

**THERMAL AND ENVIRONMENTAL EFFECTS ON OBSIDIAN
GEOCHEMISTRY: EXPERIMENTAL AND ARCHAEOLOGICAL
EVIDENCE**

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ABSTRACT

Recent EDXRF compositional studies of thermally altered archaeological obsidian from a number of late period sites in New Mexico and Arizona suggested that extreme thermal alteration may have been responsible for the depletion of elemental concentrations in the mid-Z x-ray region; a region where the most sensitive incompatible elements for the discrimination of archaeological obsidians reside. A stepped heating experiment subjecting samples of peraluminous to mildly peralkaline artifact quality obsidian to temperatures between 500° C and 1080°C indicated that at temperatures over 1000°C extreme mechanical changes occur, but the elemental composition in the mid-Z region does not vary greatly beyond that expected in typical instrumental error. It appears that the apparent depletion of elemental concentrations in the archaeological specimens is due to EDXRF analysis of surface regions where melted sands in the depositional matrix become bonded to the surface glass and subsequently incorporated into the results. If accurate analyses of burned obsidian artifacts are desired, the layer of melted sand from the depositional contexts must be removed before analysis.

KEYWORDS: OBSIDIAN, EXTREME THERMAL ALTERATION, NORTH AMERICAN SOUTHWEST, SITE DEPOSITIONAL EFFECTS, ENERGY DISPERSIVE X-RAY FLUORESCENCE

INTRODUCTION

Recently, a number of obsidian studies in pre-Classic Salado and Hohokam, as well as northern Rio Grande contexts have focused on the potential effects of pre-depositional and post-depositional burning on the trace element chemistry of archaeological obsidian (Shackley 1998a; Steffen 1999a, 1999b). These studies, while informative, were not conducted in controlled laboratory conditions focused on thermal threshold rates to determine at which temperature, if any, trace element composition may change significantly (c.f. Skinner et al. 1997; Trembour 1990). Our purpose here is to discuss the results of a controlled laboratory experiment focused specifically on the thermal effects on archaeological obsidian within a background of archaeological applications in the American Southwest, and an understanding of thermal gradients in silicic melts. The results presented here, of course, are likely applicable anywhere.

ARCHAEOLOGICAL BACKGROUND

In the past few years large scale archaeological projects in Arizona and New Mexico have, as part of problem domain generation, integrated archaeological obsidian studies into analytical research (see Bayman and Shackley 1999; Peterson et al. 1997; Shackley 1995, 1999, 2000; Simon et al. 1994). Evident for over 60 years is the periodic and often culturally produced pre-depositional and post-depositional burning of obsidian artifacts (Gladwin et al. 1938; Shackley 1988, 1990). Cremation, common in pre-Classic Hohokam and Mogollon contexts is the most obvious vector for the pre-depositional effects, but post-occupational burning of rooms and entire sites is also responsible for surface modification of obsidian artifacts (Foster 1994). Gladwin and Haury's excavations at Snaketown in predominately pre-Classic contexts are the best known studies where cremations were common and artifacts

burned to varying degrees (Doyel 1996; Gladwin et al. 1938; Hoffman 1997; Haury 1976; Figure 1 here). Recent analyses of pre-Classic and Classic period burned obsidian artifacts, often projectile points from these contexts, have indicated significant variability in the source element chemistry inconsistent with typical rhyolite glass composition (Cann 1983; Peterson et al. 1997; Shackley 1998a). Analysis of artifacts from burned contexts in Rooms 15 and 16 of the Upper Ruin at Tonto National Monument indicated partial to nearly complete depletion of trace elements in three of 19 specimens (Shackley 1998a). All of these Tonto Ruin specimens, like the Snaketown artifacts, exhibited a thin layer of melted material, likely from the surrounding matrix. As we shall see, this latter attribute is the operative issue hampering reliable trace element compositional studies, not necessarily direct high temperature effects.

THE NATURE OF SILICIC MAGMA COOLING BEHAVIOR AND CHEMISTRY

As a background to understanding both the modal trace element composition of silicic glasses and temperature properties, a slight digressive discussion of melt temperatures will be useful. Magmas erupted on the earth's surface are quite hot and dangerously explosive, particularly silicic magmas, so there have been few direct studies (Carmichael et al. 1974). Macdonald and Gibson's (1969) measurement of the peralkaline obsidian at the Chabbi eruption in Ethiopia in 1968 and Carmichael's (1967) estimates are the most appropriate here (see also Buddington and Lindsley 1964; Table 2 here). These measurements are made with mineral geothermometers using two minerals (usually titanomagnetite and ilmenite) to estimate the liquidus temperature of the silicic lava; by theory the equilibration temperature of the mineral pair closely approximates the liquidus temperature (Buddington and Lindsley 1964; Carmichael et al. 1974:6; Hildreth 1979). Those shown in Table 2 are considered

upper limits, and for this exercise the temperature that we would expect to see physical and possibly chemical changes. Given these data, our initial firing began at 500° C. The process of volatilization and subsequent removal of some compounds such as water and silica is apparently not an intervening variable (Hildreth 1979, 1981).

HIGH TEMPERATURE EXPERIMENTAL PROCEDURES

Sample Description

Thirteen samples from five different obsidian sources in the greater American Southwest and northwest Mexico were heated and analyzed. All samples were megascopically aphyric; no megascopically observable phenocrysts. For each obsidian source, at least two nodules were sampled in order to establish a source differentiation baseline beyond that previously reported (see Shackley 1995).

