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MULTIELEMENTAL ICP-MS ANALYSIS OF CLASSICAL PERIOD ARCHAEOLOGICAL CREMATED BONE AND SEDIMENT SAMPLES FROM DÊMOSION SÊMA POLYANDRIA OF SALAMINOS 35 SITE IN KERAMEIKOS, ATHENS, GREECE

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ABSTRACT

Cremated human bone fragments excavated at the monumental burial site at Salaminos 35 Street in Kerameikos of Athens, Greece, dated to the range of the 8th decade of the 5th century BC were the subject of anthropological study. A number of bone fragments included surface stains of various discolorations. The purpose of this interdisciplinary study combining a forensic anthropology-chemistry substrate was to determine which metal(s), if any, and under what circumstances may have caused the staining on the bone surfaces. To determine this, a Collision/Reaction Cell Inductively Coupled Plasma- Mass Spectrometry (CRC ICP-MS) method with microwave accelerated sample digestion was developed and validated for the quantification of metals in bone. The concentrations of Na, Mg, Al, K, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Rb, Sr, Sn, Ba, Pb and U in the stained bone samples were compared to concentrations in unstained bone fragments and sediment samples from the same contextual archaeological association. The sample analyses and comparisons showed that 14 of the 15 stained bone samples analyzed contained significantly higher concentrations of various metals. Of the seven metals readily available during Classical antiquity (Au, Cu, Ag, Pb, Sn, Fe and Hg), four were detected at significantly high concentrations (Cu, Sn, Fe and Pb) in some samples, Ag was tested for but not detected in any sample while Au and Hg were not tested for. The metals that occurred most often at significantly high concentrations were Cu, Pb, Zn, As, Mg and Fe.

KEYWORDS: ICP-MS, bone, metal, stain, polyandria, 5th century BC

1. INTRODUCTION

In early 1997 an ancient Greek burial site was uncovered during the construction works involving a historic building in downtown Athens, Greece (Rose, 2000). The burial site included several monumental limestone chambers that proved to be polyandria (communal interments of male individuals, war dead) (Agelarakis, 2013). Relative dating techniques performed on excavated materials with an emphasis on ceramics date the burial site to between 430 and 420 BC, which may correspond to the first decade of the Peloponnesian War, the so-called Archidameian war (Agelarakis, 2013; Thucydides and Henderson, 2003). The site is located in what was known to ancient Greeks as the Dêmosion Sêma, the state cemetery of Athens (Rose, 2000; Agelarakis, 2013; Thucydides and Henderson, 2003). During the anthropological inspectional analysis preface of preparing and cataloging of the cremated skeletal remains (cremains), it was noted that some of the bone fragments had distinct stains of various discolorations on their surfaces. Stains on the same bone fragment were of one color and in some cases appeared on several areas of the same bone fragment; different bone fragments had stains of different colors (Fig. 1, 2 & 3).

The origin of the stains was unknown and there is very little data in the literature pertaining to staining of archaeological bone specimens, and particularly of cremated ones (Mann et al., 1998; Robles et al., 2002; Steckoll et al., 1971); however, since at least seven metals were purposefully mined and used in the ancient Greek world (Healy, 1978) and many colors arise from inorganic metallic compounds (Buxbaum and Pfaff, 2005), such as the greens of oxidized copper or the reds of oxidized iron, it was proposed that metals were a possible cause of the stains.

Figure 1. Upper extremity bone fragment, calcined level of cremation with no stains. (Photo by A. Agelarakis)

Figure 2. Upper extremity bone fragment, calcined level of cremation with red residues-stains. (Photo by A. Agelarakis)

Figure 3. Lower extremity bone fragment, subcalcined level of cremation with green residues-stains. (Photo by A. Agelarakis)

In order to test such a hypothesis a method capable of identifying and quantifying a large number of metals simultaneously over a wide range of concentrations was needed. The literature (Bolann et al., 2007; Zwanziger, 1989) contains quantitative and qualitative analytical methods for determining metals in a bone matrix such as atomic absorption spectroscopy (AAS) (Martinez-Garcia et al., 2005; Ericson and Smithe, 1991), inductively coupled plasma-mass spectrometry (ICP-MS) (Helliwell et al., 1996; Grattan et al., 2002), inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Yoo et al., 2002; Kniewald et al., 1994), and X-ray fluorescence (XRF) (Carvalhoa et al., 2004), but few are used to identify and quantify the content of stains found on archaeological bone samples, and fewer still are capable of simultaneous identification and quantification of multiple metals over a wide range of concentrations while using a very small amount of a non-renewable sample. ICP-MS' strength is that it has all the aforementioned capabilities.

The analytical method presented in this paper was validated for the identification and quantification of Na (sodium), Mg (magnesium), Al (aluminum), K (potassium), V (vanadium), Cr (chromium), Mn (manganese), Fe (iron), Co (cobalt), Ni (nickel), Cu (copper), Zn (zinc), Ga (gallium), As (arsenic), Se (selenium), Ag (silver), Cd (cadmium), Rb (rubidium) Sr (strontium), Sn (tin), Cs (Cesium), Ba (barium), Tl (thallium), Pb (lead) and U (uranium) in bone matrix using bone meal Standard Reference Material (SRM) 1486 purchased from the National Institute for Standards and Technology (NIST, USA) that was fortified with a certified mixed standard solution (see "Materials and Methods" section). Due to the small sample size taken from the archaeological bone samples Se, Ag, Cd, Cs, and Tl concentrations were below the quantitation limit of this assay therefore only the validation data are presented for these elements (Table 1).

