EVALUATION OF PAINTED POTTERY FROM THE MESA VERDE REGION USING LASER ABLATION-INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (LA-ICP-MS)

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For decades archaeologists have struggled with the problem of accurately determining organic and mineral-based paints in pottery from the American Southwest. Using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS), we have developed a simple and cost-effective method that permits classification of painted surfaces into mineral and organic-based categories. By applying this method to Mesa Verde and Mancos Black-on-white pottery from the Mesa Verde Region, we were able to distinguish easily between mineral and organic-based paints. Preliminary data also suggest that multiple sub-groups of mineral-based paints exist within these ceramic types, indicating that multiple recipes for manufacturing paint may have been employed by prehistoric potters from this region.

Por varias décadas, los arqueólogos se han enfrentado al problema de distinguir con precisión entre pigmentos orgánicos y pigmentos de base mineral en la cerámica del suroeste norteamericano. Utilizando LA-ICP-MS hemos desarrollado una técnica simple y de bajo costo que permite la clasificación de superficies pintadas en categorías de pigmentos minerales y pigmentos orgánicos. Al aplicar este método a la cerámica Mesa Verde y Mancos Negro-sobre-blanco del área Mesa Verde, hemos distinguido fácilmente entre pigmentos minerales y pigmentos orgánicos. Los resultados preliminares sugieren que existen varios sub-grupos de pinturas minerales en dichos tipos cerámicos, lo cual indica que los alfareros prehistóricos de esta región utilizaron varias fórmulas para fabricar pigmentos.

And just as chemistry was used, although unwittingly, in the manufacture, decoration, and firing of pottery, so through chemistry can we find something of the details of its manufacture and the extent of influence of the people who made it [Hawley 1931:36].

Archaeologists working in the Southwestern United States have long recognized that black paint used to decorate pottery varies in composition both through time and through space. Florence Hawley (1929), for instance, developed a chronological framework for the greater Southwest that was based largely on the proportion of smudged, carbon, and mineral categories. Hawley also proposed the use of simple physical and chemical tests to discriminate the carbon- and mineral-painted variants. Anna Shepard (1939) used changes in paint composition to hypothesize about cultural boundaries and interactions in the La Plata district of the Northern San Juan region. Archaeologists today, like their predecessors, continue to use mineral- and organic-paint categories to examine broad spatial and temporal trends regarding use of pigments and to explore the relationships between prehistoric groups (e.g., Wilson 1996).

Classification of paints into mineral and organic-based categories tends to be subjective, and the categories may not be homogeneous. In the first case, if the use of subjective criteria leads to significant errors in classification, inferences regarding prehistoric interaction patterns or temporal trends in paint use may also be erroneous. In the second case, if the use of a catchall mineral category masks significant compositional variation in mineral pigments, then potentially important details of technology, production organization, and interaction patterns will remain hidden from archaeological view.

In a recent article, Stewart and Adams (1999) evaluate the visual criteria used by southwestern archaeologists for classification of black-on-white

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painted pottery into carbon- and mineral-based paint categories. In an attempt to evaluate the accuracy of visual categorization, they asked 19 participants of varying archaeological skill levels and expertise to categorize the paint type for 15 black-on-white sherds from the Mesa Verde region. The results of this study were then compared to objective data obtained from SEM-EDS analysis (scanning electron microscope-energy dispersive X-ray spectrometer). Stewart and Adams found an overall error of 15.8 percent exists for paints classified using visual criteria, and for paints difficult to classify (e.g., mineral paints that have soaked into the surface of the sherd and thus appear to be carbon based) the error can be as high as 57.9 percent. In addition, they found that the use of a mixed-paint category by archaeologists (paint containing both carbon and mineral) may introduce additional error to paint categorization.

Numerous studies have demonstrated the utility of using SEM analysis in chemistry-based ceramic provenance research (e.g., Neff et al. 1999; Neff et al. 2001; Rautman et al. 1999). However, we believe that laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS), another technique that has only become available recently, may be more suitable for analysis of paints. We emphasize that we are not attempting to portray SEM analysis in a negative light. SEM analysis will continue to be a powerful analytic tool for imaging artifacts and determining chemical composition of constituents at a micro level. LA-ICP-MS does, however, have several advantages over SEM for distinguishing mineral from organic paints. First, sample preparation for SEM can be destructive to the artifact and somewhat cumbersome since a typical quantitative analysis requires the sample be impregnated in epoxy, cut, polished, and carbon-coated. Second, analysis of carbon-based paint surfaces is difficult because paints are generally analyzed in cross-section and the carbon layer may not be visible (Stewart and Adams 1999: Figure 5). Third, trace elements, which include several transition metals likely to be present in ores used for pigment, are impossible to measure using SEM-EDS. Finally, while the cost of SEM-EDS is not as high as some analytic techniques, the rapidity with which samples can be prepared and analyzed using LA-ICP-MS will make this technique equally or more cost effective.[1]