Thirteen nodules from five known obsidian sources in the Arizona, New Mexico, and northern Chihuahua were split to obtain fresh surfaces and avoid contamination during analysis (Table 1, Figure 2). The five sources include both peraluminous and mildly peralkaline lavas in order to attempt to cover the spectrum of trace element variability typical of silicic glasses (see Cann 1983; Mahood and Hildreth 1983; Hildreth 1981; Shackley 1988). Each flake was weighed, measured, optically scanned, and analyzed using EDXRF prior to any heating for baseline comparative data (Table 1). Additionally, for each flake, the analyzed surface was recorded and all future XRF analyses were performed on the same surface. The Spectrace 400 instrument used in the Department of Geology and Geophysics is well reported and instrumental settings and laboratory standards are reported elsewhere (see Davis et al. 1998; Shackley 1995, 1998b;

<http://obsidian.pahma.berkeley.edu/tontobs/anlysis.htm>). A summary is included in the Appendix herein.

Heating

Obsidian samples were heated using a Blue Electric Furnace in the Petrography Lab, Department of Geology and Geophysics, University of California, Berkeley. The kiln was lined with ceramic plating and linked to a digital thermometer to accurately monitor temperature. To maintain a constant heating temperature, the kiln thermostat was checked and adjusted manually throughout each heating session. The same obsidian samples were heated during each session, and each was weighed and examined for physical changes following heating. After every heating session, flakes were submitted to EDXRF analysis. The samples were subjected to five heating sessions (Step 1 through Step 5) of increasingly higher temperatures.

Step 1: 500°C. The kiln was pre-heated to 500°C, and samples placed loosely on the ceramic plate inside the kiln. The kiln was closed and monitored until the temperature again reached 500°C. It took 30 minutes for the temperature to return to 500°C. Samples were heated at 500°C for one hour. After one hour, the kiln was turned off and the kiln door opened. Samples cooled inside the kiln for 30 minutes.

The obsidian samples were then weighed and analyzed using EDXRF. No weight or chemical changes were detected. Samples were also visually inspected for physical changes. No physical changes were apparent after heating at 500°C.

Step 2: 700°C. The kiln was pre-heated to 700°C, and samples placed loosely on the ceramic plate inside the kiln. The kiln was then closed and monitored until the temperature reached 700°C. It took 15 minutes for the interior kiln temperature to return to 700°C.

Samples heated inside the closed kiln at 700°C for one hour. After one hour, the kiln was turned off and the door opened. Samples cooled completely inside the kiln.

After cooling, samples were again weighed and analyzed using ED-XRF. No weight or chemical changes were detected. Minor physical changes were noted in one sample: Vulture #2. This sample exhibited a band of white discoloration and minor vesiculation on a small section of the flake's cortical surface. No other changes were noted.

Step 3: 800°C. The procedure for Step 3 was identical to that of Steps 1 and 2. Samples were placed loosely on the ceramic plate in the pre-heated kiln. It took 30 minutes for the internal kiln temperature to return to 800°C. One sample, Burro Creek #2, cracked from heat stress when placed on the heated ceramic plate. Samples were heated at 800°C for one hour and then allowed to cool completely inside the kiln with the door open.

After cooling, samples were again weighed and analyzed using EDXRF. No weight or chemical changes were detected. Minor physical changes were noted in three samples: Vulture #3 exhibited minor vesiculation and a white discoloration along one edge of the flake. Cow Canyon #1 showed a reddening of residual cortical material on the dorsal surface of the flake. The dorsal surface was not analyzed using EDXRF. Antelope Wells #2 exhibited melting and vesiculation of cortical material along the flake edge. Again, the cortical surface was not analyzed using EDXRF.

Step 4: 940°C. Due to thermal cracking of the one sample during Step 3, minor procedural changes were enacted during Step 4. In Step 4, the kiln was pre-heated to 350°C and samples were then placed on the ceramic plate inside the kiln and the door closed. The internal temperature was then raised to 940°C. It took one hour for the internal kiln temperature to reach 940°C. The samples remained inside the kiln at 940°C for an additional

hour. After heating, the kiln was turned off and samples were allowed to cool inside the kiln with the door closed until the temperature reached 600°C, at which point the kiln door was opened and the samples cooled completely.

Again, samples were weighed and analyzed using EDXRF. No weight or chemical changes were noted after heating at 940°C. Upon visual inspection, no additional physical changes were noted.

Step 5: 1080°C. In Step 5, samples were placed in a cold kiln to avoid thermal fractures. It took 90 minutes for the internal kiln temperature to reach 1080°C. Samples were heated at 1080°C for one hour and then allowed to cool in the kiln with the door closed for 45 minutes until the temperature reached 600°C. The kiln door was then opened and samples cooled completely.

Severe physical changes were observed in all samples after heating at 1080°C for one hour (Figure 2). Both Antelope Wells samples melted, and all other samples exhibited severe vesiculation due to off-gassing. Because of melting and expansion of the obsidian samples, some samples fused together or fused to the ceramic plate inside the kiln, making accurate weight measurements impossible. For the two samples that were not fused with the ceramic plate, Vulture #3 and Government Mountain #1, no weight changes were apparent. Given this, it seems reasonable to conclude that no heavy compounds came out of solution due to heating. Chemical changes, as shown through EDXRF, will be discussed below.

Summary of Physical Changes

Only minor physical changes, limited to thin edges and cortical surfaces, were apparent from heating prior to Step 5 at 1080°C. Heating to 1080°C caused severe physical changes to the obsidian samples, quite expectable given the predictive data on silicic magma

extrusion temperatures. Minor physical changes began after 700°C in the range of extrusion temperatures predicted by Carmichael (1967) and others. Due to melting and fusion of the obsidian samples inside the kiln at some temperature over 940°C, weight measurements were not available for most of the samples. However for the two samples that were not fused, no weight changes were apparent.