There is little data available in the literature concerned with surface stains on archaeological bone but there is a lot of data, and controversy (Zapata et al., 2006; Millard, 2005; Martinez-Garcia et al., 2006; Wilson and Pollard, 2002), available about metals incorporated into bone tissue through bioaccumulation (Beard and Johnson, 2000; Stott and Evershed, 1996; Richards et al., 2001; Pyatt et al., 2005) or diagenic processes (Wilson and Pollard, 2002; Shinomiya et al., 1998; Hedges, 2002). The data presented herein will contribute to the area of forensic investigation on archaeological bone staining and add to archaeometric information regarding human cremated materials in relation to aspects of the burial customs of war dead during the Classical period in the Athenian context of ancient Greece. Data that may shed more light on the digenesis vs. bioaccumulation controversy will be presented in a later paper and will include comparison of metal concentrations in surface vs. subsurface loci and between calcined vs. sub-calcined degrees of bone thermal alteration (for the range of project specific levels of cremains' thermal alterations see Agelarakis op. cit.).

2. MATERIALS AND METHODS

The method used for bone analysis was validated by analyzing NIST bone meal Standard Reference Material (SRM) 1486 with certified values for Mg, Fe, Pb, K, Sr, Zn and non-certified values for Al, As, Cu and Mn . The SRM was used in either its original form or as a fortified material. Fortification was performed by adding varying amounts of a certified mixed standard solution (SPEX CertiPrep Cat. No. CLMS-2AN, USA) to the SRM. The original and fortified bone meal samples were suspended in an 80:20 (v:v) mixture of 4 mL concentrated trace metal grade (TMG) nitric acid (Thermo Fisher Scientific Inc. Catalog No. A509-212, USA) and 1 mL hydrogen peroxide (EMD Cat. No. 7298-4, USA) and then placed in an accelerated microwave digestion apparatus (CEM Corporation model MARS 5, USA). The digested validation samples were diluted with 20 mL of water. To determine which of the three analysis modes (normal mode with no collision/reaction gas, helium or collision mode using helium gas, and hydrogen or reaction mode using hydrogen gas) available with the CRC ICP-MS (Agilent 7500 series, Model No. G3155A, USA) was used for this study and to determine the methods limit of quantitation (LOQ) the spiked and unspiked NIST SRM samples were progressively diluted and infused into the ICP/MS along with an internal standard (SPEX CertiPrep Cat. No. CL-ISM) using an autosampler (CETAC ASX series, USA) until predetermined acceptance criteria (accuracy of 100%±20%, precision of RSD < 20%, and linearity of $R^2 \geq 0.99$) were no longer met. The analysis mode giving the lowest LOQ with acceptable accuracy, precision and linearity for each of the metals under investigation was then used for the remainder of the study. The isotopic masses monitored were those suggested by the manufacturer's literature as least likely to be interfered with (Agilent Operator's Manual). The monitored mass and mode used for each element can be found in Table 1.

The archaeological bone samples were digested as described above for the NIST samples then subjected to four 10-fold serial dilutions. Metal concentrations were calculated using a linear calibration curve ($y = mx + b$) prepared by serial dilution of the mixed standard solution and consisted of six points including zero as follows; 0.0, 0.1, 1.0, 10.0, 100.0 and 1000.0 ppb. Quality control of the method consisted of inclusion of fortified SRM samples, solvent blank samples and calibration/tuning of the instrument with each analysis using solutions and procedures recommended by the manufacturer (Agilent Operator's Manual) and the in-house quality control procedures used at the FDA's Northeast Regional Laboratory.

The method was also used but not validated for the analysis of sediment matrix (materials representing earth samples retrieved from conglomerates of the burial site and interment contexts which accompanied the cremated remains), however good quantitative agreement between multiple dilutions of samples suggests that interference caused by endogenous material in the sediments was not occurring for any of the analytes except for tin (Sn). The analysis of tin in the sediment resulted in highly variable data within and between multiple dilutions and preparations of the same samples; therefore all comparisons for tin were between bone samples (stained and unstained) only.

The focus of this study was the identification of the nature and origin of stains documented on the surface of the bone samples; therefore to ensure that none of the elements that comprised the stain were lost, no extensive cleaning or preparatory oven drying of any of the bone samples (stained and unstained) prior to sampling was done while only the smallest surface area of the bone (approximately 0.1 – 1.5 mm deep) was taken as a sample by means of mechanical scraping.

To ensure the validity of statistical comparisons the same sampling procedure used for stained bone samples was also applied to a selective sample of unstained bone fragments from the same contextual associations and to an unstained portion of the stained bone. For the preparation of the archaeological bone samples, 15 stained and 36 unstained bone samples were scraped from the bone with a glass knife, the average sample weight was 34 mg (74% RSD) for stained bone samples, 52 mg (55% RSD) for unstained bone samples and 306 mg (13 % RSD) for sediment samples. The 27 sediment samples were selected from various locations of the same archaeological contextual associations as the stained bone sample(s), yet not necessarily from an area immediately adjacent to these sample(s). The resulting bone and sediment samples followed the extraction procedure described above for the SRM sample.

A statistical analysis was performed using the concentration data obtained from the analyzed bone and sediment samples. To determine which, if any, metals were present at a significantly higher concentration in the selected samples, a single sample t-test was used to compare (at the 99% confidence interval) the concentration of each metal in each of the stains to the concentration of the same metal in the unstained bones and sediments found in the same archaeological contextual associations.

The sixteenth sample presented here was unique. Coded as number 898, it was comprised of a green material that was embedded within an endocranial bone fragment. A section of the embedded material (76.3 mg) was selectively sampled by the forensic anthropologist, and then digested as described above for the other bone samples.

3. RESULTS

The concentrations of various metals in bone samples (stained and unstained) from an ancient Greek burial site were determined using a validated ICP-MS method. The validation consisted of four original SRM samples, seven fortified SRM samples and showed acceptable accuracy (average recovery =100.9%) and precision (average %Relative Standard Deviation or %RSD=10.6%) with the exception of aluminum whose accuracy and precision were 126.1±26.3%, respectively (Table 1). Three reagent blank samples were also analyzed as described above, no metals were detected in these blank samples (blanks contained no bone material and followed the same extraction/analytical procedure as the bone samples, data not shown).