ICP-MS is a relatively new tool for archaeological provenance studies (e.g., Gratuze 1999; Gratuze

et al. 2001; Kennett et al. 2001; Mallory-Greenough et al. 1998; Tykot 1998). In LA-ICP-MS, a laser is used to ablate a small area on the surface of a sample. Usually the area ablated is smaller than 1,000-x-1,000 microns and less than 30 microns deep. The ablated material is then transported from the laser cell and introduced into the ICP-MS torch, where an argon gas plasma capable of sustaining electron temperatures between 8,000 and 10,000 K is used to ionize the injected sample. The resulting ions are then accelerated by high voltage and passed through a series of focus lenses, an electrostatic analyzer, and finally a magnet. By varying the strength of the magnet, ions are separated according to mass and charge and passed through a slit into the detector, which records only a very small atomic mass range at a given time. By varying the instrument settings, the entire mass range can be scanned within a short period of time. ICP-MS can provide compositional data for 50–60 elements, including rare earth elements.

The Archaeometry Laboratory at the University of Missouri Research Reactor recently acquired a high-precision Thermo Elemental Axiom magnetic-sector inductively coupled plasma mass spectrometer to be used in conjunction with instrumental neutron activation analysis (INAA) and provenance studies. The ICP-MS is coupled to a Merchantek Nd:YAG 213-nanometer wavelength laser ablation unit, which permits the introduction of solid samples into the ICP-MS. The laser can be targeted on spots as small as five microns in diameter. With this small spot size and the high sensitivity of magnetic sector ICP-MS to a wide range of major, minor, and trace elements, LA-ICP-MS is a very powerful microprobe. Moreover, it is virtually nondestructive, considering the ablated areas are often indistinguishable with the naked eye.[2]

Based on a multitechnique study of obsidian sources, LA-ICP-MS compares favorably with digestion inductively coupled plasma-atomic emission spectometry (ICP-AES) and ICP-MS, X-ray flourescence (XRF), and INAA in terms of sensitivity, precision, and accuracy (Glascock 1999; Gratuze et al. 2001). Experience with the Axiom at Missouri University Research Reactor (MURR) indicates that sensitivity for most elements is in the low parts-per-million range, although some elements can be determined in the upper parts-per-trillion range. Precision for most elements is very good, with a coefficient of variation usually less than 10 percent

over three scans. Accuracy is dependent upon the method used for normalization of the data and the standards selected to transform the data. Results reported by Neff (2002a) for Ohio Red clay, a quality control standard found to be homogeneous in INAA, indicate that most LA-ICP-MS values are between .75–1.25 times the INAA value.

In our study, we analyzed paints on 37 black-on-white pottery sherds and four replicates from the Four Corners region of the American Southwest using LA-ICP-MS. The sample consisted of sherds from the MURR Archaeometry Laboratory archival collection that had previously been analyzed using INAA for projects initiated by Donna Glowacki (Glowacki et al. 1995; Glowacki et al. 2002), Alan Reed (Neff et al. 2000), and W. James Judge (Glowacki et al. 2002). The sherds were classified either as Mesa Verde Black-on-white (carbon painted) or Mancos Black-on-white (mineral painted).[3] Each sherd analyzed contained both a painted and an unpainted area.

Sample preparation for LA-ICP-MS is relatively easy. Each sample is washed in deionized water and permitted to dry prior to analysis. The laser ablation sample chamber is 2-x-5 cm and readily holds between four and 12 samples at a time. Samples too large to fit in the sample chamber were broken or cut to a smaller size for analysis.

Painted and unpainted portions of each sherd were analyzed using a 100 micron laser spot size, 50 percent power, and a repetition rate of 20 shots per second. Twenty-four of the samples and the four replicates were analyzed for Sc, Ti, V, Cr, Mn, Fe, Ni, Co, Zn, and Pb. Data from this experiment suggested the compositional variation in paint was greater than anticipated. An additional 14 samples were therefore analyzed for the original suite of elements plus Mg, Al, Cu, Sr, Ag, Sn, Ba, Au, and U to determine if additional compositional groups were present.

