggests that they were not originally formed on such a surface.

What also makes the cakes unusual in the tomb is that they have no clearly associated symbolism, are not obviously decorative, and were not finely crafted. Most Classic Maya tomb goods were embodiments of human excellence in crafting: jade beads, polychrome pots, carved bone, etc. The pigment cakes appear simply to mark raw economic wealth. An outside possibility is that the cakes may have symbolized a potent foodstuff for the deceased. The rough format of the cakes resembles iconographic representations of tamales, for example (see Houston et al. 2009:62: "[a] striking feature of the processes of making color is they resemble those used in preparing food"). The possibility of a cloth nearby recalls the covering of warm tamales. However, the cakes were not placed on any serving vessel and had no associated text to describe their meaning. In this, they were more like the utilitarian ceramics nearby, such as Vessel 21 (see Chapter 3).

Appendix II Microwear Analysis of the **Obsidian Macroblade** Kazuo Aoyama

This paper describes the results of a microwear analysis on the obsidian macroblade associated with the Early Classic Maya royal tomb (Burial 9) of El Zotz, Guatemala. Few researchers have conducted a detailed analysis of Maya stone tools, especially by means of the high-power microscopy developed by Lawrence Keeley (1980). This technique has great potential for answering critical questions regarding the organization of craft production as well as the domestic and ritual lives of the ancient Maya (Aldenderfer 1991; Aldenderfer et al. 1989; Aoyama 1995, 1999, 2001, 2007, 2009; Emery and Aoyama 2007; Lewenstein 1987, 1991; Sievert 1990, 1992; Stemp 2001, 2004; Stemp et al. 2010).

In 1987, I completed an intensive experimental use-wear study on obsidian and chert artifacts in Honduras to establish a framework for the interpretation of Maya stone-tool use (Aoyama 1989). The results of 267 explicative experiments on a range of worked materials permitted the identification of use-wear patterns by high-power microscopy. On this basis, I classified use wear on obsidian tools into 11 patterns, as inferred from a combined observation of surface striations, polish, and tiny pits. After these experiments, the study expanded to 7,000 stone artifacts from





Figure II.2. Use-wear pattern *c* and parallel striations on the lateral edge of the macroblade produced by cutting and sawing bone (200x). Photo: Kazuo Aoyama.

the Copan Valley and the region of La Entrada, Honduras (Aoyama 1995, 1999), Aguateca, Ceibal, and San Jerónimo, Guatemala (Aoyama 2004, 2007, 2008, 2009). This framework forms the basis for the lithic use-wear study on the obsidian macroblade from El Zotz. The equipment consisted of a metallurgical microscope of 50–500x magnification with an incident-light attachment (Olympus BX60M). Magnification of 200x was most frequently used, and an Olympus digital photomicrographic system (DP20-5) documented the use-wear patterns.

Activities Performed with the Macroblade from El Zotz

The nearly whole macroblade from El Zotz (14.1 x 5 x 2.6 cm, 148.6 g) was manufactured from an El Chaval obsidian macrocore (see also Appendix I). Such a large and thick macroblade has not been found at Late Classic Aguateca because El Chayal obsidian was imported to Aguateca mainly as polyhedral cores for prismatic blade production (Aoyama 2009:15). The El Zotz macroblade has a remnant natural cortex on its dorsal and ventral surfaces. It is a plunging (also known as "overshot") macroblade, meaning that the section near the distal end is the thickest. Both the proximal end and the upper half of the lateral edges were modified with a linear retouch to make sharp cutting edges, while the lower half of the lateral edges on the ventral side were rounded and dulled to make a handle by taking off larger flakes.

Following Patrick Vaughan (1985:56-57), I count each portion of a lithic artifact with interpretable use wear as an "independent use zone" (IUZ). A total of three IUZ were identified on the obsidian macroblade. Figure II.1 shows the distribution of use wear on it. Drawings are in Japanese technical style. Each lithic illustration shows the sequence of flake scar detachment. Flake scars, fissures, and ripple marks demonstrate the relationships of adjacent flake scars. Use-wear pattern *c* and parallel striations were identified on the upper half of the lateral edges of the macroblade indicating that it was used to cut and saw bone. Moreover, use-wear pattern *d* and perpendicular striations were observed on the proximal end of the macroblade indicative of whittling bone.

Pattern c: The polish surface is bright and flat but rough, pitted, and marked by clear striations. Pattern *c* is produced by cutting and sawing bone (Figure II.2).

Pattern d: The polish surface is bright, smooth, and flat, with slightly rounded extreme margins. Infrequent thin striations and a few tiny pits are observable in the polished surface. Pattern *d*,



Figure II.3. Use-wear pattern *d* and perpendicular striations on the proximal end of the macroblade indicating usage in whittling bone (200x). Photo: Kazuo Aoyama.

which is associated with perpendicular striations, results from whittling bone (Figure II.3).

In sum, the analyzed obsidian macroblade from El Zotz was a hand-held tool used to cut, saw, and whittle bone. The large and thick macroblade was well suited for such a heavy task. To judge from other lines of evidence, such as the blood-red bowls containing human fingers and teeth as well as the six children associated with the remains of the ruler in the royal tomb, the macroblade in question may well have been used for human sacrifice. Furthermore, although the substance still needs to be chemically tested preliminary results from Prof. Takamitsu Kohzuma, a colleague at Ibaraki University, were inconclusive-the surface of the macroblade was covered with red organic residue, as well as orange and blue residues (Figures II.4 to II.6).

Appendix III **Artifact Conservation** Catherine E. Magee and Tessa de Alarcon

Conservation of materials associated with El Zotz Burial 9 took place in two distinct phases. The first, from June 5 to June 23, 2010, focused on the excavation, documentation, and safe transport of materials from El Zotz itself. Additional work stabilized the stucco masks from the



Figure II.4. Red residue on the macroblade (200x). Photo: Kazuo Aoyama.



Figure II.5. Orange and blue residues on the macroblade (200x). Photo: Kazuo Aoyama.