CHANGES IN ELEMENTAL CHEMISTRY

While physical changes in the glass samples were abrupt and extraordinary, more importantly, the elemental chemistry exhibited no significant changes with a few important exceptions. For most of the samples, there was no statistically significant changes in trace element chemistry between ambient and the temperature beyond the melting point of silicic lava (ca. 1000°C), above that expected and typical in the instrumental variability of EDXRF (see Davis et al. 1998).

Table 3 exhibits the measured elemental chemistry at ambient through all heating steps to 1080°C (see also Figure 4). Those elemental changes over 10% are shown in bold and underline. These changes are not necessarily related to the most obvious physical changes and do not correlate with modal chemistry (peraluminous versus peralkaline), or other samples analyzed here from the same source. Most intriguing is the complete depletion of titanium in the Government Mountain 2 sample, while the other two from this source showed no significant change. This is not immediately explicable, nor necessarily important archaeologically as we will argue. The Vulture 3 specimen gained over 30 ppm (about a 19% change) in rubidium, although this may be related to analysis of a small amount of ceramic material incorporated into the obsidian at the last step as discussed earlier (Figure 4). The only significant shift in elemental composition was in one of the mildly peralkaline

glasses from Antelope Wells (Table 3). Both rubidium and zirconium were depleted; 20% for rubidium and 17% for zirconium. The three-dimensional and biplots of the data graphically indicate this change.

Figure 5 graphically displays the conundrum presented by the Antelope Wells data. One of the samples was affected such that source assignment *could* be a problem, however, given that only rubidium and zirconium were affected, source assignment could be confident in a typical assemblage of archaeological obsidian in the southern Southwest. What is more of a concern is the effect on only one of the samples. Sample AW-1 is well within the range of variability on these two elements for Antelope Wells. While Antelope Wells is distinctive in the Southwest north of the border, recent research in the basin and range region of northern Chihuahua indicates a number of peralkaline obsidians used in prehistory that have similarly high proportions of iron and zirconium (see Shackley 1995, 1999). This could cause a problem in this region, particularly since surveys and geoprospection are in their infancy in the Basin and Range region of northern Chihuahua unlike the portion of the Southwest north of the border (Shackley 1995, 1999). As we will argue, however, pragmatic considerations make this apparent problem, less of an issue.

SITE DEPOSITIONAL ISSUES AND HIGH TEMPERATURE INCORPORATION OF SURROUNDING MATRIX

Not surprisingly, the high temperature experiments suggested that material incorporated into the glass can modify expected trace element composition. And while we are arguing that high temperature modification of artifact quality obsidian will not necessarily inhibit confident assignment to source, another physical change will cause problems.

As mentioned earlier, artifacts subjected to high temperatures are relatively common in the Southwest, particularly in pre-Classic and Classic contexts in central Arizona due to inclusion in cremation, domestic trash burning, or deliberate or accidental domestic house fires. The most well known examples are those from cremation contexts such as the obsidian points recovered during excavations at Snaketown (Figure 6). Based on the experiments discussed above, some of these artifacts must have been subjected to temperatures near or over melting point. Most importantly here, are the examples that while not exhibiting physical evidence of melting, are coated with material incorporated into the surface at near melting temperatures (Figure 6).

However, we recently analyzed an obsidian assemblage from two rooms of the Upper Ruin at Tonto National Monument in Tonto Basin, central Arizona (Shackley 1998a). Both rooms were subjected to what appears to be a high temperature fire, probably sometime during occupation. Three of the 22 samples analyzed were pieces of debitage that exhibited various degrees of surface accumulation from the surrounding matrix, one completely covered. As you can see in Table 4, two of the samples could be assigned to the Superior (Picketpost Mountain) source with reservation due to partial depletion of trace element concentrations, and one appeared nearly completely depleted in trace elements even though a small break indicated that it was indeed obsidian (Figures 7 and 8).

What is apparent here is that while we were initially concerned that high temperatures were exclusively responsible for the depletion of trace element concentrations, the depletion is *only* apparent and due to the limitations of EDXRF. Energy Dispersive XRF at the 30kV tube voltage used in these analyses penetrates the surface only approximately 4-5 microns (μm). Therefore, any significant surface accumulations will be analyzed rather than the glass

itself. Either the surface must be cleaned, the artifact broken to present a unobstructed surface, or not analyzed at all. Newer EDXRF technology, such as KeveX's Omicron™ instrument that can analyze very small areas, may ameliorate this problem in some artifacts.

SUMMARY AND RECOMMENDATIONS: THE PRAGMATIC APPROACH

At least two conclusions can be derived from these experiments relevant to archaeological applications of EDXRF analysis of archaeological obsidian. First, there appears to be no significant change in elemental composition up to temperatures above 1000°C, particularly for peraluminous silicic glasses. This is predictable given recent theory and practical experiments in the understanding of silicic melt temperatures. Second, the real problem lies in the interaction between those artifacts that were subjected to high temperatures and accumulated surrounding matrix on the surface combined with the analytical limitations of EDXRF. But are these issues really causing significant problems in the use of obsidian compositional data in addressing archaeological problems? In this experimental analysis of 13 samples, only one exhibited significant changes in the trace element composition such that source assignment became hazardous. Indeed, this Antelope Wells sample could still be assigned to source with some degree of confidence using up to five or six of the other EDXRF measured elements that were not affected. In the case of the Upper Ruin assemblage from Tonto National Monument, only three artifacts were affected by surface accumulation and only one could not be assigned to source.

