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X-Ray Fluorescence (XRF) Analysis of Major Oxide and Trace Element Concentrations for an Obsidian Projectile Point From a Site at Furnace Creek, Cumberland Furnace, Tennessee

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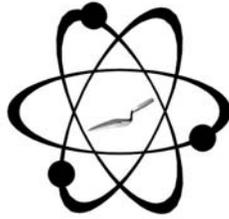
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**X-RAY FLUORESCENCE (XRF) ANALYSIS OF MAJOR OXIDE AND TRACE ELEMENT  
CONCENTRATIONS FOR AN OBSIDIAN PROJECTILE POINT FROM A SITE AT  
FURNACE CREEK, CUMBERLAND FURNACE, TENNESSEE**



by

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## INTRODUCTION

The analysis here of one obsidian projectile point from a site in north-central Tennessee indicates that the artifact was produced from one of the chemical groups at Glass Buttes, Lake County, Oregon. A short discussion of the provenance follows.

## LABORATORY SAMPLING, ANALYSIS AND INSTRUMENTATION

All archaeological samples are analyzed whole. The results presented here are quantitative in that they are derived from "filtered" intensity values ratioed to the appropriate x-ray continuum regions through a least squares fitting formula rather than plotting the proportions of the net intensities in a ternary system (McCarthy and Schamber 1981; Schamber 1977). Or more essentially, these data through the analysis of international rock standards, allow for inter-instrument comparison with a predictable degree of certainty (Hampel 1984; Shackley 2011).

### Trace Element Analyses

Trace element analyses were conducted to provide data for future comparisons with sources that may be discovered. All analyses for this study were conducted on a ThermoScientific *Quant'X* EDXRF spectrometer, located in the Geoarchaeological XRF Laboratory, Albuquerque, New Mexico. It is equipped with a thermoelectrically Peltier cooled solid-state Si(Li) X-ray detector, with a 50 kV, 50 W, ultra-high-flux end window bremsstrahlung, Rh target X-ray tube and a 76  $\mu\text{m}$  (3 mil) beryllium (Be) window (air cooled), that runs on a power supply operating 4-50 kV/0.02-1.0 mA at 0.02 increments. The spectrometer is equipped with a 200  $\text{l min}^{-1}$  Edwards vacuum pump, allowing for the analysis of lower-atomic-weight elements between sodium (Na) and titanium (Ti). Data acquisition is accomplished with a pulse processor and an analogue-to-digital converter. Elemental composition is identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background.

The analysis for mid Zb condition elements Ti-Nb, Pb, Th, the x-ray tube is operated at 30 kV, using a 0.05 mm (medium) Pd primary beam filter in an air path at 200 seconds livetime to generate x-ray intensity Ka-line data for elements titanium (Ti), manganese (Mn), iron (as  $\text{Fe}_2\text{O}_3^T$ ), cobalt (Co), nickel (Ni), copper, (Cu), zinc, (Zn), gallium (Ga), rubidium (Rb), strontium (Sr), yttrium (Y), zirconium (Zr), niobium (Nb), lead (Pb), and thorium (Th). Not all these elements are reported since their values in many volcanic rocks are very low. Trace element intensities were converted to concentration estimates by employing a quadratic calibration line ratioed to the Compton scatter established for each element from the analysis of international rock standards certified by the National Institute of Standards and Technology (NIST), the US. Geological Survey (USGS), Canadian Centre for Mineral and Energy Technology, and the Centre de Recherches Pétrographiques et Géochimiques in France (Govindaraju 1994). Line fitting is linear (XML) for all elements but Fe where a derivative fitting is used to improve the fit for iron and thus for all the other elements. When barium (Ba) is analyzed in the High Zb condition, the Rh tube is operated at 50 kV and up to 1.0 mA, ratioed to the bremsstrahlung region (see Davis 2011; Shackley 2011). Further details concerning the petrological choice of these elements in Southwest obsidians is available in Shackley (1988, 1995, 2005; also Mahood and Stimac 1991; and Hughes and Smith 1993). Nineteen specific pressed powder standards are used for the best fit regression calibration for elements Ti-Nb, Pb, Th, and Ba, include G-2 (basalt), AGV-2 (andesite), GSP-2 (granodiorite), SY-2 (syenite), BHVO-2 (hawaiite), STM-1 (syenite), QLO-1 (quartz latite), RGM-1 (obsidian), W-2 (diabase), BIR-1 (basalt), SDC-1 (mica schist), TLM-1 (tonalite), SCO-1 (shale), NOD-A-1 and NOD-P-1 (manganese) all US Geological Survey standards, NIST-278 (obsidian), U.S. National Institute of Standards and Technology, BE-N (basalt) from the Centre de Recherches Pétrographiques et

Géochimiques in France, and JR-1 and JR-2 (obsidian) from the Geological Survey of Japan (Govindaraju 1994).