Figure II.6. Orange residue on the macroblade (200x). Photo: Kazuo Aoyama.

exterior of Str. F8-1-Sub.1B and Sub.1C (the Temple of the Night Sun), now partially exposed through tunnel excavations. In 2011, head conservator Catherine E. Magee and UCLA conservation intern Tessa de Alarcon returned to the El Zotz Laboratory in Antigua, Guatemala, to further examine and treat all materials excavated during the 2010 season, conduct technical analyses where possible, restore threedimensional objects and mosaic masks, and pack materials for storage and shipment to Guatemala City. This work, which helped to preserve artifacts, animal and human remains, and architectural elements, used materials and techniques in accordance with the code of ethics and standards of practice of the American Institute of Conservation of Historic and Artistic Works. As of January 2012, all objects had been



Figure III.1. Elements of the king's headdress after conservation treatment. Photo: Catherine Magee.

presented to, and catalogued by, the Înstituto de Antropología e Historia de Guatemala.

Summary of Work

In 2010, conservation efforts focused on the gridded excavation of objects from the tomb chamber (see Chapter 1). In some instances, this included lifting multiple objects with cyclododecane, a temporary wax binder for sealing friable and structurally weak materials; this material slowly sublimes when exposed to air. Molten cyclododecane was used to consolidate and, when solidified, lift several key objects from the tomb as blocks. These could then be transported to the El Zotz Laboratory in Antigua to be excavated without the constraints on time and resources at the field site. Such objects included Jade Mask 2, located in Sector B6 of the tomb, animal remains

within Vessel 18B, the Spondylus shell beads forming the king's necklace, and several of the fragile, three-dimensional stucco objects. Other delicate materials, primarily thin stucco objects, were faced with Japanese tissue and consolidated with B-72 in acetone, methycellulose in distilled water, or a combination of these two adhesives. The high-humidity environment of the tomb chamber proved challenging, often preventing a methylcellulose consolidant from drying. After several tests on small stucco fragments, it was determined that the best method involved a combination of water-based Rhoplex, CM Bond, and methylcellulose to provide some strength to the stucco and adhere the Japanese tissue facings. Additional B-72 was applied to the facing fabric after application and slow-drying of the water-based consolidant. Objects were



Figure III.2. Pyrite mirror associated with the king's headdress. Photo: Catherine Magee.

packed at the site, using a combination of toilet paper, Kim wipes, aluminum foil, polyurethane foam, and plastic containers.

Conservation efforts in 2010 also addressed the condition of the stucco masks decorating the facade of F8-1-Sub.1B and Sub.1C. Multiple applications of stucco in antiquity had begun to delaminate, mainly due to root growth between the limestone substructure and the stucco

layers. Examples of this delamination were photographed prior to treatment. Lime plaster was prepared using screened construction fill from the El Diablo pyramid itself and lime mixed with purified drinking water. Rootlets were removed prior to consolidation if their removal did not compromise the structural integrity of the stucco. In areas where weak adhesion was desired, the lime plaster was applied

with metal spatulas to the crevices and along the edges of the delaminating stucco layers. Where greater adhesion was required, the delaminating surface was first dampened with water before the application of lime plaster along delaminating edges. In some instances, small lime plaster bridges were made to secure especially fragile stucco layers or applied beneath protruding areas of stucco at risk of crumbling. The new lime mortar is lighter in color and softer than the original plaster. After treatment, digital images were taken to detail the different types of stucco consolidation used at El Diablo.

In 2011, conservation efforts focused on the restoration and reconstruction of three-dimensional stucco and jade objects from Burial 9, laboratory excavations of multiple objects that had been block-lifted with cyclododecane, evaluation of the conditions of artifacts remaining in their original packing, and the repacking of objects for transport to the Museo Nacional de Arqueología y Etnología in Guatemala City. The majority of the items recovered were small fragments or chips of painted stucco or unidentifiable organic remains. For many of these items, treatment was limited to removal from their original packaging, cleaning with soft brushes and ethanol as necessary, exposure to UV light to prevent mold growth, and rehousing in clean packing materials. Several items, however, received intensive treatment, such as the headdress of Skeleton A (the probable ruler) and associated pyrite mirror, jade mosaic masks, and the remains of several polychrome stucco objects, including a four-footed vessel with three-dimensional feet in the shape of peccary snouts and a lid featuring a three-dimensional water bird with a fish caught in its beak, a small red bowl, and several fragments bearing the remains of painted hieroglyphs.

Skeleton A's Headdress and Pyrite Mirror

The king's headdress was originally block-lifted during the 2010 field



excavations using cyclododecane faced with gauze, wrapped in aluminum foil, and placed inside a plastic bag to prevent the cyclododecane from subliming between the 2010 field excavations and the 2011 conservation work in the Antigua laboratory. In the laboratory, the block lift was exposed to air beneath an incandescent light/heat source to allow the cyclododecane to sublime, using soft brushes and an air puffer to remove loose material. Although the headdress was not completely reconstructed, several features related to its form were noted during conservation (Figure III.1). An oblong shell ornament formed part of the (viewer's) lower right of the headdress, with a possible bone inclusion on the lower left side. A curved organic material including bone, possibly following the curvature of the top of the king's cranium, was found in the middle of the block lift adjacent to organic textile remains; it, too, was consolidated with B-72 in acetone. Wood was detected under this curved organic material, progressing toward the top of the block lift, at the northern end of the tomb. Most

Figure III.3. Complex items from Burial 9 were block-lifted using cyclododecane wax, later sublimed beneath incandescent light in the laboratory. Photo: Catherine Magee.

likely, however, this material represented remains of the funerary bier, rather than an original part of the headdress. The wood was consolidated with B-72 in acetone. Approximately three-quarters of the headdress's pyrite mirror was recovered from the headdress (Figure III.2). Its reflective surface faced up, comprising small cubes of polished hematite (mostly disintegrated) set into a plaster-like material atop the clay mirror backing. On the non-reflective surface of the mirror, several fine layers of stucco covered the clay substrate, including a final thin layer of white stucco with hieroglyphs painted in black. The sides of the mirror were covered with intense green and red paint, a palette found on other stucco objects from the tomb. The friable painted glyphs and thin stucco layers were faced with methyl-cellulose in distilled water and Japanese tissue to provide strength or consolidated with B-72 in acetone.