What we conclude is that melting temperatures have no significant effect on the elemental composition of obsidian (at least those elements of interest here), but the surface accumulation of surrounding matrix on some artifacts can affect our ability to assign artifacts to source. This latter issue can be ameliorated by using more advanced technology,

removing the coating in some manner, or eliminating that artifact from the analysis. So, the physical changes that occur due to extreme heat do not necessarily present a problem in assigning source provenance.

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Table 1. Physical data for the experimental obsidian samples.

Obsidian Source	Specimen #	Max. Length	Max. Width	Max. Thickness	Pre-heated weight	weight 500°C	weight 700°C	weight 800°C	weight 940°C	weight 1080°C
Vulture, AZ	2	1.85	1.7	1.1	2.9	2.9	2.9	2.9	2.9	2.9 additional material ¹
Vulture, AZ	3	1.8	1.7	0.3	1	1	1	1	1	1
Burro Creek, AZ	1	2.8	2.1	0.85	3.8	3.8	3.8	3.8	3.8	3.8 additional material
Burro Creek, AZ	2	2.7	2.2	0.5	3.1	3.1	3.1	3.1	3.1	3.1 broken
Burro Creek, AZ	3	1.7	1.5	0.35	1	0.9	0.9	0.9	0.9	0.9 broken
Antelope Wells, NM	1(7-B-8) ²	3.2	2.5	0.8	5.9	5.9	5.9	5.9	5.9	5.9 ceramic fused
Antelope Wells, NM	2(13-B-1)	1.9	1.35	0.6	1.3	1.3	1.3	1.3	1.3	1.3 ceramic fused
Cow Canyon, AZ	1	2.7	1.5	0.5	1.3	1.3	1.3	1.3	1.3	1.3 additional material
Cow Canyon, AZ	2	3	1.9	1	5.6	5.6	5.6	5.6	5.6	5.6 broken
Cow Canyon, AZ	3	3	1.4	0.6	1.4	1.4	1.4	1.4	1.4	1.4 broken
Government Mt., AZ	1	4.7	3.1	1.05	10.9	10.9	10.9	10.9	10.9	10.9
Government Mt., AZ	2	3.8	2.05	0.5	2	2	2	2	2	2 broken
Government Mt., AZ	3	2.6	3.15	0.8	6.3	6.3	6.3	6.3	6.3	6.3 broken

¹ At some point during heating to 1080° C, the ceramic sample base in the kiln shattered and some of this material was incorporated into the melted glass.

² Designation for Antelope Wells sample splits also reported in Shackley (1995).

Table 2. Estimated melt extrusion temperatures for various lavas (from Carmichael et al. 1974). Rhyolite temperature underlined.

Kilauea, Hawaii	Tholeiitic basalt	1150–1225°C	T. L. Wright et al. (1968)
Parícutin, Mexico	Basaltic andesite	1020–1110°C	Zies (1946)
Nyiragongo, Congo	Nephelinite	980°C	Sahama and Meyer (1958)
Nyamuragira, Congo	Leucite basalt	1095°C	Verhoogen (1948)
Taupo, New Zealand	Pyroxene rhyolite: pumice flows	860–890°C	Ewart et al. (1971)
	Amphibole rhyolites: lavas, ignimbrites, pumice flows	735–780°C	
	Rhyolite lavas	<u>790–820°C</u>	
Mono Craters, California	Rhyolite lavas	<u>790–820°C</u>	Carmichael (1967a)
Iceland	Rhyodacite obsidians	900–925°C	
New Britain, Southwest Pacific	Andesite pumice	940–990°C	Heming and Carmichael (1973); Lowder (1970)
	Dacite lava, pumice	925°C	
	Rhyodacite pumice	880°C	

Table 3. Elemental concentrations for the analysis of the five source standards at ambient through 1080°C. Bold and underlined concentrations are those Ti, Mn, Fe, and Rb – Nb that exhibited more than 10% change. All measurements in parts per million (ppm).