Of the 15 archeological bone stains analyzed in this study, 14 had significantly higher levels of certain metal elements than the unstained bone and sediment samples to which they were compared. The metals occurring with significantly high concentration in the bone stains, listed in order of frequency of occurrence, were Cu (copper), Pb (lead), Zn (zinc), As (arsenic), Fe (iron), Mg (magnesium), Sn (tin), K (potassium), V (vanadium), Cr (chromium), Al (aluminum), Rb (rubidium), Sr (strontium), Mn (manganese), and Ni (nickel). Results for metals found in individual stained bone samples can be found in Table 2 and are listed in order of statistical significance (i.e. "p" value) in each bone, if more than five metals occurred with high significance ($p < 0.01$) in any sample only the highest five metals are presented. Averages for the same metals found in unstained bone and sediment samples can also be found in Table 2 for comparison.

The sample coded as "898" was not considered a stain on a bone sample but rather a fraction of a foreign material, an embedded lump, within the endocranial domain of a cranial bone fragment. As an aggregation it showed very distinct boundaries that clearly superimposed the cortical layer of the endocranial surface. This sample was analyzed for identification of its integral chemical components only and was not compared to any other sample. It contained approximately 40% copper, the concentration of all other metal elements found in this sample were many orders of magnitude lower than this, individual results can be found in Table 3.

It is suggested that the green aggregate may represent in its coalesced form the mineral malachite that had smelted as a result of thermal alteration effects during pyre exposure; that would explain (as a shadow of chemical evidence) why the usual alloying elements or ore impurities of copper (tin, zinc, arsenic and lead) were not found in high concentrations. Certainly the suggested presence of malachite, having first percolated and subsequently settled to harden and solidify within a depression of the endocranial cortical bone contour and into peripherally exposed lacunae of cancellous bone provides confidently for the timing of the smelting and flocculation process of the mineral during pyre exposure; conterminous with the cleaving up of cranial compact bone surfaces that exposed cancellous components. Further, the potentiality of malachite use in the esoteric workings of the realm of burial customs and practices may conjecturally reflect in a manner particular to funerary preparations as a specter of ideational meanings in the treatment and adornment of the body of the fallen warrior, notably in the occasion of symbolically soothing and dressing with a salve a sustained fatal cranial wound by sharp injurious impact as identified through palaeopathological assessments of the anthropological study.

4. DISCUSSION

The forensic chemistry-archaeometric investigations described in this paper revealed high concentrations of various metals in stains found on the archaeological bone samples from the Athenian polyandria. This data indicates that the stains found on the archaeological bones were confidently metallic in origin. However, in most sample cases it could be difficult, strictly based on a chemical archaeometric investigation without any contributions from the analysis of the anthropological record, to determine with (overwhelming) confidence the exact time period that the metals were introduced; considering the plethora of taphonomic conditions that may have prevailed over the millennia. Hypothetically, the metals could have been introduced at any time between a *peri-mortem* juncture and the post interment period up to relatively recent historic times. Continued interdisciplinary research relative to the specific burial ground promises to yield additional data from the archaeological record, principally of the burial features, artifactual materials and offerings. These understandings in turn may better elucidate important aspects of archaeometric analyses on organic and inorganic assemblages of the site whereby a better assessment of the analytical results could be made; the aim being to hopefully better understand and interpret facets of funerary habits and rituals during the Classical period in Athens particularly regarding polyandria.

Nevertheless, the data provided by the chemical analysis offer no slender evidence that the stains contained high concentrations of metals commonly used by the ancient Greeks such as Pb (lead), Cu (copper), Fe (iron) and Sn (tin) and their major alloying or impurity elements (Potts et al., 1992; Winkel et al., 2008; Soudi et al., 1970; Pacevski et al., 2012; Sun et al., 2000; El-Taher and Alharbi, 2012; Becker et al., 1968), and that most of the cremains retrieved from the excavation were unstained. This anthropological study offers support to the thesis that at least some of the staining was caused by cultural materials and artifacts such as burial offerings. These objects may have come into contact with bone surfaces at particular junctures from pre-interment to interment (i.e. during pyre exposure, and post-pyre funerary rituals) and circumstances may have involved the contact of bone surfaces with metallic components of burial offerings, rather than the leaching of metallic elements or ionic exchange from sediment components in taphonomic circumstances. The latter may not offer support to an argument postulating that architectural elements of the monumental funerary buildings such as bronze or iron mails may have caused a prevalence of the bone staining given that in such cases the sediment samples and sediment conglomerates recovered and analyzed from the same archaeological contexts would have reflected at least a measure of such contamination. Prior to analysis it was surmised that the stains were of metallic origin, with the archaeometric results presented here there is now objective evidence that metals were involved in the staining process.

Unstained bone and sediment samples from the same archaeological contextual association, within the burial site, were included in this study due to the debate over possible diagenic changes occurring in archaeological bone samples (Zapata et al., 2006; Millard, 2005; Martinez-Garcia et al., 2006; Wilson and Pollard, 2002). The debate centers mainly on the potential flux of material (e.g. metal elements) either into or out of archaeological samples (Wilson and Pollard, 2002) and the surrounding material, and the validity of conclusions reached if diagenetic issues have not been factored into the data analysis. Therefore, the metal concentrations in the adjacent sediments and unstained bones were determined and compared to the metal concentrations of the stained bones. If the stained bone samples had significantly higher metal concentrations (as in fact was substantiated by the chemical archaeometric investigation) than both types of unstained bones (i.e. from an unstained portion of the stained bone and from separate unstained bones from the same archaeological contextual association) and sediment samples it would be unlikely that the stains had been caused by metal(s) leaching from the sediment to the bone. Hence, it was rendered as highly unlikely, if not precluded, that the prevalence of metallic in nature stains on cremain surfaces had been caused by, or disposed to, incorporation of metals on bone surfaces by bone diagenic processes in relation to sedimentological exchange. It is therefore suggested, submitting from the broader realm of forensic anthropology, that a number of requisite conditions and essential parameters must have been in effect for the causality of the metallic stains. These may be inclusive of *perimortem* wounds sustained by iron or bronze weapons' edges that penetrating into bone components could have left their physical metallic trace evidence in blunt trauma loci, in cut marks or on impacted bone components in through and through sharp force injuries. Further, a number of *post mortem* anthropogenic activities are considered associated with the domain of funerary customs and practices fitting to war dead with a focus on burial artifacts offered (Thucydides and Henderson, 2003) some of which could contain metal based components. The inclusion of unstained bones, and sediments, in the statistical comparison was also helpful in identifying the most significant differences to stained bone surfaces which, in turn, made it easier to determine what particular metal or metal alloy/impurity may have most likely caused the staining.