Jade Mosaic Masks

Two separate jade masks were treated during the 2011 laboratory season. The first,

Mask 1, recovered from Sector A5 within the tomb chamber, could not be reconstructed, but was excavated and consolidated. The 83 pieces of this jade mosaic mask were block-lifted during the 2010 field excavation of the El Diablo royal tomb using cyclododecane. In the field, molten cyclododecane was poured over the sector containing the disarticulated pieces of the mosaic. When solidified, the wax block was wrapped in aluminum foil and placed in a plastic box padded with polyurethane foam for transport to the El Zotz laboratory in Antigua. The block lift arrived at the laboratory intact, with jade, bone, red pigment, stucco, and powdery organic residue noted on the surface of the cyclododecane. The cyclododecane wax was allowed to sublime for two days to loosen the encased materials before the block was excavated from its underside (Figure III.3). A tan- or pink-colored material (possibly a thin layer of stucco) was found on the underside of the individual jade pieces, as well as a white material (again, possibly thin stucco) with stains or imprints of wood grains below the tan/pink layer. This suggests that the individual jade tesserae adhered to a wooden substrate, with two separate layers of adhesives. Textile remains were found within the cyclododecane of the block lift, some with a layer of red pigment and fragments of bone attached, though these are most likely part of Skeleton A's funerary attire rather than part of the jade mosaic mask. Although this mask could not be reconstituted, a labeled drawing was made of the individual pieces of jade and their orientations and the textiles, pigment, and bones were consolidated with Japanese tissue facing and methylcellulose or B-72 in acetone.



Figure III.4. Mask 2, partially reconstructed in the laboratory. Photo: Tessa de Alarcon.

The second jade mask, Mask 2, recovered from Sector B6, was originally lifted with cyclododecane in two separate blocks. This mask includes a mosaic jade face, along with inlaid shells to create the mouth, teeth, and brow (Figure III.4). The backing appeared to be more complex than that of the jade mosaic mask from Sector A5. In several areas, such as the nose and eyes, shaped bone was found directly beneath the jade or shell pieces, possibly used to level the tesserae over the initial supportive backing. Dark brown powdery residues suggest that this original base was made of wood. Some jade tesserae have textile remaining on the undersides, while others show vestiges of stucco, particularly in the case of the eye pieces. The majority of the backing or support was severely deteriorated and friable, however, and its original form and composition remain unclear.

As noted, the mask from Sector B6 was lifted in two separate blocks of cyclododecane wax. As with the jade mask from Sector A5, both wax blocks were flipped over and excavated from the underside. A soft brush and air puffer were used to clean the object, conserving the loose soil and organic materials removed during cleaning. To record the in situ locations of the tesserae, a tracing of each lift was made on clear plastic using a felt-tip marker before complete sublimation of the cyclododecane. Although the two block lifts were thought to represent two separate masks during excavation in 2010, overlaying these two drawings demonstrated that the two wax blocks formed a single mask. A desk lamp with an incandescent bulb accelerated the sublimation of the cyclododecane, along with a hair dryer on low heat where the wax was thickest. In addition to the soft brush and air puffer, tweezers were also used to remove large debris, jade tesserae, and shell pieces. Fragmented jade tesserae were faced with Japanese tissue and methylcellulose, which were also placed across the tesserae on the front of the mask to maintain their alignment and orientation; these were removed with water and acetone as excavation progressed. Japanese tissue and methyl cellulose were also employed to face a compact mixture of clay-like material, bone, and organic residues found beneath the jade tesserae. This backing possibly represented the original support for the mask. Some shell pieces began to delaminate as the cyclododecane sublimed and were consolidated with B-72 in acetone. Organic materials still in contact with the jade tesserae were also consolidated with B-72 in acetone, in order to preserve them in situ. The complete support behind the left shell eye was recovered intact and consolidated with B-72 in acetone. Once the mask was reconstructed, the jade tesserae were adhered using Paraloid B-72 in acetone and backed with Japanese tissue and B-72 in acetone to create a rigid support. Where the original support was preserved in situ, B-72 in acetone was used without the addition of the Japanese tissue backing. A fragment of mica representing the pupil or iris of the right eye was located during secondary examination of the loose materials conserved during excavation of the block lifts. This fragment was



Figure III.5. Painted stuccos formed a thin layer atop wooden sculptural elements. Photo: Jorge Pérez de Lara.

cleaned enzymatically and attached to the right eye with paraloid B-72 in acetone. Finally, a mount for the mosaic mask was made from Ethafoam (a resilient, medium-density, closed-cell polyethylene material), its individual pieces joined to one another and to a mat board backing using the PVA adhesive Jade 401. The mount was joined to the mask using a Japanese tissue hinge, attached to the mask and the mount using Paraloid B-72 in acetone. This hinge was added to ensure that the mount would be fixed to the mask and provide sufficient support for the jade fragments. An advantage was that it would still allow the mask to be lifted to reveal the areas of preserved, original backing.



Figure III.6. Fragments of three-dimensional stucco objects with imprints of black-painted designs on interior of stucco lavers. Photo: Catherine Magee.

Polychrome Stuccoes (Quadrapod Vessel, Red Bowl, and Fragments with Hieroglyphs)

The quadrapod vessel with peccary snout feet, the red bowl, and other three-dimensional stucco objects consist of innumerable painted and unpainted stucco fragments of various sizes. These were faced in situ with Japanese tissue in 2010. In 2011, the materials were removed from their original packaging for evaluation and consolidation before being repackaged for transport to the Museo Nacional de Arqueología y Etnología. Where possible, these fragments were cleaned using a soft brush, and flaking paint was stabilized with methylcellulose in distilled water. Some of the stucco fragments were

consolidated with B-72 in acetone while still in the tomb chamber. This allowed lifting and stabilization, in that CM Bond and Rhoplex in water were not able to penetrate the fragments. Methylcellulose rather than B-72 was applied to allow subsequent conservators to reshape the fragments with greater ease. Stucco fragments with hieroglyphs or potential hieroglyphs, however, were consolidated with B-72 and adhered to a Japanese tissue backing with methylcellulose for stability.