TEMP	SAMPLE	Ti	Mn	Fe	Zn	Ga	Pb	Th	Rb	Sr	Y	Zr	Nb	
ambient	V2	1115.1	336.8	8412.5	36.4	17.9	26.8	15.6	136.0	37.0	19.2	128.2	18.9	
	V3	945.4	317.5	8229.7	43.8	20.8	27.8	32.2	140.9	39.2	19.5	121.7	18.4	
	BC1	569.5	466.6	9088.7	44.1	23.7	40.6	37.3	350.8	1.2	70.9	99.6	41.6	
	BC2	563.6	462.8	9188.9	42.2	19.6	39.8	37.4	351.6	3.4	69.6	96.2	43.7	
	BC3	476.0	446.0	8882.9	50.2	20.2	37.1	33.5	334.4	6.5	66.7	91.4	47.5	
	AW1	1661.7	1044.1	23944.0	191.7	24.6	49.6	41.5	365.6	3.8	136.8	1308.5	97.8	
	AW2	2009.2	971.1	21450.1	161.0	25.5	42.0	33.8	314.9	3.6	119.3	1190.6	96.7	
	CC1	1243.1	589.8	10096.4	61.4	18.8	27.9	14.4	157.3	120.2	24.3	144.1	20.5	
	CC2	1143.2	502.0	9421.6	44.0	16.7	22.5	17.1	141.5	113.5	25.7	133.0	20.3	
	CC3	1090.8	492.4	9091.3	46.1	13.6	23.9	18.5	143.6	111.3	24.4	130.7	18.5	
	GM1	510.7	550.7	9513.7	57.9	22.3	35.9	18.2	116.1	77.5	19.5	82.8	57.0	
	GM2	559.7	578.1	10355.7	57.5	25.0	37.6	17.1	117.9	79.7	19.2	79.5	58.8	
	GM3	526.7	551.0	9512.1	55.3	22.8	35.7	16.7	111.7	75.1	20.7	79.2	52.4	
	500°C	V2	1318.4	348.1	8777.3	38.7	14.9	23.9	21.1	143.4	39.1	17.9	124.6	25.4
		V3	1024.5	317.0	8177.0	32.3	18.7	26.9	26.4	141.2	34.5	23.8	131.0	21.6
BC1		620.5	484.1	9091.5	43.0	20.9	37.3	38.6	357.1	4.2	70.4	96.6	45.8	
BC2		642.9	496.6	9403.0	44.5	21.7	41.8	36.1	355.7	3.3	69.2	97.2	44.1	
BC3		536.9	446.2	9040.0	44.1	17.8	41.4	39.5	337.2	3.6	69.9	91.7	43.4	
AW1		1681.0	999.6	23473.0	174.5	23.2	45.3	40.6	360.4	4.2	134.5	1298.6	105.0	
AW2		1769.4	954.4	22128.2	187.6	24.2	42.6	38.9	312.5	4.6	118.9	1153.1	93.7	
CC1		1119.3	549.5	9969.1	98.9	26.9	30.4	18.8	148.9	111.5	29.8	136.9	17.8	
CC2		1143.9	425.1	8895.4	47.4	16.4	22.0	17.0	137.3	107.5	25.2	129.9	21.1	
CC3		1193.4	536.4	9970.7	54.1	20.6	29.3	18.4	157.7	119.0	24.9	138.8	23.3	
GM1		622.8	532.4	9400.1	60.4	21.6	38.1	15.5	113.3	78.6	21.4	84.1	51.8	
GM2		479.4	628.3	10398.8	59.1	26.2	35.2	0.0	124.1	82.2	23.4	84.8	55.1	
GM3		533.5	500.8	9176.7	54.5	21.4	33.1	15.9	112.4	76.1	19.5	75.0	49.1	
700°C		V2	1055.8	323.4	8158.4	34.8	19.4	32.6	13.4	130.9	36.3	16.8	123.8	16.1
		V3	994.1	341.3	8527.3	34.3	16.7	23.2	15.9	139.4	37.1	18.2	129.0	23.7
	BC1	675.3	457.9	9064.8	42.6	19.5	41.8	28.9	344.4	4.8	69.5	96.4	43.6	
	BC2	581.2	466.5	9395.5	54.7	23.9	41.3	40.8	348.1	2.9	69.6	96.7	48.6	
	BC3	618.9	455.2	9004.2	46.8	20.0	42.7	41.1	333.1	3.3	66.6	90.0	42.2	
	AW1	1714.0	946.5	22531.4	173.9	25.4	41.6	34.8	354.8	5.6	136.5	1287.2	95.0	
	AW2	1974.0	942.1	22596.9	188.2	22.6	50.0	49.3	313.4	2.5	118.8	1152.8	89.8	
	CC1	1315.9	530.4	9672.4	46.9	19.4	25.4	18.4	143.1	117.9	25.2	137.4	19.3	
	CC2	1124.1	486.2	9312.5	49.0	16.3	23.9	21.0	143.4	111.2	24.1	133.6	17.2	
	CC3	1354.0	594.4	10286.6	51.6	20.1	23.6	15.0	156.5	122.7	30.2	139.7	19.7	
	GM1	542.3	535.0	9455.3	55.3	21.6	36.3	18.7	117.8	77.8	16.7	81.8	57.6	
	GM2	485.6	628.5	10367.6	61.4	25.0	40.8	22.9	122.7	84.0	20.9	81.1	50.8	
	GM3	495.2	561.6	9711.8	54.7	22.8	37.1	20.4	117.3	78.1	23.5	82.9	54.4	
	800°C	V2	1168.2	350.1	8541.3	47.2	23.5	27.7	20.5	140.0	37.8	16.8	125.3	15.6
		V3	1010.4	329.2	8365.2	38.6	19.8	28.4	19.8	144.1	33.2	20.0	129.8	20.7
BC1		542.0	498.7	9245.9	53.3	22.4	43.0	38.5	364.1	4.1	69.6	98.7	45.4	
BC2		628.1	465.8	9004.4	49.3	23.0	41.0	38.2	345.4	3.0	66.9	93.6	44.4	
BC3		551.9	452.0	8922.2	36.4	18.2	38.8	40.1	343.4	2.6	69.7	92.4	40.9	
AW1		1807.1	1032.4	24632.8	183.6	24.1	45.9	46.1	379.1	4.8	137.3	1333.3	101.2	
AW2		1794.8	864.5	20596.9	151.3	24.6	44.6	43.3	308.5	5.0	117.5	1149.8	95.2	
CC1		1097.8	439.9	8930.8	48.2	14.9	21.2	17.3	141.0	109.6	24.6	128.2	22.1	
CC2		1177.3	495.5	9517.7	72.1	22.8	28.4	20.0	151.6	110.6	22.6	134.5	17.6	
CC3		965.8	464.4	9140.8	44.9	19.9	22.6	21.0	145.2	108.7	24.9	129.5	18.6	
GM1		532.1	568.3	9636.0	53.9	23.0	34.1	20.5	119.9	81.2	22.9	84.5	53.9	
GM2		582.2	671.5	10551.8	70.6	22.8	39.7	20.6	123.5	85.0	23.6	82.7	59.8	
GM3		524.4	570.3	9599.6	56.6	21.3	34.1	21.3	118.7	77.9	21.1	81.3	54.0	

Table 3 (cont.)