### **Major and Minor Oxide Analysis**

Analysis of the major oxides of Na, Mg, Al, Si, P, K, Ca, Ti, Mn, and Fe is performed under the multiple conditions elucidated below. The composition of alkalis Na<sub>2</sub>O and K<sub>2</sub>O, and silica (SiO<sub>2</sub>) in rocks allows for elemental determination of rock type (LeBas et al. 1986; Table 1).

The fundamental parameter analysis (theoretical with standards), while not as accurate as destructive analyses (pressed powder and fusion disks) is usually within a few percent of actual, based on the analysis of USGS RGM-1 obsidian standard (see also Shackley 2011). The fundamental parameters (theoretical) method is run under conditions commensurate with the elements of interest and calibrated with four USGS standards (RGM-1, rhyolite; AGV-2, andesite; BHVO-1, hawaiiite; BIR-1, basalt), and one Japanese Geological Survey rhyolite standard (JR-1).

### **Conditions of Fundamental Parameter Analysis<sup>1</sup>**

#### **Low Za (Na, Mg, Al, Si, P)**

Voltage	6 kV	Current	Auto <sup>2</sup>
Livetime	100 seconds	Counts Limit	0
Filter	No Filter	Atmosphere	Vacuum
Maximum Energy	10 keV	Count Rate	Low

#### **Mid Zb (K, Ca, Ti, V, Cr, Mn, Fe)**

Voltage	32 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Pd (0.06 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	Medium

**High Zb (Sn, Sb, Ba, Ag, Cd)**

Voltage	50 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Cu (0.559 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	High

**Low Zb (S, Cl, K, Ca)**

Voltage	8 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Cellulose (0.06 mm)	Atmosphere	Vacuum
Maximum Energy	10 keV	Count Rate	Low

<sup>1</sup> Multiple conditions designed to ameliorate peak overlap identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background.

<sup>2</sup> Current is set automatically based on the mass absorption coefficient.

The data from the WinTrace software were translated directly into Excel for Windows and SPSS software for statistical. In order to evaluate these quantitative determinations, machine data were compared to measurements of known standards during each run. RGM-1 a USGS rhyolite standard is analyzed during each sample run for obsidian artifacts to check machine calibration (Table 1). Source provenance was determined by reference to source data at the laboratory, and Ambroz et al. (2001).

**DISCUSSION**

While the raw material used to produce this projectile point is certainly the high-silica rhyolite obsidian from Glass Buttes, Oregon, the prehistoric provenience could be questionable (see data in Table 1). This contracting stem point is poorly made and exhibits grinding on both faces. While this could be aboriginal, it is a technique used by modern knappers in point

production (Whittaker 2004). Nevertheless, prehistoric people did move obsidian across the North American continent in all time periods, and hydration analysis could resolve the issue (Dillian et al. 2010; Norton 2008). Glass Buttes, Oregon is a major source of archaeological obsidian in western North America (see [http://www.obsidianlab.com/image\\_maps/image\\_maps.html#or](http://www.obsidianlab.com/image_maps/image_maps.html#or)). The Glass Butte source complex is one of the larger volcanic fields dominated by Quaternary rhyolite eruptive events in the Cascade Range of northwestern North America. There are multiple chemical groups, and this artifact appears to be Group C, Glass Buttes based on comparison to data published in Ambroz et al. (2001).

