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2013-2014 PROJECTS

MAGNETIC AND GEOCHEMICAL CHARACTERIZATION OF IN SITU OBSIDIAN, NEW MEXICO*:*
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Students: *ALEXANDRA FREEMAN*, Colorado College, *ANDREW GREGOVICH*, Colorado College, *CAROLINE HACKETT*, Smith College, *MICHAEL HARRISON,* California State Univ.-Chico, *MICHAELA KIM*, Mt. Holyoke College, *ZACHARY OSBORNE*, St. Norbert College, *AUDRUANNA POLLEN*, Occidental College, *MARGO REGIER*, Beloit College, *KAREN ROTH*, Washington & Lee University

TECTONIC EVOLUTION OF THE FLYSCH OF THE CHUGACH TERRANE ON BARANOF ISLAND, ALASKA*:*
Faculty: *JOHN GARVER*, Union College, *CAMERON DAVIDSON*, Carleton College
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GEOCHEMICAL VARIABILITY OF OBSIDIAN IN WESTERN NEW MEXICO WITH LABORATORY-BASED PXRF

KAREN ROTH,Washington and Lee University
Research Advisor: Jeffrey Rahl

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Research Advisor: Jeffrey Rahl

INTRODUCTION

The American Southwest is dotted with obsidian-capped rhyolite flows produced by Neogene and Quaternary volcanism (Shackley, 2005). Prehistoric populations in the region discovered that obsidian, due to its siliceous composition and conchoidal fracturing, can be fashioned into hard projectile points with sharp edges. They collected obsidian at these flows, and their migration and trade spread it across the region. Southwestern obsidian points have been found at archaeological sites ranging from Mexico to Wyoming (Glascock, 1999; Shackley, 2005). By matching obsidian artifacts to their flow sources, archaeologists can reconstruct trade routes and population contacts throughout the American Southwest.

The first such sourcing studies, in the 1950s, used the variability in obsidian specific gravity between flows as a distinguishing characteristic. In the late 1960s and early 1970s archaeologists began using x-ray fluorescence (XRF) to characterize the geochemistry of obsidian. Due to melt fractionation, different obsidian flows contain different concentrations of trace elements; this geochemical variability provides a more precise method of sourcing artifacts (Shackley, 2005).

Prior XRF work with New Mexican obsidian has concentrated on chemically distinguishing and assigning artifacts to the various sources, frequently by using trace element bivariate plots and confidence ellipses to group and discriminate typical source concentrations (e.g. Duff et al., 2012; Glascock, 1999; Shackley, 1998; Shackley, 2005). The instrument utilized has primarily been desktop XRF. This study builds upon such work by employing portable XRF and multivariate statistical tests to characterize and distinguish the geochemistry of spatially referenced populations of samples, collected at a finer intra-site scale to establish inter-region, inter-site, and intra-site variability.

GEOLOGIC SETTING

This study uses obsidian from six source sites in two regions: Mule Creek (Fig. 1) and Mt. Taylor (Fig. 2), both in New Mexico. Mule Creek, part of the Mogollon-Datil volcanic field, is located in southwestern New Mexico near Silver City. Mt. Taylor, a composite volcano and part of the Jemez Lineament, is located in western New Mexico near Grants. The volcanism that produced the obsidian-bearing rhyolite in these regions dates to 17 Ma for Mule Creek and to 3.2 – 3.5 Ma for Mt. Taylor (Crumpler, 1982; Ratte, 2004). Within each region, obsidian was collected at three geographically distinct sites, each about 0.2 – 0.5 km^2 in area; the Mule Creek sites are known as North Sawmill Creek, Antelope Creek, and West Antelope Creek, and the Mt. Taylor sites are known as Grants Ridge, Horace Mesa, and La Jara Mesa. Obsidian is exposed as black marekanite nodules ranging from 2 – 4 cm in diameter at Mule Creek sites and 3 – 12 cm in diameter at Mt. Taylor sites. Marekanite nodules occur *in situ* in hydrated perlitized outcrop and as float weathering out from the ground surface. Obsidian specimens at all sources often bear percussion marks from ancient knappers testing the obsidian quality. At some sites (e.g., Grants Ridge) the marekanites contain sanidine phenocrysts, 2 – 5 mm long; at others (e.g., Horace Mesa) the marekanites are aphyric.