To ensure that none of the bone stain was lost during sample preparation no extensive cleaning of the bone was done. This sometimes led to small quantities of sediment particles being included in the final sample. The weight added to the sample by this sediment contamination (determined by weighing the undissolved material remaining after sample preparation) was corrected for in subsequent calculations but may skew the results somewhat. The small percentage of sediment traces in the sample did not account for the generally higher concentrations of metals in the unstained archaeological bone samples as compared to normal human bone (Yoo et al., 2002; Becker and Spandaro, 1968; Kwapulinski et al., 2003), and the high variability of metal concentrations in the unstained bone data as can be seen in the high % Relative Standard Deviation (%RSD) shown in Table 2. The data variability in unstained bone may be due in part to multifactorial parameters which include, but may not be limited to: a) the suspected diversity of burial artifacts offered to the individual fallen warrior represented (Thucydides and Henderson, 2003); b) the anatomic locus, particular morphologic anatomy and histologic composition of the subperiosteal surface of the compact bone fragments in question; c) the particular placement of the warrior's body in relation to the potency of the pyre effects and the microenvironment association of each skeletal component in spatial relations to metal based artifacts, as well as the offering of burial goods inclusive of metal components after the placement of cremains within the funerary urns/cypress coffins (Thucydides and Henderson, 2003; Kwapulinski et al., 2003) and d) the differences in bone histology changes afforded by non-isothermic exposure during the cremation process which led to a discrepancy in matters of bone preservation available for forensic anthropologic study.

Archaeometric data which may contribute valuable information to the controversial subject of diagenic changes in cremated bones will be presented in a later publication that will include comparisons of metal concentrations of calcined vs. sub-calcined bones, periosteal compact bone surface vs. sub-surface bone and metal correlations between bone types and sediments.

5. CONCLUSIONS

The archaeometric analysis conducted by means of implementing the ICP-MS analytical method confidently provided qualitative and quantitative evidence on the chemical nature of a representative sample of cremain surfaces' stains. It positively identified the nature/kind of their elemental metallic consistency, lending support to the proposed concept that direct exposure of the anthropological remains with metallic objects both at *perimortem* and/or peri-interment contexts were the causative agents of the stains rather than random diagenic circumstances of taphonomic nature. It thus appears that what were visually perceived during the inspectional stage of forensic anthropologic study as distinct discolorations in the form of stains on cremain surfaces were determined to be, through chemical archaeometric analysis, residues of a metallic nature. These, based on their elemental nature and physical and chemical processes that pertained (such as calcination/oxidation, and the potential of having reached their thresholds of phase transition and thermal decomposition) due to the high temperatures attained during pyre exposure and of subsequent long term taphonomic oxidation processes showed a variety of discoloration hues and values (see available examples in table 4).

In further correlating the chemical archaeometric results with data derived from the forensic anthropologic analysis additional details may be elucidated (in reasonable confidence) concerning the relative interval at which juncture the metallic elements accumulated as filament clusters on cremain surfaces. A number of these preserved through the complexities of post-pyre funerary customs' handling and interment conditions, the potential of decisive, historically documented, anthropogenic impacts afforded on the Kerameikos area and the region of the burial monuments namely blatantly desecrated by Philip V during the second Macedonian war (Diodorus and Goold, 1999) and brazenly looted by Sulla during the siege of Athens (Plutarch et al., 2000), the taphonomic weathering of millennia, the archaeological excavation and recovery processes, as well as the stages that preceded the anthropological analysis whereby they were identified. Further, a good number of cremains it is suggested, although having appeared through inspectional analysis unstained by distinct hues of discoloration as observed with the stained cremains could and would have been exposed to traces of the same metallic compounds by spatial nearness to the same circumstances and processes explained above; hence the variability of metal concentrations among the unstained bone data (see Table 2) revealed by the chemical archaeometric investigation.

The fact (as derived through the laboratory analysis of the anthropological record) that the metallic stains/residues on cremain surfaces were superimposed by layers of bone dust and ashes of old, and in some cases with the added component of sediment particles in conglomerate form yet void of any discernible metallic stains/residues implicitly provides for a *terminus ante quem*, predating the interval of the accumulation and settling of the metallic stains/residues (in the form of traces, films and filaments) on cremain surfaces subsequently to be superimposed by bone dust and ash residues as well as delicate webs of sediment particles occasionally in conglomerate form.

Hence, broadly restricting, if not rendering implausible the competing explanatory causality of the metallic stains on cremains by diachronic diagenic in nature taphonomic circumstances, it can be deduced with considerable confidence that their cause and effect may be delved into *perimortem* conditions whereby deeply penetrating thrusting weapons' edges and projectiles embedded in visceral and endosteal domains had left residual fragments or that such embedded weapons' components could not/had not been extracted in their entirety from the bodies of the warriors, and/or in funerary practices both in regards to the private family considerations and to duties of the State fitting to the repatriated war dead (Thucydides and Henderson, 2003). Namely, emphasis is placed in the adornment of the fallen, placed at the pyre, dressed with symbolic in nature decorations and functional accessories with versions of body armor and weapons' attires (comprising metal elements) to accompany the newly departed to the meadows of asphodels, and any number of additional discrete offerings that were to be presented to the collected cremains of individual warriors in funerary vases by family members and the public alike during the three days of the public *prothesis* before *ekfora* and burial to polyandria according to the Athenian burial custom of their fathers (*patrios nomos*) (Thucydides and Henderson, 2003).