Variations were noted in the striations and imprints found on the underside of many of the polychrome stucco fragments. This suggests that original objects were formed of different substrates before being covered in

stucco. Some of the stucco fragments clearly show imprints or staining characteristic of wood grains; in fact, some fragments were found attached to preserved wood as well (Figure III.5). Others displayed striations and patterns that could not be attributed to wood, but neither could they be conclusively identified as another type of material. Several appear to have been carved from organic materials, possibly gourds, augmented by a clay-like material, a substance clearly visible in the remains of the stucco water-bird head. Other objects seem to possess two distinct layers of stucco, an interior layer that is roughened and striated, perhaps to assist in adhesion of the exterior layer, and an exterior layer that is finer and

smoothed. This last layer also holds the polychrome paint or hieroglyphs observed in many of the objects. The rough and fine stuccoes easily delaminate from one another.

Many of the three-dimensional objects, including the peccary snout feet of the quadrapod vessel, consist of primarily pink stucco with red and green detailing to indicate designs. Some fragments were also found to have retained imprints of hieroglyphs or painted designs highlighted in black. These hieroglyphs were discerned on the interior rough stucco layer, suggesting that they were originally painted on the organic substrate before the polychrome, molded stucco covering was applied (Figure III.6).

Appendix IV Materials Analysis Kristina A. Cheung, Nuoya Xie, Zhaoying

Yao, Christian Fischer, Vanessa Muros, Sergey Prikhodko, and Ioanna Kakoulli

Materials and Methods

Thirteen archaeological specimens (small pieces separated from original artifacts) were brought to the laboratory for analysis, including organic specimens resembling wood, basket, cord, bone, pseudomorphic textiles, clay, specular hematite pigment (from cubes of hematite found in the tomb), fragments of green painted stucco, and red pigment powders found inside shells. Table IV.1 lists all specimens alongside the archaeologists' initial descriptions of the samples and preliminary stereomicroscopic observations.

Sample Preparation

Minute quantities of material (in powder or bulk form) were removed from representative areas of the fragmentary archaeological specimens and prepared for analysis using two different types of samples: dispersions and polished cross-sections.

For the preparation of dispersion samples, a few particles were removed (by scraping) from the areas of interest on each specimen and placed on a clean glass microscope slide. Meltmount thermoplastic resin with RI=1.62 was melted (in a double boiler over a hot plate at 50° C) and dropped over the particles. A glass cover slip was positioned on the top and the glass microscope slide was then put on a hot plate to allow the resin to become soft. Using an eraser tip from a standard pencil, mild pressure was exerted over the glass cover slip dispersing the particles in the resin while removing all trapped air. This provided a semi-permanent mound for the particles to be analyzed under polarized light microscopy (PLM).

For the preparation of cross-sections, stratigraphic or bulk samples were removed from each archaeological specimen using the point of a scalpel. Owing to the small size of the samples, a special preparation technique was used for embedding (Plesters 1956). Buehler EpoxiCure epoxy resin (mixed with EpoxiCure Epoxy Hardener as prescribed) was poured into custom-made rubber cubic molds (1.5 x 1 x 1 cm) to fill half their volume. After the resin hardened, the sample

Sample	Archaeologists' description	Stereomicroscope observations	Digital Photomicrography
01 Sector C13	Remains of some kind of basket; possibly cord.	• Off-white/cream color with some brown areas; fragile, porous structure; orange- brown surfaces with white particles.	
02 Sector B3	Possibly bone, but seems to be either coated or mixed with another material.	• Brown clay-like color and texture; porous; white particles sprinkled throughout the surface.	
03 Sector B6	Clay + pigment that may have been used to wrap the body.	 Top side: brown body with red color on surface. Bottom side: brown with white particles. Spongy and porous structure; clay/earth-like texture; white fibers; iridescent spots. 	
04 Sectors B9, C9, B10, C10	Clay + pigment/ painted stucco that may have been below the body.	 Top side: pinkish color. Bottom side: dark black/ brown color; white particles. Porous structure; web of white fiber on surface. 	
05 Sector B5	Clay + pigment that may have been used to wrap the body.	 Dark brown areas with bright red/orange paint; clay-like material; grey web-like fibers on surface; areas of iridescent black spots; dispersed white particles. 	
06 Sector B9	Clay matrix.	• Dark brown with web-like structure; whitish and yellow particles on surface.	
07 Sector D5	Remains of some kind of cordage.	• Dark brown matrix with parallel striations; wood-like; possibly basket.	
08 Sector D3	Remains of some kind of cordage.	• Dark brown matrix; wood-like; possibly basket; white and other spherical particles; silver-looking threads dispersed throughout.	
09 Sector D6	Green painted stucco tragments.	 Top side: green paint and brownish yellow debris on the top; dark green line across green surface with correspond- ing darker brown line on back side of fragment; Bottom side: whitish layer. 	
10 Sector A6	Some kind of grey material with impressions.	• Top side: off-white surface with grainy areas of dark brown/black color; white particles dispersed throughout; pseudomorphic textile texture; powdery white particles on edges.	
P1 Sector E10	Pigment from cubes (Sector E10).	• Largely containing brick-red particles and tabular black translucent particles (perhaps specular hematite) and specs of white particles.	
P2 Sector B7	Pigment from Spondylus shell (Sector B7).	 Brick-red particles with a few distinctive orange particles and black translucent tabular particles (perhaps specular hematite) and a few white friable particles. A few hard pebble-like particles are also present. 	A
Р3	Unknown pigment associated with shell necklace.	• Mainly orange particles with yellowish/brownish impurities (possibly organic).	

Table IV.1. Listing of specimens with archaeologists' initial descriptions of the samples and preliminary stereomicroscopic observations.