A basic problem in LA-ICP-MS is that it is difficult to monitor the amount of material removed by the laser and transported to the ICP. Conditions such as hardness of the material, position of the sample in the laser chamber, whether or not the surface of the artifact is flat, and other conditions affect how much material reaches the ICP torch and, thus, the intensity of the signal monitored for the various atomic masses of interest. In addition, instrumental drift in the ICP-MS over several hours or days affects count rates. All of these factors affect how data are normalized and standardized. The data collected for this project had reasonably good precision, but at this early stage in our research, we decided not to transform the data into element abundance data. Instead, our data are expressed as ratios between count rates obtained on the painted area of the sherd and data obtained from an unpainted area of the same sherd. We have since developed a standardization method that yields reasonably accurate elemental abundance data.

The use of ratios assumes that the unpainted (slipped) area of the pottery sample does not significantly vary between samples.[4] Thus, the ratio value for paint can be subjected to a quantitative analysis similar to that used for elemental abundance data (e.g., Neff 2002b). Comparing these data to data obtained from ceramics produced outside the Mesa Verde region may not be valid since clays from outside the region may be significantly different in their composition. However, our purpose in this case is to evaluate the feasibility of the method. INAA studies of pottery from the Mesa Verde region indicate that chemical composition of Mesa Verde and Mancos Black-on-white does not vary at levels that would impact the paint classifications we have made (Glowacki et al. 1995; Glowacki et al. 2002).