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Table 1 – Method Validation and Within-Study Quality Control Sample Results

Element	Nominal Conc. ppm*	Mass	Tune Mode	LOQ** (ppb)	Spiked NIST SRM Used For Validation			Spiked NIST SRM Used For Batch QC			Unspiked NIST SRM Used For Validation			Combined Total		
					Recovery (%)	n	SD	Recovery (%)	n	SD	Recovery (%)	n	SD	Recovery (%)	n	SD
Na	5050.3	23	hydrogen	10	108.5	16	8.0	114.5	10	13.5	108.2	44	12.7	109.2	70	11.9
Mg	4710.3	24	hydrogen	10	103.7	16	4.1	97.4	10	9.5	88	45	8.2	92.9	71	10.1
Al	51.3	27	normal	10	120.4	6	15.0	134.6	4	39.2	BQL			126.1	10	26.3
K	462.3	39	hydrogen	10	113.5	10	12.4	101.8	10	8.9	N/A			98.4	50	12.8
V	50.3	51	hydrogen	0.1	99.9	15	3.7	101.4	7	8.5	92.2	30	9.0	100.4	22	5.5
Cr	50.3	52	helium	1	100.7	15	5.4	95.8	8	9.5	N/A			99	23	7.3
Mn	51.3	55	hydrogen	0.1	97.8	16	6.7	105.1	10	15.4		N/		107.2	50	14
Fe	149.3	56	hydrogen	4	84.9	12	5.9	95.7	9	8.2	A N/A			90.2	36	10.3
Co	50.3	59	helium	0.1	98.2	16	6.3	98.6	10	13		N/		98.3	26	9.2
Ni	52	60	helium	0.1	94.2	16	8.7	92.3	10	15.6	A			93.4	26	11.6
Cu	51.1	63	normal	1	102.1	13	4.2	89.4	10	11.8	114.3	24	13.5	96.6	23	10.4
Zn	197.3	66	helium	5	95.6	12	2.1	86.1	10	11.3	91.1	15	12.4	87.4	52	12.4
Ga	63.2	69	hydrogen	0.2	104.8	16	5.6	98.4	10	13.4	N/A	N/A		102.4	26	9.7
As	50.3	75	helium	1	102.7	12	8.7	99.6	10	12.2	N/A	N/A		101.3	22	10.3
Se	50.4	78	helium	10	100	9	17.1	99.4	10	15.6	BQL	45		99.7	19	15.9
Rb	50.6	85	helium	0.1	104.2	16	6.4	102.8	10	12.6	84.6	30	13.9	103.7	26	9
Sr	314.3	88	normal	0.8	100.4	16	3.3	95.1	10	10.6		N/A		100.9	71	8.4
Ag	50.3	107	helium	1	103.1	12	3.4	108.8	4	8.4	N/A	BQL		104.5	16	5.4
Cd	50.3	111	hydrogen	0.1	102.7	16	6.7	99.6	10	7.9				101.5	26	7.2
Sn	50.3	118	helium	0.3	ND	ND	ND	100.5	10	16.1	N/A	N/A		100.5	10	16.1
Cs	50.3	133	helium	0.1	108.8	16	9.2	100.5	10	9.1		N/		105.6	26	9.9
Ba	361.7	137	helium	0.8	95.5	8	4.8	93.7	10	4.8	A	N/A		94.5	18	4.7
Tl	50.3	205	helium	0.1	106.8	16	4.6	101	9	9.7		N/		104.7	25	7.2
Pb	51.6	208	helium	0.1	102.2	16	9.1	96.1	10	8.9		A		98.8	39	10.1
U	50.3	238	normal	0.1	107.2	16	5.7	102.1	10	11.8	102.3	45	8.8	105.2	26	8.7

* = This value is for the spiked samples, for unspiked samples subtract 50.3 ppm

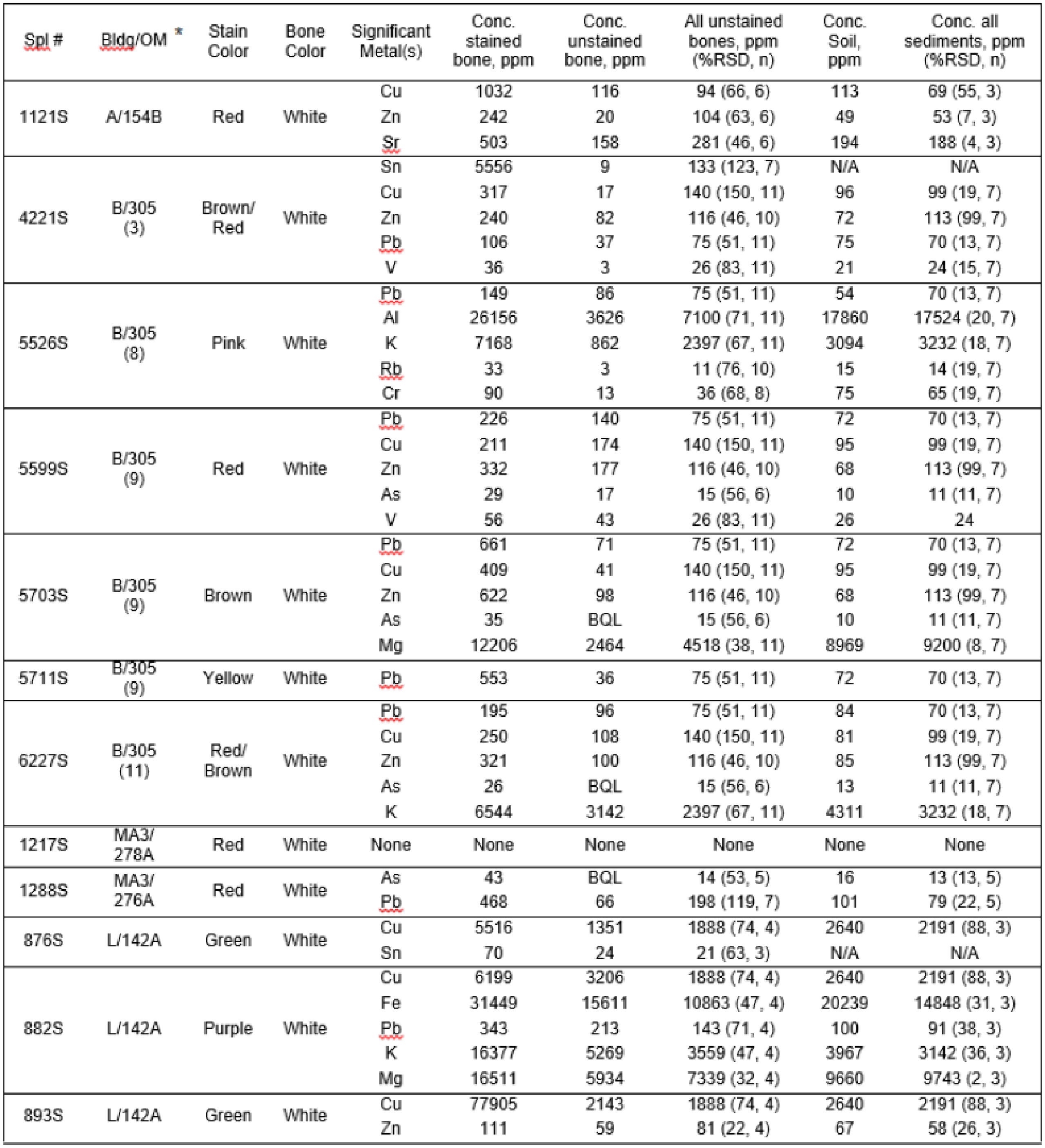
** = This is the LOQ of the analytical solution infused into the ICP-MS