TEMP	SAMPLE	Ti	Mn	Fe	Zn	Ga	Pb	Th	Rb	Sr	Y	Zr	Nb	
940°C	V2	1069.2	295.0	8476.0	40.3	17.5	31.7	23.8	140.8	37.5	18.4	130.1	13.8	
	V3	1074.0	342.6	8508.8	37.2	17.7	28.2	14.2	142.4	36.9	19.5	127.7	20.7	
	BC1	636.5	495.3	9160.5	48.0	22.3	42.8	40.1	350.3	3.1	70.5	101.7	48.6	
	BC2	573.9	487.2	9113.8	40.9	21.3	38.1	35.4	348.3	2.3	71.0	98.6	42.5	
	BC3	580.7	467.6	9155.9	53.4	21.7	38.8	29.4	352.0	2.1	66.8	98.5	41.4	
	AW1	1652.6	1034.0	23297.7	177.7	24.7	47.8	52.9	367.3	4.2	136.5	1306.2	99.1	
	AW2	2201.9	1046.4	22446.3	165.4	24.3	42.5	36.5	317.2	4.9	120.5	1192.3	97.6	
	CC1	1303.9	640.9	10596.6	57.9	21.6	29.4	19.9	164.5	121.8	23.6	143.3	19.9	
	CC2	1151.9	542.5	9470.2	46.6	17.9	25.4	12.9	146.1	114.5	28.1	136.1	22.0	
	CC3	1282.4	541.0	9972.7	53.9	20.1	22.2	19.7	158.1	121.3	28.1	141.2	18.1	
	GM1	563.3	526.0	9333.0	59.6	22.4	35.3	20.0	114.6	81.9	20.0	77.5	58.2	
	GM2	0.0	613.2	10645.7	66.5	24.9	38.5	19.2	123.3	85.5	24.2	83.8	57.9	
	GM3	529.1	553.4	9583.4	60.8	19.2	36.8	17.7	115.8	75.6	21.0	79.9	51.9	
	1010°C	V2?	1014.3	344.6	8415.0	34.6	18.0	59.8	31.6	150.8	37.9	19.4	135.9	22.0
		V3	1639.3	476.2	10662.4	62.5	27.2	57.0	23.3	172.9	45.7	23.5	146.4	25.1
BC1?		550.1	515.6	9673.5	44.3	24.5	59.9	39.1	358.0	3.5	72.1	97.1	43.7	
BC2?		511.2	400.0	9006.5	49.1	22.3	43.5	29.9	324.7	3.0	65.6	96.1	42.8	
BC3		624.4	571.4	10325.9	56.6	23.7	60.1	39.4	383.7	5.2	74.8	102.7	41.7	
AW1		1654.8	1006.4	22707.6	173.3	26.7	43.8	40.6	339.5	5.1	125.8	1240.9	95.7	
AW2		1735.5	651.1	17325.5	136.8	14.9	39.0	24.0	253.1	6.5	105.8	986.7	80.4	
CC1		1165.3	520.9	9620.3	96.8	23.8	43.7	15.0	147.3	111.8	25.2	131.8	16.6	
CC2?		1191.6	502.3	9528.1	46.2	18.1	41.8	21.0	144.9	113.4	28.0	138.1	19.3	
CC3?		1142.5	506.4	9597.7	51.4	18.0	32.3	23.6	151.3	108.5	25.8	123.6	21.0	
GM1		496.3	537.4	9397.2	54.3	20.5	50.9	14.2	109.6	75.7	21.8	82.8	51.5	
GM2?		0.0	478.1	9163.5	56.0	20.7	40.8	16.1	108.8	74.7	23.3	75.7	46.2	
GM3		473.7	491.7	9204.2	56.9	20.9	52.2	15.9	109.3	76.8	20.2	79.4	52.8	

¹ Those samples marked with a “?” are samples that deformed too much to determine which sample of the source group that particular sample belonged.

Table 4a. X-ray fluorescence concentrations for archaeological samples from Rooms 15 and 16, Upper Ruin, Tonto National Monument (from Shackley 1998a). All measurements in parts per million (ppm).

SAMPLE	Ti	Mn	Fe	Rb	Sr	Y	Zr	Nb	Source ¹
140	1243.5	208.3	8365.7	101.7	30.0	17.9	100.6	1.2	Superior*
156	771.3	301.4	6285.3	75.8	12.9	16.5	54.0	16.1	Superior*
294	597.8	26.8	4116.1	4.8	14.2	0.0	7.0	4.1	burned*

¹ These are source probabilities based on best linear fit of the calibration utility (Shackley 1995). Those samples marked with "*" can only tentatively be assigned to source due to a less than adequate fit with the available source standards. These samples appear to be burned and/or chemically weathered such that the elemental chemistry may be altered.

Table 4b. Superior (Picketpost Mountain), Arizona source standard mean and central tendency data (Shackley 1995).