We conducted one experiment designed to demonstrate the distinctive chemical signature obtained from mineral painted surfaces. The surfaces of two samples, ADR007 and WJJ008, were mapped using LA-ICP-MS (Figure 1). We placed an arbitrary grid over each sample and collected data for Mn and V at 100 micron intervals. These transition metals were selected for analysis because they typically occur in iron-based minerals. Data for each element at each location were expressed as a ratio of the lowest count obtained for that element on that specimen. Thus, the lowest value for each specimen is 1.0. These data were then converted into a contour map with Surfer (Golden Software). The value of 1.0 was set to correspond to the color white and high values were set to equal the color black. Color differences in this figure show the non-uniform distribution of V and Mn in these paints in a manner similar to the backscattered electron images Stewart and Adams (1999, Figure 6) use to depict the nonuniform distribution of Fe in a sample decorated with mineral-based paint. ADR007, classified as Mancos Black-on-white, clearly has manganese and
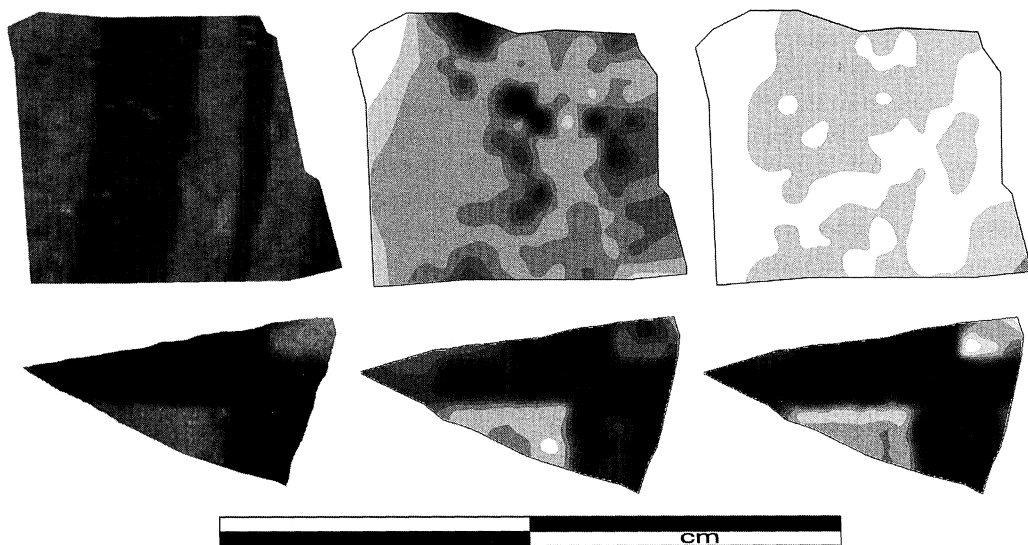
Figure 1. Left: Digital photograph of ADR007 and WJJ008. Center: LA-ICP-MS derived map depicting manganese concentrations on the surfaces of ADR007 and WJJ008. Right: LA-ICP-MS derived map depicting vanadium concentrations on the surfaces of ADR007 and WJJ008. Darker areas on the elemental maps indicate higher elemental concentrations.
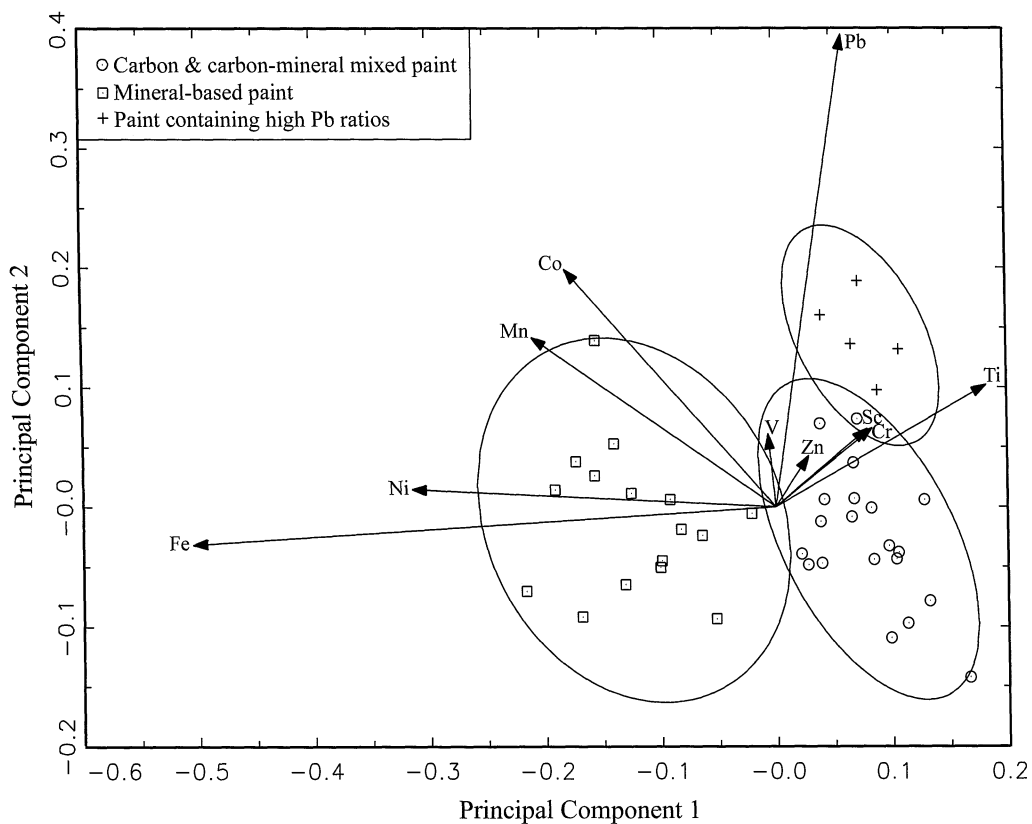

Figure 2. Principal components biplot showing the coordinates for the variables (elements) with the various paint groups. Ellipses represent 90% confidence levels for group membership.

Table 1. Paint Classification and Ratio Values for Analyzed Sherds.

