

## Energy Dispersive X-Ray Fluorescence Analysis of Obsidian From Dog Hill and Burns Butte, Oregon

### Abstract

Non-destructive energy dispersive x-ray fluorescence analyses were performed on rhyolitic volcanic glasses (obsidian) from Dog Hill and Burns Butte, Oregon. Sixteen trace and rare earth element concentrations were determined in parts per million (ppm) for each source, showing that these two obsidian sources are quite distinctive geochemically despite their close proximity to one another. In addition to demonstrating geochemical differences between sources, the results of the present study show that archaeological artifacts from east-central Oregon can be matched to these sources on the basis of quantitative measurements of trace element compositions.

### Introduction

Over the past few years, archaeologists increasingly have employed techniques derived from the physical sciences to assist in detecting patterning in archaeological residues. In western North America considerable interest has accrued to obsidian studies, because obsidian can be used to establish chronological sequences through obsidian hydration dating (*cf.* Friedman and Long 1976, Michels and Tsong 1980), and because the geochemical composition of volcanic glass makes it possible to determine the parent geological source of materials used to manufacture prehistoric tools. Once the parent sources of obsidian have been characterized geochemically, artifacts from archaeological sites can be analyzed and assigned to their probable geologic source on the basis of similarities in trace and rare earth element composition. This step completed, it is then possible to attempt reconstructing prehistoric distribution (or exchange) routes and to investigate changes or continuities in direction and intensity using different categories of tools to monitor different kinds of prehistoric activities (*e.g.* Hughes 1986b, Hughes and Bettinger 1984).

Despite the large number of obsidian source deposits in Oregon, comparatively little research on prehistoric obsidian trade has been conducted in the state. This dearth of research has not occurred because of lack of interest by prehistorians, but rather because there has been, until recently, rather little published on the actual

trace element composition of volcanic glasses. In short, it would be premature to inaugurate broad studies of obsidian artifacts from archaeological sites without first compiling a quantitative data base on obsidian source compositions to which the prehistoric objects can be compared. Although trace and rare earth concentration values have been published for some sources (*e.g.* Jack and Carmichael 1969, Laidley and McKay 1971, Higgins 1973, Hughes 1986b, Nelson 1984<sup>2</sup>), these data represent only a fraction of the total resource base. The immediate goals of this study are to present quantitative trace and rare earth element measurements on obsidian from Dog Hill and Burns Butte and, more generally, to contribute to the establishment of a geochemical data base for Oregon volcanic glasses that can be used in the service of prehistory.

One additional obstacle for archaeological research has been that previous quantitative measurements of Oregon obsidians, with one exception (Hughes 1986b), have been derived from analyses requiring crushing and powdering of some portion of the sample. This, of course, is undesirable from an archaeological perspective because of the importance of conserving unique and non-replaceable prehistoric artifacts. The non-destructive x-ray fluorescence technique employed in the present study avoids this drawback, since it is capable of generating quantitative estimates of trace and rare earth element com-

<sup>2</sup>The marked disagreement between trace element concentration values for Nelson's (1984:53, source #57) "Burns" source and those generated here for Burns Butte (see Table 2) suggests that Nelson's sample was not collected from Burns Butte, but from a different locality in the vicinity of Burns.