Figure IV.1. Portable XRF spectra (counts [log] vs. energy in keV) of pigments Samples P1 (red spectrum), P2 (blue spectrum), and P3 (orange spectrum). P1 and P2 are almost identical, showing high Fe content and minor Si and Ca. Characteristic X-ray lines for Hg and S in P2 and P3 spectra are attributed to the presence of cinnabar.

itself was then placed in the mold with the outer surface (top surface) facing downwards. More resin was poured over the sample until it was completely covered and put under vacuum using a Buehler Cast N' Vac 1000. Once the resin had set, the samples were cut perpendicular to the outer surface using Buehler silicon carbide grinding papers from 240 to 600 grit. Because of the water-sensitive nature of most of the samples, grinding and polishing could not be done with water-based emulsions. Samples were ground using ethanol and dry polished using Micro-Mesh polishing pads from 1500 to 8000 grit until a well-polished surface was established. In some cases wet polishing was carried out using the alcohol-based Leco Ultra Lap Diamond Extender Part No. 812-433 together with Leco Microid Diamond Compounds Part No. 810-871 of 3 microns and Part No. 810-870 of 1 micron. Polishing suspensions were spread on Buehler MasterTex polishing cloths attached to the Leco GP-25 polishing turntable.

Characterization Methods The specimens in Table IV.1 were analyzed qualitatively and quantitatively to infer information as to their chemistry, microstructure, and properties. All samples displayed some degree of physical and chemical alteration due to the burial environment. To distinguish between products of alteration and original materials, the analysis of the samples involved a multi-scale and multi-analytical approach from the macro- to the molecular-length scale, using stereomicroscopy, digital microscopy, field emission gun (FEG) scanning electron microscopy (SEM) at variable pressure (VP) coupled with energy dispersive X-ray spectroscopy (EDS), portable X-ray fluorescence (pXRF), ultraviolet/visible/near infrared reflectance spectroscopy (UV/ Vis/NIR), and X-ray diffraction. The combination of these noninvasive and non-destructive imaging and analytical techniques provided complementary information and enabled the use of the same sample for multiple analyses with minimal sample preparation and manipulation.

Initial observations of the unmounted samples were recorded using an Omano stereomicroscope between

7x and 45x magnification and a Canon PowerShot A630 digital camera. These samples were also examined under a KEYENCE VHX-1000 digital microscope at 20x, 50x, 100x, and 200x magnification, focusing on specific areas of interest in each sample, which would be later analyzed using VPSEM-EDS.

Dispersion samples of pigmented areas were analyzed using a Leica DMRM polarized light microscope under plane polarized and cross-polarized light. The habit, color, pleochroism, relief, refractive index, birefringence, and extinction angles were used to characterize each particle phase identified in the sample.

For elemental analysis, the Thermo Scientific Niton XL3t Series GOLDD technology handheld portable XRF was used, with a silver anode and silicon drift detector. Readings were taken with an 8 mm spot size in both Soil and Mining Mode for 120 seconds for each measurement. In situ study of the samples with this technique gave qualitative and semi-quantitative information regarding the relative concentrations of major, minor, and trace elements found in each sample and complemented other microanalytics.

X-ray diffraction (XRD) analysis was performed on the pigment powder samples and on Sample 09, which contained a green-painted layer on white stucco. For the analysis, a few particles of the area of interest were mounted on a glass spindle and analyzed using a Rigaku R-Axis Spider X-ray diffractometer. XRD spectra were recorded at 50 kV/40 mA using a Cu-Ka target for 900 seconds. XRD data was processed and matched against reference spectra from the International Center for Diffraction Data files using the JADE, v8.2 software from Materials Data Inc.

Ultraviolet/visible light/near infrared (UV/Vis/NIR) reflectance spectroscopy was performed using the FieldSpec3 by Analytical Spectral Devices, with high spectral resolution (3 nm @ 700 nm and 10 nm @ 1400/2100 nm) and wide spectral range between 350–2500 nm. The flexible spot-size analyzer for contact analysis facilitated the systematic study of specific areas. The high spectral and spatial resolution of the spectrometer was particularly useful for fingerprint identification of different mineral phases and organic compounds in the samples due to its high sensitivity to both the electron transitions in the



Figure IV.2. Digital micrograph of Sample 03 at 100x magnification, showing cinnabar alteration with black spots. Photo: Kristina Cheung.

visible part of the spectrum and the overtones from the organic molecules in the near infrared.

Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) analysis was performed using an FEI Nova NanoSEM 230 scanning electron microscope with field emission gun and variable pressure capabilities, equipped with a Thermo Scientific NORAN System 7 X-ray Energy Dispersive Spectrometer. All archaeological specimens were first examined noninvasively (no conductive coating was sputtered over the surface) at low vacuum, so as not to damage, dehydrate, or alter the delicate nature of the specimens. Imaging was provided using secondary electron (SE) detection with the low vacuum detector (LVD) providing topographical details of the samples. For this analysis, each sample was placed on an aluminum foil holder, which was secured to the SEM stub using double-sided carbon tape. In addition, samples were blown with compressed air before being placed inside the SEM chamber to remove any loose surface debris. Chamber pressure for all in situ analyses was held at 50 Pa

Polished cross-sections were also

analyzed with SEM-EDS for spatially resolved inter- and intra-layer visualization and characterization. A gaseous analytical detector in variable pressure was used for the detection of backscattered electrons (BSE), providing images with compositional contrast; atoms of heavier elements elastically scatter electrons more strongly compared to those of lighter elements, resulting in higher signal detection for elements with higher atomic numbers. Thus, areas of the sample that are mostly composed of heavy elements appear brighter than areas composed of light elements in an image obtained using BSE, providing useful information when studying the heterogeneous and multilayered samples.

Elemental spectra and maps of characteristic X-ray photon emissions were acquired using EDS. The analysis of well-polished surfaces was crucial for more precise quantitative measurements because of the shallow probing depth of electrons interacting with the surface. In addition, flat surfaces minimize the deflection of BSE in different directions, maximizing the collection of electrons by the detector located symmetrically about the incident beam of electrons. EDS spot analysis enabled comparisons of peak intensities, providing data regarding relative concentration of elements found in the specimen, and elemental mapping of certain areas provided a visual of the profile distribution of certain elements.