Anid	Paint Type	Mg	Al	Sc	Ti	V	Cr	Mn	Fe	Ni	Co	Cu	Zn	Sr	Ag	Sn	Ba	Au	Pb	U
ADR003	M			.65	.46	.80	.58	1.92	7.68	2.04	2.51		.72						1.00	
ADR005	C			.90	1.16	1.11	1.41	2.45	1.29	1.32	1.82		.89						1.75	
ADR007	C/Fe			1.04	.91	.97	1.43	1.68	3.16	1.16	1.01		1.27						1.70	
ADR008	M			.92	.69	.74	.75	1.23	8.09	3.17	1.27		1.59						.35	
DMG247	Pb			1.15	1.13	.92	.58	2.03	1.76	1.08	1.79		.45						15.24	
DMG247[a]	Pb			1.70	1.75	1.18	1.24	2.03	1.23	1.08	1.21		.73						11.19	
DMG247[a]	Pb			1.54	2.22	1.09	1.93	5.10	1.40	1.15	3.14		1.21						12.13	
DMG250	C			1.04	1.20	1.03	2.93	2.74	1.10	1.03	1.95		1.92						.65	
DMG255	C			.47	.53	.49	.63	.76	.58	.90	.57		.80						1.00	
DMG256	Pb			.65	.67	.74	1.32	2.78	.81	.96	2.27		1.16						5.64	
DMG257	C			1.04	.94	1.04	1.46	1.29	1.06	.99	1.29		1.63						.72	
DMG546	M			.56	.48	.93	1.07	4.39	14.91	2.74	3.89		1.29						1.00	
DMG546[a]	M			.41	.52	.66	.77	7.58	19.25	7.79	5.04		1.61						.86	
DMG547	C/Fe	1.08	1.29	1.04	1.13	.85	.71	2.17	2.50	1.55	.67	.89	1.13	1.38	1.38	.99	.91	.60	.77	1.23
DMG548	C			.77	.71	.88	.79	.89	.97	.96	.91		.97	.50	6.85	.76	.39	3.15	.32	1.58
DMG552	C	1.08	1.34	1.10	1.02	1.45	1.26	.22	1.04	.64	.35	2.12	1.02						.35	
DMG553	C/Fe			1.27	1.50	1.30	1.39	2.46	2.83	1.82	.74		1.42						.97	.77
DMG554	M	.55	1.54	.56	.44	1.13	1.75	1.90	7.56	5.42	1.87	.62	1.53	.25	.52	.85	.21	2.42	.97	1.22
DMG555	C	1.56	1.93	.92	.84	1.37	1.52	.93	.85	.84	.91	9.38	1.10	.81	4.50	.88	.74	1.12	2.02	1.35
DMG556	C	1.60	1.61	1.36	1.14	1.74	1.56	2.76	1.13	1.12	3.01	2.70	.90	1.28	2.49	2.12	1.46	.41	2.22	
DMG557	C/Mn			1.57	1.26	1.47	.73	8.84	1.14	1.21	3.95		6.44						1.04	
DMG558	M	1.63		1.21	.30	1.51	.83	6.15	18.74	14.50	.49	1.22	1.44	1.39	.90	.66	2.97	.25	3.03	.94
DMG560	M		.70	1.22	.75	1.09	4.02	1.47	4.95	4.32	1.65		2.44						.86	
DMG561	M			.59	.44	1.19	1.40	1.26	19.49	15.16	6.97		1.91						1.70	
DMG563	M			.50	.35	.99	.62	2.26	23.74	9.95	1.14		.72						.44	
DMG563[a]	M			.76	.22	1.50	.59	4.03	36.89	6.87	2.70		.72						.38	.36
DMG564	C/Fe	1.00	.87	.74	.91	1.10	2.74	.85	3.40	2.83	1.08	.56	1.11	.85	2.02	.62	.78	1.57	.84	
DMG565	M	1.62	.78	1.03	.24	1.36	.22	4.72	1.84	2.43	2.49		.65	1.30	2.56	.61	1.04	1.34	.45	1.85
DMG567	C	1.18	1.70	1.30	1.01	1.45	1.99	.56	1.20	1.47	1.15	1.27	2.26	1.07	.74	.49	.20	1.30	.76	.80
DMG568	C/Fe		1.05	.95	1.21	1.58	1.28	.37	2.97	5.11	1.11	.63	1.10						.88	
DMG569	C			.90	.85	.79	.87	.96	.83	.99	1.77		1.41	1.43		1.81	1.48	.61	.65	.67
DMG587	C	1.27		1.11	1.01	.91	1.33	2.92	1.48	1.49	.77	2.94	1.94	1.20	1.38	2.33	2.55	4.27	1.08	3.14
DMG590	Pb	1.67	1.70	2.14	3.04	5.14	2.89	1.62	2.50	3.75	3.24	1.57	4.20	2.01	2.54	1.25	.76	.98	5.98	.92
DMG591	C/Mn	1.59	1.05	1.10	1.33	1.12	1.00	4.05	.58	.98	.58	2.20	.88	3.37	2.84	.65	2.47	.31	.21	.89
DMG592	C	1.64	.67	.89	.96	.83	.95	1.88	1.09	1.00	1.25	1.23	1.33	1.29	7.73	1.54	3.83	.03	.59	1.13
DMG594	C	1.47	1.30	1.13	.81	.93	.99	1.84	1.32	1.46	1.18	6.28	.91		3.52				1.62	
WJJ004	M			.57	.43	.89	.76	7.67	7.36	2.11	2.25		.77						1.12	
WJJ008	M			.48	.46	2.38	.88	6.82	16.65	3.68	2.43		1.08						2.17	
WJJ013	M			1.18	.71	1.32	.99	6.26	8.06	6.97	35.48		.97						2.24	
WJJ122	M			.67	.52	.61	.93	9.44	8.25	2.23	1.92		.56						.39	
WJJ124	M			.73	.54	.83	.85	5.09	9.72	3.02	1.56		.68						.56	