BQL = Below Quantitation Limit

ND = Not Determined

N/A = Not Available

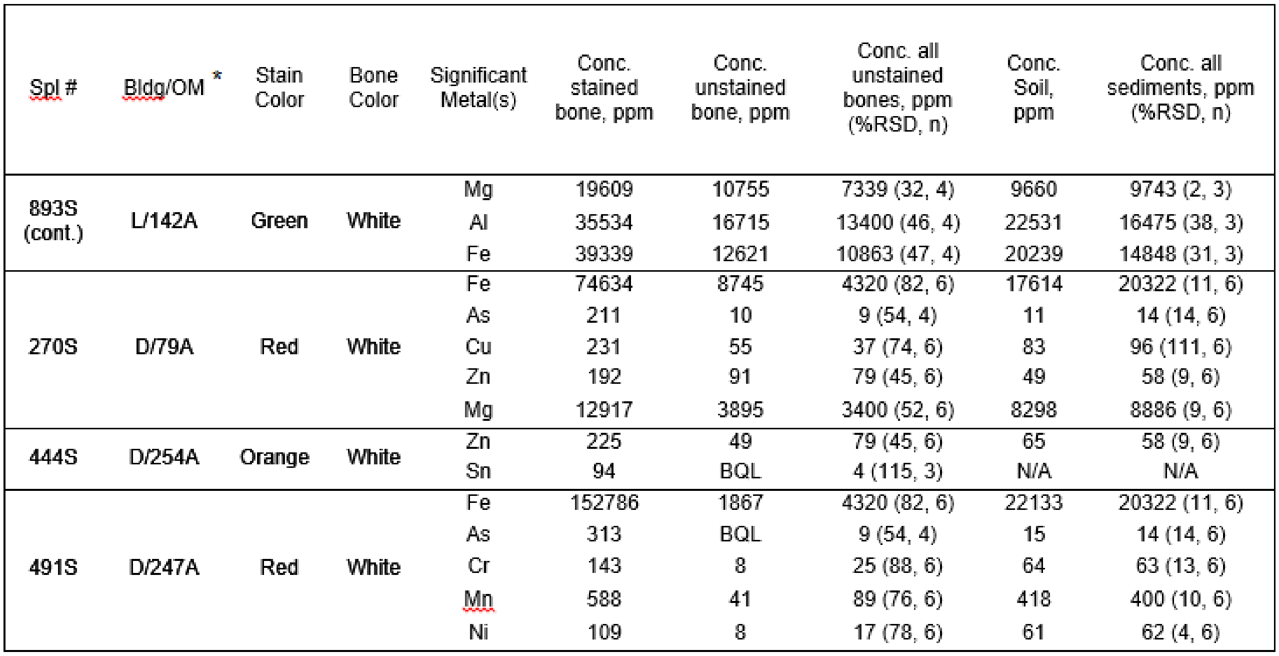
Table 2 - Metal concentrations (ppm) in bone and surrounding sediment