Preliminary Results and Discussion

Red powder, from pigment ingots (Sample P1) and from pigment-containing shells (P2 and P3), was analyzed using PLM, pXRF (Figure IV.1), and XRD. PLM results of Samples P1 and P2 revealed the presence of specular hematite as indicated by the occurrence of reddish-brown granular particles identified as hematite associated with tabular flakes with shiny luster and quartz particles. XRD analysis confirmed the presence of hematite and quartz and has also suggested the presence of a calcium-based clay material. The results were further supported by XRF spectroscopy, determining the presence of Fe as the major element and Si and Ca as minor elements. Traces of



Figure IV.3. SE image of biological colonies found in black regions of cinnabar on Sample 03. Photo: Kristina Cheung.



Figure IV.4. BSE image of a cross-sectional view of Sample 03, showing remnants of cinnabar (bright particles) near the pit crater-like formation with microorganisms at the bottom and side walls. Photo: Kristina Cheung.

Ti were also identified that might suggest the presence of a titaniferous mineral associated with hematite. Sample P2 also contained traces of another pigment (most likely an accidental contaminant due to depositional processes) with angular habit, intense red body color, and high relief. This red powder was identified as cinnabar, a mercury (II) sulfide compound (α-HgS). Its presence was further validated by XRF with the identification of Hg and S. Sample P3 was identified as pure cinnabar.

The specular hematite ingots (15 of them) found at this archaeological site are the first of this quantity and form to be recorded in the Maya region. The pigment is not indigenous to the area, which confirms that these ingots must have been traded from another location. The scarcity of the pigment ingots is most likely due to their status as valued trade objects. Similarly, cinnabar is a very important and highly prized pigment that has been associated with burials of important Maya rulers and funerary rituals of venerated ancestors (Batta et al. 2013; Bell et al. 2000; Harrison-Buck et al. 2007; Vázquez de Agredos Pascual 2007).

In addition to raw pigment samples, the red color was also evident in painted surfaces of Samples 03, 04, and 05. Samples 03 and 05, described as clay that may have been used to wrap the body of the king, look very similar in appearance and were found near the same location in the tomb. Sample 04 (possibly a piece of painted stucco) is pigmented with a lighter red color. UV/Vis/NIR reflectance spectroscopy on all three samples revealed absorptions in the visible range indicative of electronic processes of Fe³⁺, attributed to the presence of hematite. XRF analysis confirmed the presence of Fe with higher concentrations in Samples 03 and 05. The red pigments in Samples 03 and 05 also appear brighter than the red found in Sample 04. This could be related to the occurrence of cinnabar, since Hg and S were also detected in Samples 03 and 05 using XRF and EDS. UV/Vis/NIR reflectance spectra of these strongly red-tinted layers clearly displayed the characteristic band-gap of S centered at ~610 nm, confirming the presence of cinnabar.

Close examination of Sample 03 demonstrated evidence of degradation with the formation of black spots in the cinnabar-rich layer applied over a clay body (Figure IV.2). Morphological characterization of these black areas using secondary electron (SE) imaging revealed the presence of microbiological colonies, most likely responsible for or contributing to the formation of the black staining



(Figure IV.3).

Qualitative EDS spectra on the surface of the black spots Although these are only speculations, we suggest two

showed significant levels of C, O, and Ca and depletion of S and Hg. BSE images of a polished cross-section of this area indicated a pit crater in the sample (down to the clay body) and the deposition of microorganisms close to the walls of the crater (Figure IV.4). EDS spot analysis on the polished sample confirmed the high levels of C, O, and Ca in the black regions and mainly Al and Si in the clay matrix (Figure IV.5). While the C and O could be easily attributed to the presence of organic matter and the Al and Si to the clay body, the occurrence of Ca is much less clear. It could potentially be a by-product of microbiological metabolism in the form of calcium oxalate. possible pathways for the formation of the black stains: (1) the presence of microorganisms (bacteria, fungi, or algae, including cyanobacteria) that can cause the breakdown of the sulfide mineral leading to the biotransformation (Vázquez de Agredos Pascual 2007) or bioleaching (Bosecker 1997; Hansford and Vargas 2001; Suzuki 2001) of Hg, and (2) the presence of detoxification bacteria that can remove Hg by reduction of Hg²⁺ to Hg⁰ and final volatilization of Hg (Mathema et al. 2011; Nascimento and Chartone-Souza 2003; Wagner-Döbler 2003). As the dark areas do not contain much of the original Hg and S, the staining may be of organic origin formed by microorganisms (Martin-Sanchez et al. 2012; Vasanthakumar et al. 2013).

Sample 02 was described by the archaeologists as possibly bone mixed or coated with another material; however, photographs taken with the digital microscope suggest this sample may actually be a fragment of gourd, which was



Figure IV.5. (Top) EDS spectrum of biological black regions in Sample 03, showing intense characteristic X-ray emissions of C, O, and Ca; (bottom) EDS spectrum of the clay matrix beneath the cinnabar layer with major elements, Al and Si.



Figure IV.6. UV/Vis/NIR reflectance spectrum of Sample 06, with the second derivative of boxed region shown beneath. Peaks of the second derivative correspond with absorptions in the original spectrum, showing fundamental vibrations of cellulose-based material at 1680, 1730, and 1760 nm.

commonly used to make vessels. The cross-section of this sample looked morphologically similar to wood, with alternating dense and porous areas. The UV/Vis/NIR reflectance spectrum shows absorptions at 1680, 1730, and 1760 nm corresponding to overtones (higher harmonics) of molecular fundamental vibrations of cellulose-based material, strengthening the hypothesis that this material may be gourd "wood" (Figure IV.6). However, further investigation is necessary to precisely match the organic signature of this material. XRF analysis revealed the presence of some inorganic elements, such as Fe, Ca, P, and Si. Phosphorous is likely present on the sample from the decomposition of the human remains, while Fe, Ca, and Si may be present as a result of the burial soil that has cemented the fibrous material.