Element	Mean	Std Dev	Minimum	Maximum	N
Ti	831.84	148.94	708.9	1298.1	13
Mn	489.01	19.63	455.8	536.6	13
Fe	7873.22	163.89	7518.1	8175.4	13
Rb	130.23	2.74	125.7	136.3	13
Sr	19.09	2.03	15.9	21.7	13
Y	25.26	2.13	20.5	28.7	13
Zr	99.83	2.64	94.5	104.9	13
Nb	32.51	1.79	29.3	35.4	13
Ba	243.7	5.57	237.0	254.6	13

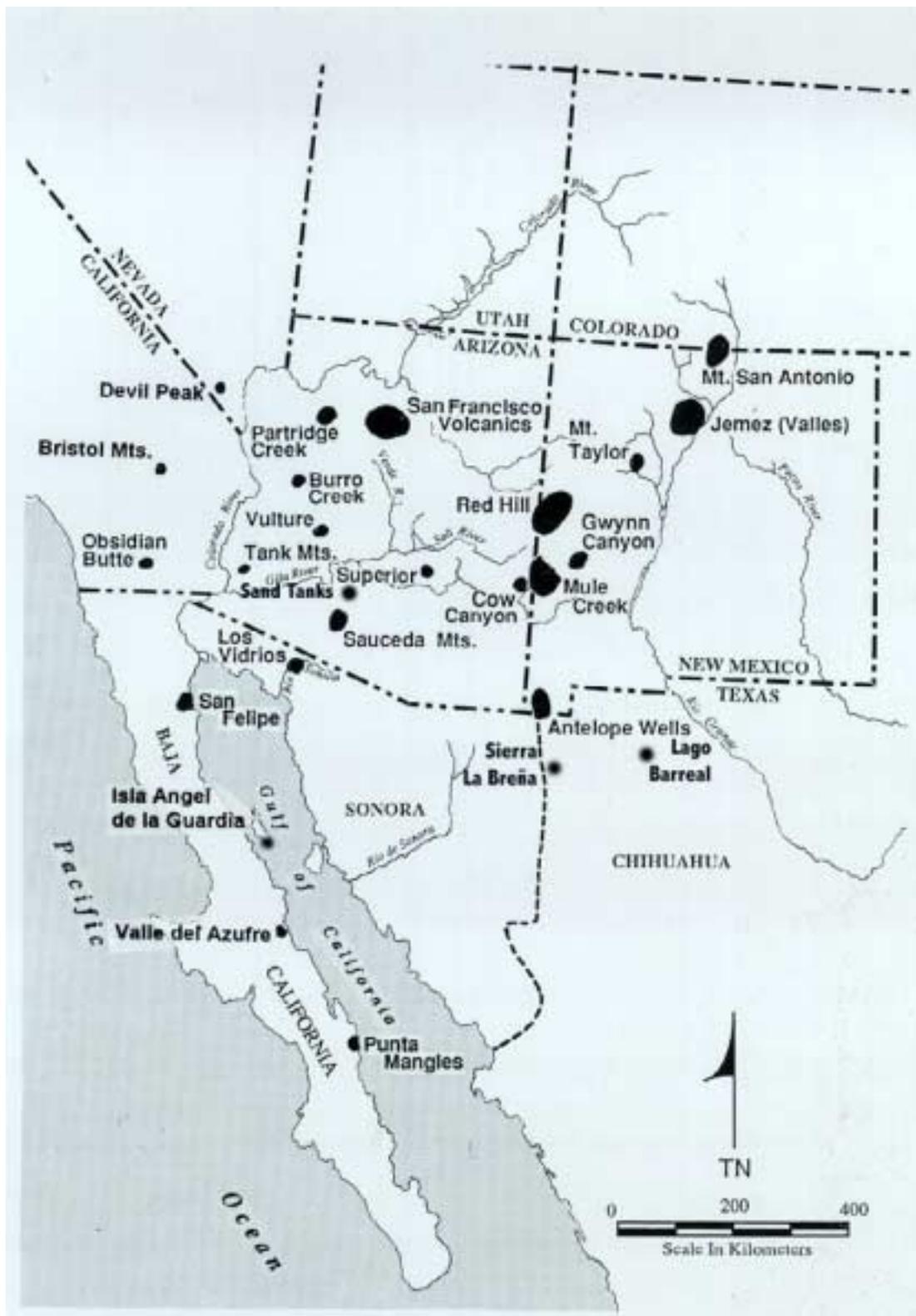


Figure 1. Sources of archaeological obsidian in the greater North American Southwest.



Figure 2. Experimental samples before heating.



Figure 3. Samples after heating to 1080°C. Off-white material is the broken ceramic base plate incorporated into glass while heating to this temperature (see text).