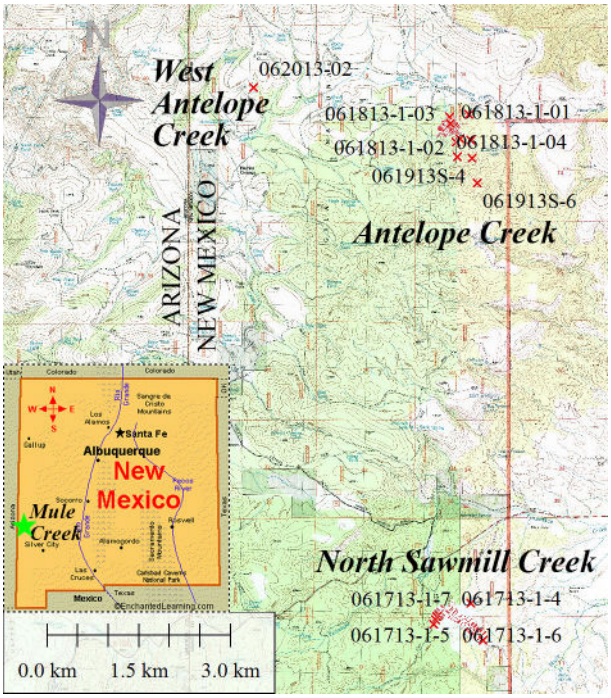

Figure 1. Location of samples at the three Mule Creek sites: North Sawmill Creek, Antelope Creek, and West Antelope Creek.

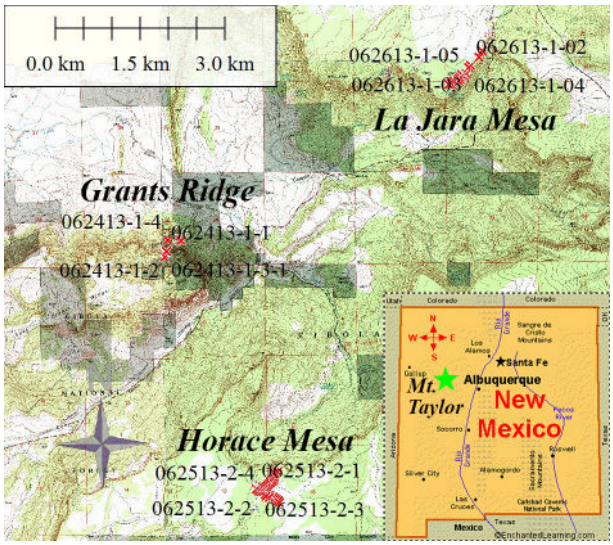

Figure 2. Location of samples at the three Mount Taylor sites: Grants Ridge, Horace Mesa, and La Jara Mesa.

DATA COLLECTION

Field methods

North Sawmill Creek. Samples were collected in three transects (oriented approximately southeast, northeast, and west) from the center of the site. Along each transect, the largest nodule available was sampled from the ground surface every five meters.

Antelope Creek. Seventeen populations were collected, with the samples of each population taken from one spot less than two meters squared. Each population was either chipped out from the perlite outcrop or collected from the ground surface in the center of tributary wash channels. Eleven of the populations were collected at points spread around the main area of the site, and six of the populations were collected at points on the southeastern ridge of the site. One transect around the perimeter of the central wash was also collected.

West Antelope Creek. Samples were collected from the ground surface within an area of approximately 160 m^2. These were not individually georeferenced.

Grants Ridge. Thirteen populations were collected along a hillside outcrop and in the wash channel in front of the outcrop, moving upslope.

Horace Mesa. Samples were collected along three transects (oriented approximately southwest, northeast, and southeast) from the center of the site. In each direction, samples were collected from the ground surface within segments 30 meters long (parallel to the transect) and 10 meters wide (perpendicular to the transect).

La Jara Mesa. Samples were collected along three parallel transects directed southwest from the site entry point. Due to the low nodule density, every nodule found was collected from the ground surface.

Laboratory methods

A total of 491 samples were analyzed: 180 from North Sawmill Creek, 220 from Antelope Creek, 10 from West Antelope Creek, 12 from Grants Ridge, 46 from Horace Mesa, and 23 from La Jara Mesa. Samples were washed in an ultrasonic machine for 20 – 25 minutes each to remove surface dirt that might affect the chemical analyses. They were split if necessary to create a flat surface, to reduce x-ray attenuation. The chemical composition of each sample was measured using portable energy dispersive x-ray fluorescence.

In an ED-XRF analysis, atoms in the sample are excited by x-rays emitted from the instrument. Energized electrons become dislodged from the

atoms' inner shells, leaving vacancies that are filled by outer shell electrons. This loss of energy is released in the form of fluorescent x-rays that return to the instrument's detector. Since electron shells are quantized, the fluorescent radiation has an energy characteristic of the element producing it. The intensity of each returning energy level is used to calculate the concentration of the corresponding element in the sample.