Sample 06 (Table IV.1), a dark brown porous sample, was recorded during excavation as a clay-based material. UV/ Vis/NIR reflectance spectroscopy suggested the presence of clays and traces of organic materials. XRF analysis further indicated the presence of Hg. SEM using SE imaging of a small piece separated from Sample 06 indicated a



Figure IV.7. (a) SE micrograph of a detail of Sample 06 showing the lattice-shaped (spongy) structure; (b) magnified area indicated by the white square in *a*, showing the needle-like crystals and one of the spherical-shaped particles; (c) spectrum of the elemental composition of the spherical particle within the area marked with a black square in *b*, showing Ca, Si, O, P, and Al as the major elements with C, Mg, and Fe as minor elements. Photos: Kristina Cheung.





Figure IV.8. (a) BSE micrograph of a cross-sectional view of a small piece of Sample 06; (b) BSE detail view of the area indicated by the white square in *a*, showing sphere-shaped clusters and quill-like structures. Photos: Kristina Cheung.







Figure IV.10. UV/Vis/NIR reflectance spectra comparing [1] standard malachite to [2] the green pigment in Sample 09.

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Figure IV.9. BSE micrograph (top left) and EDS elemental maps on a cross-sectional view of one of the spherical particles, showing the spatial distribution of the predominant elements detected: C, Ca, and P. Ca and P are the major elements found in these spherical structures (pointing towards a calcium phosphate mineral) while C corresponds to the voids filled with resin. Images: Kristina Cheung.

lattice-shaped (spongy looking) structure with needle-like crystals and spherical particles (\emptyset 50–80 µm) (Figure IV.7). EDS analysis of the backbone of the spongy structure revealed the presence of Si and Al and traces of P, Ca, and Mg, while the spherical particles showed higher concentrations of P and Ca (Figure I.7c) compared to the backbone structure. The analysis of a polished cross-section indicated the presence of particles (assumed to correspond to the microspheres mentioned above) made of a system of concentric lamellae grouped together in circular arrangements (Figure IV.8). Elemental analysis and mapping of these circular features using EDS showed the characteristic X-ray emissions of Ca and P, suggesting the presence of a calcium phosphate phase (Figure IV.9). The results so far are preliminary and further analysis of this sample is required to provide more understanding about its precise identity and relationship to its archaeological context.

Sample 09 is a relatively thin sample with a green paint layer (top) of approximately 100 microns and a white layer (substrate) of approximately 0.7 mm. Brown residues beneath the white layer may indicate the presence of a wooden structure that has been lost. It is believed to have decorated the bier on which the buried king was placed. The sample was first analyzed noninvasively using XRF and UV/Vis/NIR spectroscopy from both sides. XRF results of the top layer (green) indicated the presence of Cu and, to a lesser extent, Ca and P. UV/Vis/NIR reflectance spectra comparing standard malachite ($CuCO_{2}(OH)_{2}$) to the green layer in Sample 09 showed absorptions at ~800 nm and 1300 nm corresponding to electronic transitions of Cu ions and between 2200 and 2400 nm indicative of the CO₂ and OH groups characteristic of malachite (Figure IV.10). An absorption at ~1920 nm is likely due to the presence of water within the sample. XRF results of the



Figure IV.11. (a) Digital photomicrograph of Sample 09 showing the green malachite layer on top of the white stucco layer; line 1–1' indicates the area sectioned for the preparation of a cross-section; (b) the cross-section as indicated in $\frac{1}{a}$; (c) BSE micrograph showing detail of *b* indicated by white square, illustrating the interface between the green layer and the stucco layer; the brightness of the green layer indicates its higher density compared to the darker stucco layer beneath. Photos: Kristina Cheung.

white substrate showed Ca and P with less evident traces of Cu. UV/Vis/ NIR reflectance spectroscopy yielded interesting results in the organic region with small absorptions corresponding to the first overtones of CH stretching, suggesting the presence of organic matter in the white substrate.

The examination of a polished section of Sample 09 using SEM clearly revealed the two distinct layers: a green layer (top) appearing brighter under BSE detection and a Ca and P-rich layer (substrate) consisting of angular aggregates in a microcrystalline matrix (Figure IV.11). EDS analysis of this sample showed the presence of Cu in the green paint layer (Figure IV.12) and Ca and P in the aggregates of the white substrate (Figure IV.13). EDS analysis of the microcrystalline matrix revealed a Ca-rich layer with some P (Figure IV.14). The results from this analysis suggest that the binder might have been calcium hydroxide—Ca(OH), transformed into CaCO₂ after carbonation through a chemical reaction with the CO₂ of the atmosphere—mixed with crushed bone (as inert aggregate) to form the white stucco substrate.

Outside the burial, one of the lip-tolip vessels recovered in 2012 (Vessel 9D from Cache 9) contained whitish-grey powder that resembles pulverized bone. Further investigation is required to identify the composition of this powder and its relation to the white stucco substrate. DNA analysis will also be conducted to identify whether the bone fragments in the white stucco layer are human or animal.

Summary and Conclusion

Paint (green)

Substrate

The scientific protocol behind this study, based on minimally invasive, nondestructive micro-analytical techniques, was important for the characterization of fragile archaeological samples. Although most of the work done so far has been primarily qualitative, these

preliminary findings on the morphology, elemental composition, and molecular structure of the excavated samples provide invaluable information on the original materials. New information on the materials used in funerary decoration and as burial offerings was brought to light, particularly in the use of bone being crushed to form ornamental stucco (or for use as glue; Sarah Newman, personal communication, 2013) and the identification of cinnabar and exotic specular hematite unique in form and quantity. These findings suggest a direct association with elite Maya burials and funerary rituals.