#### REFERENCES CITED

- Ambroz, J.A., M.D. Glascock, and C.E. Skinner  
2001 Chemical Differentiation of Obsidian within the Glass Buttes Complex, Oregon. *Journal of Archaeological Science* 28:741-746.
- Davis, K.D., T.L. Jackson, M.S. Shackley, T. Teague, and J.H. Hampel  
2011 Factors Affecting the Energy-Dispersive X-Ray Fluorescence (EDXRF) Analysis of Archaeological Obsidian. In *X-Ray Fluorescence Spectrometry (XRF) in Geoarchaeology*, edited by M.S. Shackley, pp. 45-64. Springer, New York.
- Dillian, C.D., C.A. Bello, and M.S. Shackley  
2010 Long-Distance Exchange of Obsidian in the mid-Atlantic United States. In *Trade and Exchange: Archaeological Studies from History and Prehistory*, edited by C.D. Dillian, and C.L. White, pp. 17-36. Springer, New York
- Govindaraju, K.  
1994 1994 Compilation of Working Values and Sample Description for 383 Geostandards. *Geostandards Newsletter* 18 (special issue).
- Hampel, Joachim H.  
1984 Technical Considerations in X-ray Fluorescence Analysis of Obsidian. In *Obsidian Studies in the Great Basin*, edited by R.E. Hughes, pp. 21-25. Contributions of the University of California Archaeological Research Facility 45. Berkeley.
- Hildreth, W.  
1981 Gradients in Silicic Magma Chambers: Implications for Lithospheric Magmatism. *Journal of Geophysical Research* 86:10153-10192.

Hughes, Richard E., and Robert L. Smith

1993 Archaeology, Geology, and Geochemistry in Obsidian Provenance Studies. *In Scale on Archaeological and Geoscientific Perspectives*, edited by J.K. Stein and A.R. Linse, pp. 79-91. Geological Society of America Special Paper 283.

Mahood, Gail A., and James A. Stimac

1990 Trace-Element Partitioning in Pantellerites and Trachytes. *Geochemica et Cosmochimica Acta* 54:2257-2276.

McCarthy, J.J., and F.H. Schamber

1981 Least-Squares Fit with Digital Filter: A Status Report. In *Energy Dispersive X-ray Spectrometry*, edited by K.F.J. Heinrich, D.E. Newbury, R.L. Myklebust, and C.E. Fiori, pp. 273-296. National Bureau of Standards Special Publication 604, Washington, D.C.

Norton, M.R.

2008 Obsidian Research in Tennessee and Alabama. *Tennessee Archaeology* 3:123-130.

Schamber, F.H.

1977 A Modification of the Linear Least-Squares Fitting Method which Provides Continuum Suppression. In *X-ray Fluorescence Analysis of Environmental Samples*, edited by T.G. Dzubay, pp. 241-257. Ann Arbor Science Publishers.

Shackley, M. Steven

1988 Sources of Archaeological Obsidian in the Southwest: An Archaeological, Petrological, and Geochemical Study. *American Antiquity* 53(4):752-772.

1995 Sources of Archaeological Obsidian in the Greater American Southwest: An Update and Quantitative Analysis. *American Antiquity* 60(3):531-551.

2005 *Obsidian: Geology and Archaeology in the North American Southwest*. University of Arizona Press, Tucson.

2011 An Introduction to X-Ray Fluorescence (XRF) Analysis in Archaeology. In *X-Ray Fluorescence Spectrometry (XRF) in Geoarchaeology*, edited by M.S. Shackley, pp. 7-44. Springer, New York.

Whittaker, J.C.

2004 *American Flintknappers*. University of Texas Press, Austin.

Table 1. Oxide and Elemental concentrations for the artifact and USGS RGM-1. Measurements in parts per million (ppm) or percent by weight as noted.

SAMPLE	Ti	Mn	Fe	Zn	Rb	Sr	Y	Zr	Nb	Ba	Pb	Th	SOURCE
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	
FURNACE CR, TN RGM1-S4	989	338	7479	42	99	69	25	104	10	1496	16	17	Glass Buttes C, Oregon
	1516	291	13025	39	148	106	24	227	5	801	21	14	
	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	MnO	Fe2O3	Σ			
	%	%	%	%	%	%	%	%	%				
FURNACE CR, TN RGM1-S4	3.772	0	12.276	76.728	4.552	1.142	0.106	0.047	1.017	99.64			
	4.054	0	13.087	73.915	4.832	1.372	0.295	0.043	2.153	99.751			