Note: [a] = replicates; M = mineral-based paints; C = carbon-based paint; C/Mn = carbon and manganese mixed paints; C/Fe= carbon and iron mixed paints; Pb = paint containing lead.
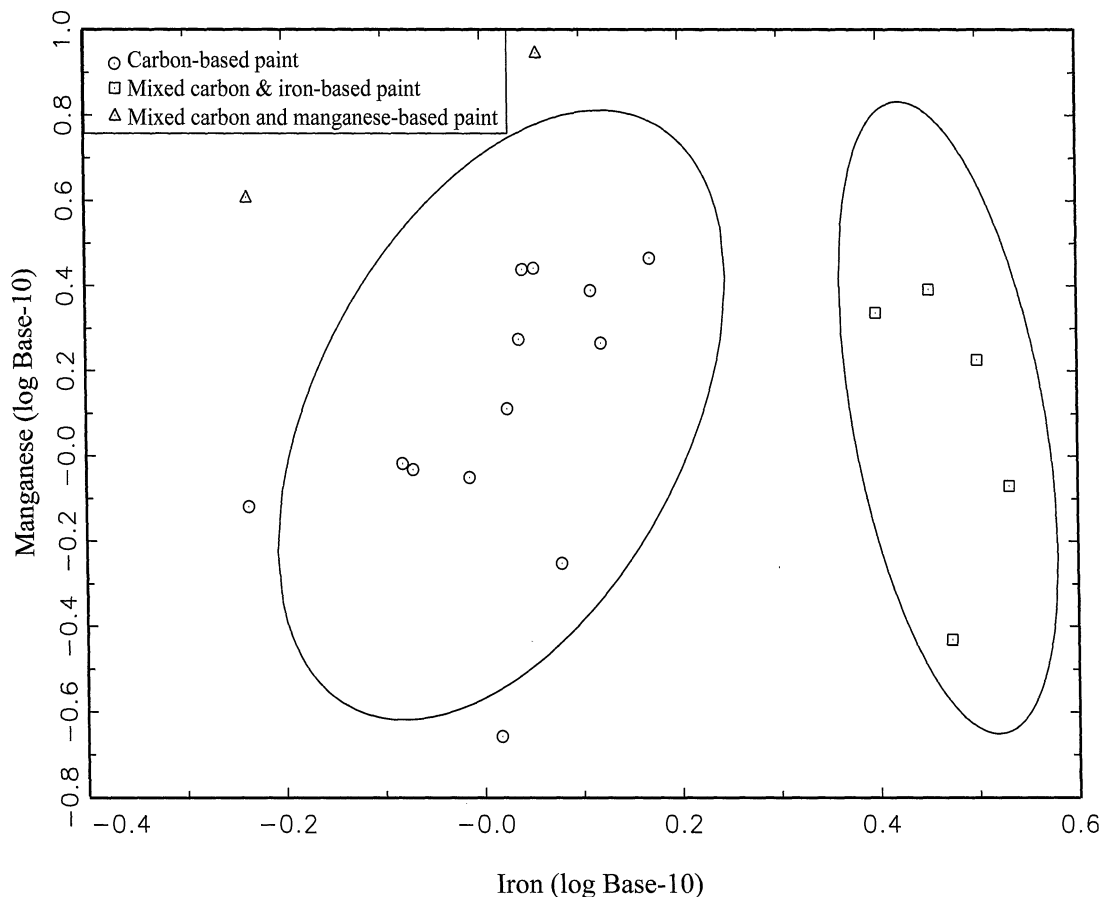
Figure 3. Bivariate plot showing separation of the carbon-based paint group from the mixed paint (mineral and carbon) groups. Ellipses represent 90% confidence levels for group membership