Further systematic analysis of all samples with microanalytical techniques will provide more detailed chemical characterization. Other specific points that will receive further detailed study include: the mechanisms and kinetics for cinnabar degradation in Sample 03, provenance investigations to reveal the origin of the specular hematite in Samples P1 and P2, further investigations on the Hg presence in Sample 06 and identification of its lattice-shaped structure and microspheres in the context of the burial and funerary paraphernalia, and aDNA (ancient DNA) testing of the bone fragments found in Sample 09. Conclusive information regarding these specific points and an overall more comprehensive study of all samples will provide important insight into the culture and practices of this ancient civilization.



Figure IV.14. EDS spectrum of the substrate matrix of Sample 09, showing Ca-rich layer with P present, suggesting a calcium hydroxide binder which may have been mixed with crushed bone to form the white stucco.



Figure V.1. Layers of woven fabrics with yarns of varying size. Photo: Margaret Ordoñez.



Figure V.2. Layers of four different unbalanced, plain-weave fabrics. Photo: Margaret Ordoñez.





Figure V.3. Degraded fabric with penetrating holes possibly caused by sewing threads. Photo: Margaret Ordoñez.

Figure V.4. Fabric in one layer of a two-layer fragment. Photo: Margaret Ordoñez.

Appendix V Textiles Margaret T. Ordoñez

The extant textiles in Burial 9 represent an assortment that might have survived because they were layered during the burial or somehow preserved when the funerary bier collapsed. Weave structures are not always discernible because of their condition, but unbalanced plain weaves occur as might be expected from back-strap loom production. All of the identifiable spun yarns in fabrics are single, z-spun, but, contrary to common experience, 2-ply sewing threads are z-spun and Z-twisted, a less durable construction than when the spin and twist are opposite (here, lowercase "z" indicates the spin direction of singles, while uppercase "Z" denotes the direction in which singles are plied together). Deterioration of the fibers prevents identification, but some fragments contain more than one fiber or plant material. Cotton is common in Maya fabrics (e.g., Lothrop 1992:37), often mixed with fibrous plant material from stems or leaves; in addition, small thin layers of barkcloth survived.

The greatest variety of textiles occurred at the tomb's top center, near or atop Vessels 2A/2B and 3B (see Chapter 3). Yarns visible along broken edges of multilayered fabrics vary from fine to coarse. The horizontal row of small holes, just below center in Figure V.1, represents missing weft yarns in an unbalanced plain-weave fabric; the corresponding interwoven warps are smaller.



Figure V.5. Fabric on opposite side of layer in Figure VI.4. Photo: Margaret Ordoñez.



Figure V.6. Single layers of differently colored fabrics. Photo: Margaret Ordoñez.





Figure V.8. Degraded cloths with two layers of cinnabar-coated fabrics separated by an unknown material. Photo: Margaret Ordoñez.

Figure V.7. Side view of layer within the fragment in Figure VI.6. Photo: Margaret Ordoñez.



Figure V.9. Fabric layers from Sectors C6 and D6, near center of the tomb. Photo: Margaret Ordoñez.

The ~0.2 mm diameter holes indicate that the fabric had about 55 weft yarns per centimeter (y/cm). The warp yarn count would have been higher, showing that this is a finer fabric than others in the tomb.

The fragment pictured in Figure V.2 has four layered fabrics with different sized yarns and fabric counts. The uppermost vertical strip of fabric is the most tightly woven of the four, while the fabric in the next layer has a lower fabric count and is in better condition. The plain-weave structure is obvious as are the two different types of yarn. The dark-colored, large (up to 0.75 mm diameter), vertically positioned weft yarns are twisted plant material, while the smaller warp yarns appear to be cotton. The dark color could be natural coloration, deterioration, or dye. The estimated fabric count is $18-20 \times 6 \text{ y/cm}$, coarse compared to the other fabrics in the fragment. The textile on the proper right is fine, with a fabric count of 16 $\times 10 \text{ y/cm}$. Despite the disoriented weave and poor yarn condition in the fourth fabric on the other side, it appears to differ structurally from the others.

A number of fragments have large

2-ply, Z-twist yarns that are not part of a woven structure and are identified as sewing threads. The proper left side of the fabrics in Figure V.2 reveals at least two stitches, along with a loose sewing thread extending from the lower edge. Despite its construction (z-spin, Z-twist), it has held together. In addition, several fragments have holes that could be stitch holes; one in Figure V.3 even has a sewing thread exposed within such a hole. Dark material in other holes may be degraded thread.

The fabric pictured in Figure V.4 may include one or more sewing



Figure V.10. Degraded barkcloth with characteristic spacing of fibrous components. Photo: Margaret Ordoñez.

threads, but the wide variety of yarn sizes and amount of spin makes identifying plied yarns difficult and presents a confusing picture of fabric structures. Could this be a twined construction or just another plain weave? The open-work structure has interstices larger than some of the yarns, and the seven large yarns per centimeter produced a very open fabric.

This intriguing composition lay on the opposite side of the textile in Figure V.5. The face of the fabric features a system of large yarns that completely cover the opposing yarn system, if it has survived. A number of fragments contain this distinctive warp- or weft-faced fabric with hollow yarns, which could be the result of mineralization preserving the outer perimeters of yarns and not the cores.

Single layers of a fabric within a multilayer fragment are not unusual. Figure V.6 shows single layers of several fabrics in a fragment from the upper section of the tomb, but explaining how the multiple fabrics layered in this fragment came to be raises a number of questions (Figure V.7). Did someone place the many layers of different fabrics there for a utilitarian reason? As an offering or tribute? Are the holes related to sewing threads? What causes the different colors? Dyes? Coatings? Some coating or layering with cinnabar did occur as evident in Figure V.8.

Some cinnabar appears on fabrics under and to the left of the ruler's midsection and associated with Vessel 11a. A black, often crystalline, material occurs more often than cinnabar on these fabric surfaces and between layers (Figure V.9). A frequent, easily identifiable fabric from this area near the center of the tomb is similar to the one in Figure V.5, but other textiles with smaller yarns also landed there. A few thin fragments of degraded barkcloth (Figure V.10), with characteristic spacing of fibrous components, occur lower in the tomb at the man's knee level, associated with Vessel 16B. No doubt many of these fabric fragments are related to his apparel.

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