Spl #	Bldg/OM *	Stain Color	Bone Color	Significant Metal(s)	Conc. stained bone, ppm	Conc. unstained bone, ppm	All unstained bones, ppm (%RSD, n)	Conc. Soil, ppm	Conc. all sediments, ppm (%RSD, n)
1121S	A/154B	Red	White	Cu	1032	116	94 (66, 6)	113	69 (55, 3)
				Zn	242	20	104 (63, 6)	49	53 (7, 3)
				Sr	503	158	281 (46, 6)	194	188 (4, 3)
4221S	B/305 (3)	Brown/ Red	White	Sn	5556	9	133 (123, 7)	N/A	N/A
				Cu	317	17	140 (150, 11)	96	99 (19, 7)
				Zn	240	82	116 (46, 10)	72	113 (99, 7)
				Pb	106	37	75 (51, 11)	75	70 (13, 7)
				V	36	3	26 (83, 11)	21	24 (15, 7)
5526S	B/305 (8)	Pink	White	Pb	149	86	75 (51, 11)	54	70 (13, 7)
				Al	26156	3626	7100 (71, 11)	17860	17524 (20, 7)
				K	7168	862	2397 (67, 11)	3094	3232 (18, 7)
				Rb	33	3	11 (76, 10)	15	14 (19, 7)
				Cr	90	13	36 (68, 8)	75	65 (19, 7)
5599S	B/305 (9)	Red	White	Pb	226	140	75 (51, 11)	72	70 (13, 7)
				Cu	211	174	140 (150, 11)	95	99 (19, 7)
				Zn	332	177	116 (46, 10)	68	113 (99, 7)
				As	29	17	15 (56, 6)	10	11 (11, 7)
				V	56	43	26 (83, 11)	26	24
5703S	B/305 (9)	Brown	White	Pb	661	71	75 (51, 11)	72	70 (13, 7)
				Cu	409	41	140 (150, 11)	95	99 (19, 7)
				Zn	622	98	116 (46, 10)	68	113 (99, 7)
				As	35	BQL	15 (56, 6)	10	11 (11, 7)
				Mg	12206	2464	4518 (38, 11)	8969	9200 (8, 7)
5711S	B/305 (9)	Yellow	White	Pb	553	36	75 (51, 11)	72	70 (13, 7)
6227S	B/305 (11)	Red/ Brown	White	Pb	195	96	75 (51, 11)	84	70 (13, 7)
				Cu	250	108	140 (150, 11)	81	99 (19, 7)
				Zn	321	100	116 (46, 10)	85	113 (99, 7)
				As	26	BQL	15 (56, 6)	13	11 (11, 7)
				K	6544	3142	2397 (67, 11)	4311	3232 (18, 7)
1217S	MA3/ 278A	Red	White	None	None	None	None	None	None
1288S	MA3/ 276A	Red	White	As	43	BQL	14 (53, 5)	16	13 (13, 5)
				Pb	468	66	198 (119, 7)	101	79 (22, 5)
876S	L/142A	Green	White	Cu	5516	1351	1888 (74, 4)	2640	2191 (88, 3)
				Sn	70	24	21 (63, 3)	N/A	N/A
882S	L/142A	Purple	White	Cu	6199	3206	1888 (74, 4)	2640	2191 (88, 3)
				Fe	31449	15611	10863 (47, 4)	20239	14848 (31, 3)
				Pb	343	213	143 (71, 4)	100	91 (38, 3)
				K	16377	5269	3559 (47, 4)	3967	3142 (36, 3)
				Mg	16511	5934	7339 (32, 4)	9660	9743 (2, 3)
893S	L/142A	Green	White	Cu	77905	2143	1888 (74, 4)	2640	2191 (88, 3)
				Zn	111	59	81 (22, 4)	67	58 (26, 3)

* OM = OMAS, a specific area within the building

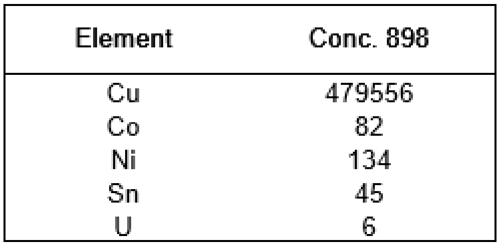
Table 2 con't Metal concentrations (ppm) in bone and surrounding sediment



Spl #	Bldg/OM *	Stain Color	Bone Color	Significant Metal(s)	Conc. stained bone, ppm	Conc. unstained bone, ppm	Conc. all unstained bones, ppm (%RSD, n)	Conc. Soil, ppm	Conc. all sediments, ppm (%RSD, n)
893S (cont.)	L/142A	Green	White	Mg	19609	10755	7339 (32, 4)	9660	9743 (2, 3)
				Al	35534	16715	13400 (46, 4)	22531	16475 (38, 3)
				Fe	39339	12621	10863 (47, 4)	20239	14848 (31, 3)
270S	D/79A	Red	White	Fe	74634	8745	4320 (82, 6)	17614	20322 (11, 6)
				As	211	10	9 (54, 4)	11	14 (14, 6)
				Cu	231	55	37 (74, 6)	83	96 (111, 6)
				Zn	192	91	79 (45, 6)	49	58 (9, 6)
				Mg	12917	3895	3400 (52, 6)	8298	8886 (9, 6)
444S	D/254A	Orange	White	Zn	225	49	79 (45, 6)	65	58 (9, 6)
				Sn	94	BQL	4 (115, 3)	N/A	N/A
491S	D/247A	Red	White	Fe	152786	1867	4320 (82, 6)	22133	20322 (11, 6)
				As	313	BQL	9 (54, 4)	15	14 (14, 6)
				Cr	143	8	25 (88, 6)	64	63 (13, 6)
				Mn	588	41	89 (76, 6)	418	400 (10, 6)
				Ni	109	8	17 (78, 6)	61	62 (4, 6)

* OM = OMAS, a specific area within the building

Table 3 – Metal concentrations (ppm) in sample coded number 898 imbedded in bone.