Each sample was analyzed with a Bruker Tracer III-SD Handheld XRF Spectrometer for 180 seconds at 40 kV and 55 μA, using a 12 mil Al, 1 mil Ti, 6 mil Cu filter to focus on the trace elements of interest. The resulting peak intensities were ratioed to the Compton peak of rhodium and converted to ppm concentrations using a modified version of the Bruker obsidian calibration (Speakman and Shackley, 2012). The concentrations of 10 elements were calculated: Mn, Fe, Zn, Ga, Rb, Sr, Y, Zr, Nb (all from the Kα peak), and Th (from the Lα peak).

DATA ANALYSIS

One-way multivariate analysis of variance

The inter-region, inter-site, and intra-site variability in concentration was compared using a one-way multivariate analysis of variance (MANOVA). A MANOVA tests the null hypothesis that vectors of means of observations are the same among populations (Harris, 1975). Rejection of this hypothesis indicates that the dependent variables (in this case, concentration of each trace element) vary due to change in the independent variable (in this case, sample location) rather than random error. MANOVA is an expansion of the Hotelling's T^2 statistic to more than two populations and an expansion of univariate analysis of variance (ANOVA) to more than one dependent variable (Harris, 1975). It is thus a multi-population, multivariate form of the Student's t-test, which tests the null hypothesis that the means of two populations are the same. The use of a MANOVA instead of multiple t-tests or ANOVAs controls the familywise error rate, reducing the increased probability of Type I errors from multiple hypotheses in multiple tests.

Calculations were performed in the MATLAB Statistics Toolbox using the *manova1* function. This function uses the Wilks' lambda test statistic as a hypothesis-testing criterion. Wilks' lambda is a measure of the proportion of variance in the dependent variables that cannot be attributed to the independent variable (Harris, 1975). The *p*-value for this test statistic provides the choice of accepting or rejecting the null hypothesis; if the *p*-value is less than the analysis' significance level, α, then the null hypothesis is rejected, meaning in this case that chemical composition varies with sample location.

Sixteen MANOVAs were run: four to assess the intra-site variability at North Sawmill Creek, Antelope Creek, Horace Mesa, and La Jara Mesa, six to assess the inter-site variability between each of the six sites and its neighboring sites in the region, and six to assess the inter-region variability between each of the six sites and sites in the other region. The number of samples available from West Antelope Creek and from Grants Ridge was too small to permit assessment of intra-site variability at these two sites. Since MANOVA is sensitive to differences in population size, the same number of populations and samples per population was used for the three scales of assessment at each site to permit comparison between, for example, variability at Antelope Creek and variability between Antelope Creek and the other Mule Creek sites. For each site, the maximum number of populations and samples per population that number and geographic spread of samples permitted was used. At sites without single-outcrop populations, transects or subsets of transects were used as populations, with subsets selected to maximize geographical distance between populations (for intra-site assessments) or to evenly represent all transects (for inter-site and inter-region assessments). Table 1 summarizes the parameters of each MANOVA and lists the resulting *p*-value.

As expected based on the results of Shackley (1998, 2005, 2008), the null hypothesis is rejected (meaning composition varies with location) for all the inter-region assessments and most of the inter-site assessments. Obsidian can be chemically distinguished between Mule Creek and Mount Taylor and among the Mule Creek sites.

Table 1. Results of the MANOVAs performed to compare concentration variability at the intra-site, inter-site, and inter-region scales. A population is a spatially-distinct suite of samples; a sample is an individual obsidian nodule representing one pXRF measurement. The level of significance for all runs was α = 0.05.

Run	Populations (n)	Samples/pop. (n)	*p*-value	*p*-value < alpha ?
Sawmill intra-site	3	35	2.8089E-01	No
Sawmill inter-site	3	35	2.1222E-02	Yes
Sawmill inter-region	3	35	7.9425E-06	Yes
Antelope intra-site	3	35	8.0280E-02	No
Antelope inter-site	3	35	1.7652E-04	Yes
Antelope inter-region	3	35	2.3140E-05	Yes
W. Ant. inter-site	3	10	4.0910E-02	Yes
W. Ant. inter-region	3	10	5.2680E-16	Yes
Grants inter-site	3	12	1.1507E-01	No
Grants inter-region	3	12	9.5408E-23	Yes
Horace intra-site	3	12	9.6438E-01	No
Horace inter-site	3	12	1.1507E-01	No
Horace inter-region	3	12	3.5143E-26	Yes
La Jara intra-site	3	7	7.7111E-01	No
La Jara inter-site	3	7	2.0658E-01	No
La Jara inter-region	3	7	9.6541E-11	Yes

However, the null hypothesis is not rejected (meaning composition is not dependent on location to a statistically-significant level) for all the intra-site assessments. At this scale, the obsidian flows are sufficiently homogenous that samples cannot be chemically distinguished between one part of the site and another.