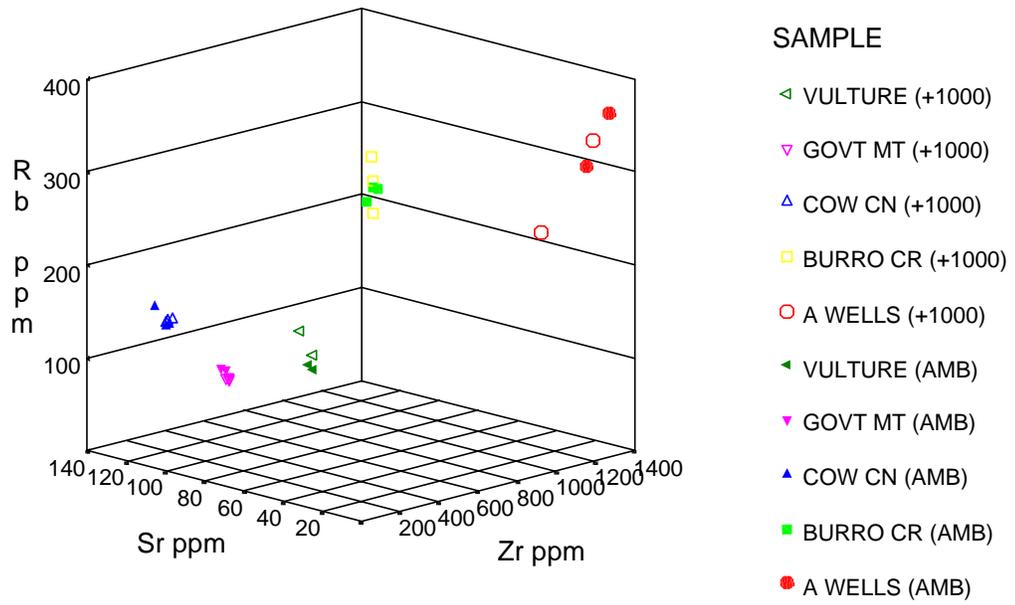


Figure 4. Rb, Sr, Zr ambient and 1080°C concentrations for experimental samples. AMB=ambient measurements; +1000=1080°C measurements.

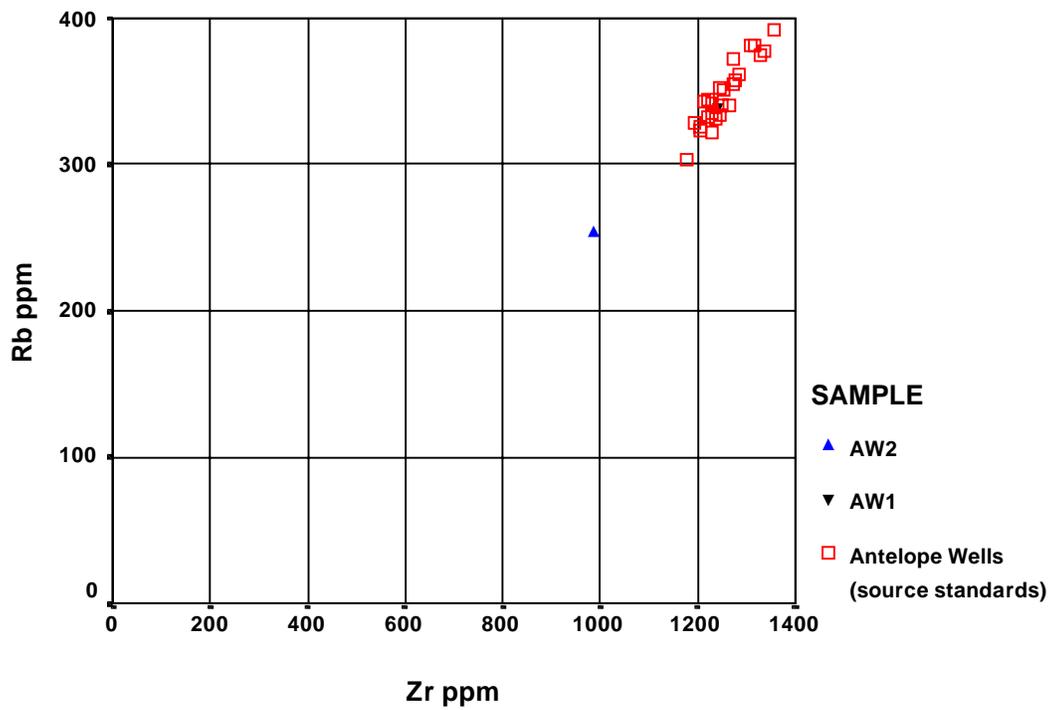


Figure 5. Rb versus Zr biplot of elemental concentrations for Antelope Wells samples and source standards after heating to 1080°C.



Figure 6. Top: Selected Snaketown Serrated points from Snaketown. Approximately 40% are burned to some degree. All could be assigned to source (from Shackley 2000); Severely burned and physically modified projectile points from Snaketown. Note incorporation of matrix on center and right specimens (from Gladwin et al. 1938, plate XXXVII bottom).



Figure 7. Burned obsidian sample with surface accumulation of matrix (Sample 294, Room 16, Upper Ruin; courtesy WACC/NPS).

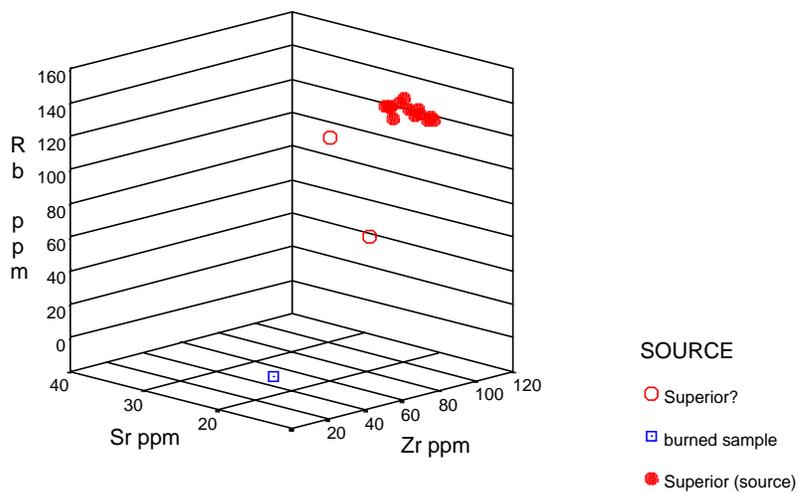


Figure 8. Rb, Sr, Zr three-dimensional plot of three artifacts from the Upper Ruin, Tonto National Monument, and Superior (Picketpost Mtn) source standards.