vanadium levels that are similar in both painted and unpainted areas. If there were a high manganese or vanadium concentration in the painted portion of this sample, then the painted areas would have been expressed in the elemental map as black and would correspond to the actual painted areas of the sherd. The fact that the paint on ADR007 is apparently organic-based is at odds with its classification as Mancos Black-on-white. On WJJ008, also classified as Mancos Black-on-white, the painted area clearly has a higher manganese and vanadium content than the unpainted area. Additionally, the actual painted area of the sherd correlates closely to the dark areas on the elemental maps. Finding mineral paint on this specimen accords with its classification as Mancos Black-on-white. These maps provide a clear picture of the effectiveness of our method for identifying mineral and organic paints.

The original purpose of this experiment was to test whether LA-ICP-MS was an appropriate method for separating mineral from carbon-based paints. We were surprised, however, by the amount of variation in paint "recipes" (Figure 2). Considering the full data set of paint analyses, it appears that different compositional groups exist in both the mineral and carbon-based paint categories (Table 1). Pottery assigned to the mineral group showed variation in the iron ratio values from 4.95 to 36.89. Other elements (e.g., Ni, Mn, and Co) also showed significant variation. While classification of paint as carbon based is only assumed (since this element is not easily measured by LA-ICP-MS), the low levels of transition metals in the paint for these samples support their classification as carbon based. However, samples assigned to the carbon-based paint group exhibit variation that additional sampling may help quan-

tify. For example, DMG555 and DMG594 are classified as having a carbon-based paint, but these samples have copper ratios of 9.38 and 6.28 respectively. DMG552 and DMG592 are also classified as having a carbon-based paint, but they have silver ratios of 6.85 and 7.73 respectively. This suggests that the carbon paint category may not be as clear-cut as it seems. Three samples (and two replicates) had lead ratios greater than 5.0. If lead had not been included in the study as an analyte of interest, these sherds would have easily been subsumed by the carbon-based paint group. Since lead normally occurs in clays in the low parts-per-million range, a high Pb ratio (as well as Ag, Cu, Zn, etc.) indicates a paint mixture in which lead is present but not necessarily as a major constituent. The paint in these samples may therefore be a carbon-paint mixture containing minor amounts of high-lead mineral such as galena. Interestingly, galena contains Ag and is frequently found with sulfides containing Cu and Zn. We classified several specimens as being composed of a mixed carbon- and mineral-paint (Figure 3). Carbon-iron mixed paints had iron ratios ranging from 2.5–3.4 and carbon-manganese paints had manganese ratios ranging from 4.05–8.84. It is possible that additional mixed-paint groups may become apparent in future studies.

Analysis of paint on pottery from the Mesa Verde region has demonstrated not only that carbon- and mineral-based categories can be readily distinguished using LA-ICP-MS, but that significant chemical variation exists in paints used to decorate Mancos and Mesa Verde Black-on-white. These differences may reflect different selections of raw materials by potters for ceramic decoration, differences between production loci, or temporal variation. It is also possible that the mixed paint categories may be a transitional paint type that occurs between Mancos Black-on-white and Mesa Verde Black-on-white. Additional sampling, development of a better method for data standardization, and analysis of a wider range of elements will ultimately permit these issues and others to be addressed.

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Notes

1. We have yet to establish prices for LA-ICP-MS analy-
ses at the MURR Archaeometry Laboratory. However, based
on the minimal time required for sample preparation and the
rapid analytic time, we believe the cost per sample will be
nominal. Depending upon the number of analytes, prepara-
tion and analysis of up to 80 samples per day is not unrea-
sonable. For those desiring analysis of 30–40 elements,
25–40 samples can be prepared and analyzed per day.

2. The small size of the ablation chamber (~2-x-5 cm)
does however make it necessary to remove a piece from
many sherds as discussed below.

3. We recognize that categorization of Mesa Verde Black-
on-white as a carbon-based paint and Mancos Black-on-
white as a mineral-based paint is not straightforward;
occasionally Mancos Black-on-white can have a carbon-
based paint and Mesa Verde Black-on-white can have a min-
eral-based paint. Stewart and Adams (1999) provide an
overview of problems associated with Mancos and Mesa
Verde Black-on-white typology.

4. Subsequent experiments have demonstrated that the
unpainted and unslipped areas of these sherds comprise a sin-
gle homogeneous compositional group that is chemically dis-
tinct from the black painted surfaces.