The null hypothesis is not rejected at the inter-site scale for the three Mount Taylor sites, indicating that at least two of the sites are chemically indistinguishable. To identify these indistinguishable sites, three Hotelling's T^2 tests with $\alpha = 0.05$ were performed on the Mount Taylor sites using the MATLAB function *T2Hot2ih.m* (Trujillo-Ortiz and Hernandez-Walls, 2005). Hotelling's T^2 is the two-population version of MANOVA, testing the null hypothesis that vectors of means of observations are the same between the two populations (Harris, 1975). Test 1, with populations Grants Ridge and Horace Mesa, produced $p = 0.0000 < \alpha$; Test 2, with populations Grants Ridge and La Jara Mesa, produced a $p = 0.0000 < \alpha$; and Test 3, with populations Horace Mesa and La Jara Mesa, produced a $p = 0.1114 > \alpha$. The rejection of the null hypothesis for Tests 1 and 2, but not for Test 3, indicates that Grants Ridge is chemically distinguishable from both Horace Mesa and La Jara Mesa, while Horace Mesa and La Jara Mesa are chemically indistinguishable from each other. This agrees with the conclusions of Shackley (2013). Figure 3A demonstrates this result visually.

Principal component analysis

A principal component analysis was run to identify the dependent variables (element concentrations) that contribute most to any inter-region, inter-site, or intra-site variability present. Such an analysis identifies the elements that are most useful in assigning samples, and thus artifacts, to a source. Principal component analysis simplifies multiple variables into a new set of variables, the principal components, each of which is a linear combination of the original variables. Each successive principal component contains the successive greatest variance in the original variables, showing the directions of most variability in the structure of the original data (Harris, 1975). The analysis was performed in the MATLAB Statistics Toolbox using the *pca* function. Figures 3 and 4 demonstrate elements containing more and less variability in distinguishing among the three Mount Taylor sites. Zr and Nb, with larger coefficients for component 1 (Fig. 4), represent more variability and separate the samples into discrete populations (Fig.3A). Ga and Th, with smaller coefficients for component 1, represent less variability and do not separate the samples into discrete populations (Fig. 3B). Since the first principal component contains a greater variance than the second, an element (e.g. Rb for the Mount Taylor sites) may have a large coefficient for the second principal component but still not separate samples if it has a small coefficient for the first principal component.

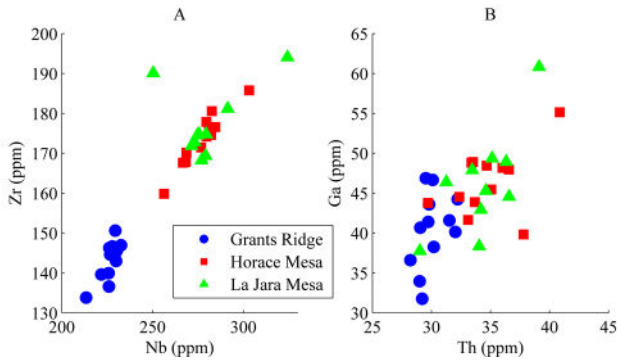

Figure 3. Bivariate plots of trace elements that contribute more (A) and less (B) to variability among the three Mount Taylor sites, as determined by the principal component analysis. The trace elements in A are noticeably more effective at separating the sites.

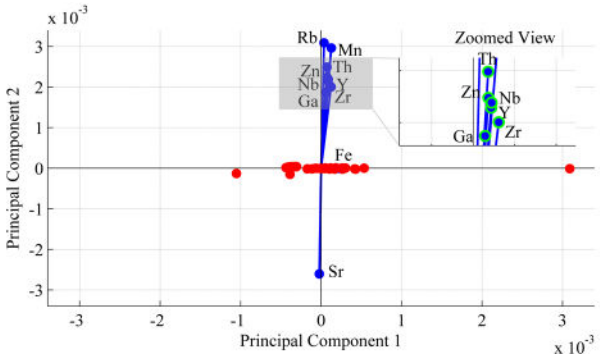

Figure 4. Biplot of the first two principal components from the PC analysis on the three Mount Taylor sites. Each blue/green point/vector combination represents the PC coefficients for an element, and each red point represents a sample. The direction and length of each blue vector indicates that element's contribution to the variability.

CONCLUSION

The geochemical variability of Mule Creek and Mount Taylor was compared at different scales. Chemical concentration was found to vary statistically at the largest scale, between the Mule Creek and Mount Taylor regions, and at the intermediate scale, between sites within each region, but not at the smallest scale, within a site. This supports the conclusions of Hughes (1993) that obsidian trace element composition is homogenous on the small scale of across a flow. Thus, georeferencing of samples within a site is most likely not necessary for geochemical studies.

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