Element	Conc. 898
Cu	479556
Co	82
Ni	134
Sn	45
U	6

Table 4 – Example of cremain stain Munsell color readings , Funerary Building B

Stains on Cremain Surfaces: Chroma-Hue Identifications Based on Munsell Readings / Combined Funerary Building "B" Stratigraphic Contexts								
No.	Stain: Munsell Chroma-Hue Reading	No.	No.	Stain: Munsell Chroma-Hue Read-	No. IDs	No.	Stain: Munsell Chroma-Hue Reading	No. IDs
1	Black 2.5YR 2.5/0; 5YR 2.5/1; 7.5YR 2/0; 5Y	1	42	Iron oxide/Yellow 10YR 3/3.5 -	1	83	Dark reddish 2.5YR 3/6	3
2	Blue 5B 7/1	1	43	Iron oxide reddish 10YR 3.5/6	3	84	Dark reddish iron 2.5YR 3/2	1
3	Blue green cupric acid 5B-5G 7/1.5	2	44	Orange 5YR 7/7; 7.5 YR 7/7	74	85	Slight reddish 10R 6/8	8
4	Bluish green 5BG 6/1	2	45	Dark orange 5YR 6/8	1	86	Reddish/brown/green 2.5YR 5/2	1
5	Bluish orange 5.2YR 6/8; 5B 7/1	1	46	Light orange 5YR 7/8	1	87	Reddish brown 2.5YR 5/4	24
6	Bluish red 10R 5/2	1	47	Pale orange 7.5 YR 7/8	7	88	Slight reddish brown 2.5YR 6/4	1
7	Brown 7.5YR 5/4	18	48	Slight orange 7.5 YR 7/7	11	89	Reddish orange 5YR 7/8	27
8	Dark brown 7.5YR 3/2	3	49	Pale orange and pink 7.5 YR 8/4	1	90	Dark reddish orange 2.5YR 4/8	1
9	Pale brown 7.5YR 6/2	2	50	Orange and blue 5B 7/1; 7.5 YR	1	91	Slight reddish orange 5YR 7/6	1
10	Slight brown 7.5YR 6/4	3	51	Orange and purple 7.5YR 8/6; 10R	1	92	Reddish pink 5YR 6/4	1
11	Pale brown/Orange 2.5YR 6/4	1	52	Orange/Black 7.5YR 3/4	1	93	Yellow 2.5Y 8/8	72
12	Brown orange 5YR 6/6	3	53	Dark orange/brown 7.4YR 4/4	1	94	Dark yellow 10YR 7/8	4
13	Brown red 2.5YR 5/4	1	54	Orange brown 7.5YR 5/6	26	95	Pale yellow 2.5Y 8/4	9
14	Brown rust color 5YR 4/6	1	55	Dark orange brown 7.5YR 4/4	1	96	Pale yellow and green 10YR 8/1.5	1
15	Rust color brown 5YR 4/6	1	56	Slight orange brown 7.5YR 6/6	4	97	Slight yellow 10YR 8/3	5
16	Brownish 7.5YR 5/4	11	57	Orange brownish 7.5YR 6/8	1	98	Slight pale yellow 10YR 8/3.5	3
17	Light brownish 5YR 6/4	2	58	Orange red 5YR 5/8	10	99	Yellow/Orange/Green 2.5Y 7/6	1
18	Slight brownish 5YR 6/3.5	1	59	Slight orange red 5YR 6/8	2	100	Yellow/Pale green 2.5Y 7/4	1
19	Brownish orange 2.5YR 5/8	14	60	Orange reddish 5YR 6/8	1	101	Yellow/Slight greenish 2.5Y 7/8	1
20	Brownish red 2.5YR 4/4	6	61	Orange/Reddish/Pink/Iron (Fe)	1	102	Yellow/Yellowish orange 10YR 6/8	1
21	Slight brownish red 2.5YR 5/4	2	62	Orange yellow 7.5YR 7/8	10	103	Yellow and orange 7.5YR 7/8	1
22	Brownish yellow 7.5YR 6/6	2	63	Orange/Yellow/Brown 7.5YR 6/8	1	104	Pale yellow and orange 10YR 7/5	1
23	Cupric acid 5G 6.5/2-5.5/2	6	64	Orange/Yellow/Brownish 7.5YR	1	105	Yellow brown 10YR 6/6	6
24	Slight cupric acid 5GY 6.5/1	1	65	Oragnish 5YR 7/8	36	106	Yellow green 2.5Y 6/8	3
25	Gray N/4; 7.5YR 6/0; 2.5YR 5.5/0	1	66	Sligh orangish 7.5YR 7/8	2	107	Yellow green/brownish red 2.5Y 4/4	1
26	Gray black 2.5YR N3/; 7.5YR 3/0; 2.5YR	1	67	Dark orangish/Reddish 7.5YR 5/8	1	108	Yellow greenish 5Y 7/6	1
27	Green 5G 6/2; 5Y 4/3.5	6	68	Orangish brown 2.5YR 5/8	3	109	Yellow orange 10YR 6/8	28
28	Mild green 5G 6/2	1	69	Orangish red 2.5YR 4/8	2	110	Slight yellow orange 10YR 6/6	1
29	Pale green 5G 7/2; 5Y 7/2.5	6	70	Orangish yellow 7.5YR 7/8	1	111	Yellow orangish 10YR 7/8	1
30	Green and blue 5BG 6/1	2	71	Slight pale pink 5YR 8/3	1	112	Yellow red 5YR 5/6	1
31	Green and orange 5G 7/2; 5YR 7/8	1	72	Pinkish 5YR 8/3.5	5	113	Yellow red iron oxide 5YR 5/4	1
32	Green/Brown/Orange 5G 4.5/2; 5YR 3/3;	1	73	Slight pinkish 5YR 8/2	1	114	Yellowish 2.5Y 8/6	31
33	Green blue 5BG 6/1	1	74	Pinkish orange 5YR 7/5	2	115	Pale yellowish 2.5Y 7/4	5
34	Green bluish 5G 6/1	1	75	Red 10R 4/7	43	116	Yellowish brownish 10YR 5/5	1
35	Greenish 5GY 7/1	4	76	Dark red 10R 3/6	1	117	Yellowish greenish 5Y 7/8	2
36	Pale greenish 5G 7/1; 5Y 6/7	2	77	Slight red 10R 5/4	1	118	Yellowish orange 10YR 7/7	13
37	Slight pale greenish 5GY 7/1	1	78	Red/orange/yellow 5YR 6/6	1	119	Light yellowish orange 10YR 8/6	1
38	Greenish yellow 5Y 7/3	2	79	Red brown 2.5YR 4/4	2	120	Slight yellowish orange 10YR 8/5	1
39	Iron oxide 10YR 3/3.5; 2.5YR 2.75/4	15	80	Dark red/orange/yellow 5YR 5/8	1	121	Yellowish orange and red 10YR 5/7	1
40	Iron oxide/Black 2.5YR 2.5/1	1	81	Red orange 5YR 5/6	2	122	Yellowish red 5YR 5/6	1
41	Iron oxide/Orange 2.5YR 4/8; 10YR 4.5/8	1	82	Reddish 2.5YR 4/8	82	123	N/A	51