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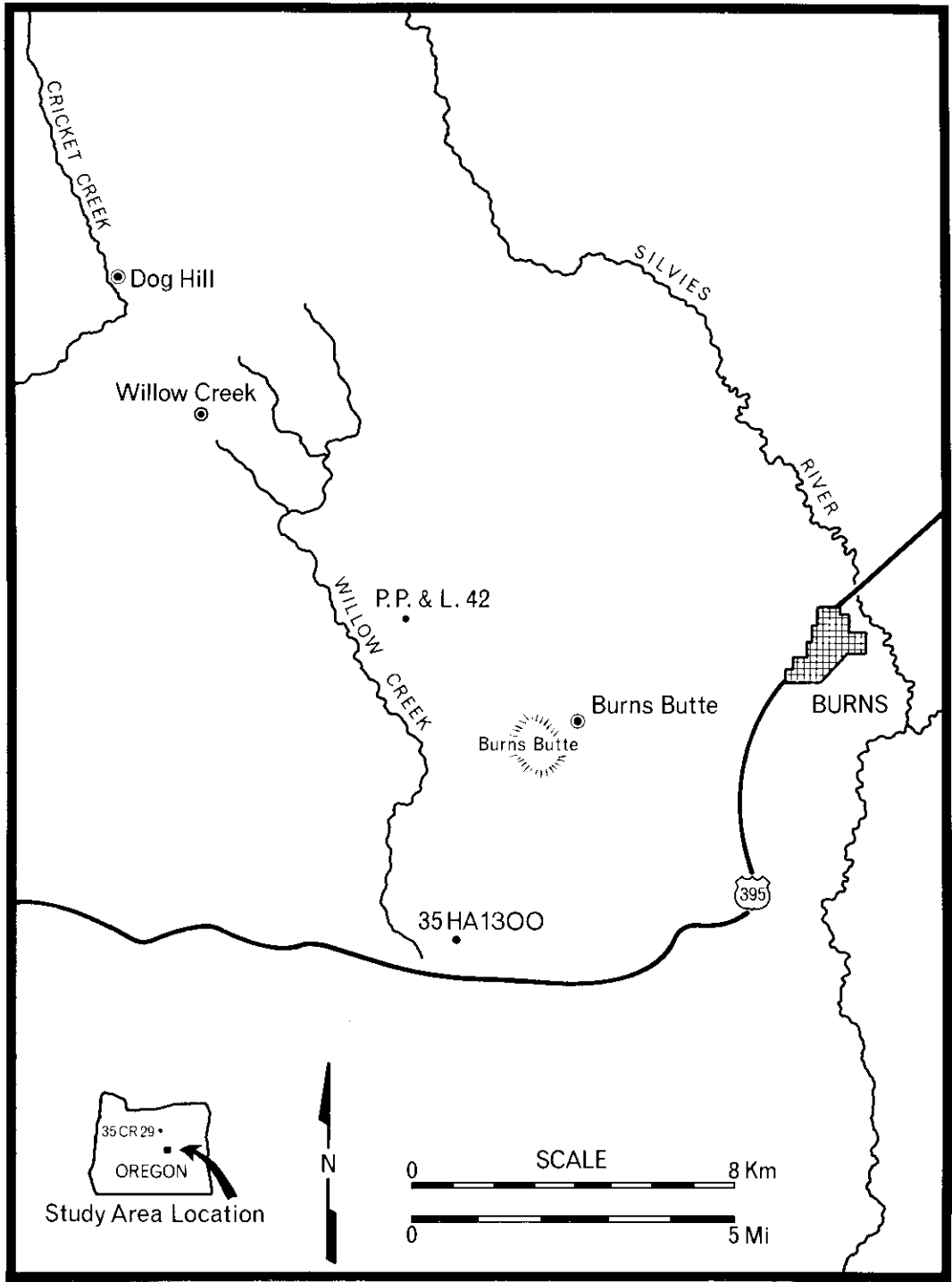


Figure 1. Map of east-central Oregon showing the locations of obsidian sources (dots within circles) and archaeological sites (dots) discussed in the text.

position that compare favorably to those determined using more time-consuming, destructive methods (*cf.* Hughes 1984, 1986a, 1986b; Nelson 1984). Once the elemental geochemistry of existing glasses is determined, it will be possible to employ these data in the study of prehistoric obsidian trade in central Oregon and adjacent areas.

### The Setting

The Dog Hill obsidian source is located in the southeastern portion of the Snow Mountain Ranger District, Ochoco National Forest, Harney County, Oregon (see Figure 1). The source occupies portions of T22S, R29E, NW1/4 of Section 17 with smaller portions in the E1/2 of NE1/4 of Section 18 and the SE1/4 of the SE1/4 of Section 7 as depicted on the U.S.G.S. Burns 3 NE, 7.5 minute quadrangle, 1978 (W.B.M.). As recorded by U.S. Forest Service personnel, the source and associated prehistoric quarrying debris covered more than 200 acres on both sides of Cricket Creek, located west of the Silvies River. Samples from Dog Hill occurred locally as cobbles and nodules ranging from 20 cm to ca. 1-2 cm in diameter; these were collected systematically from three discrete zones where obsidian quarrying debris was abundant (see below).

Burns Butte has been known for some time (*e.g.* Gail 1966, Ericson 1981:182) as a source for high-quality volcanic glass (see Figure 1). Unlike the sampling procedure employed at Dog Hill, samples from Burns Butte were collected only from one restricted area in T23S, R30E, NW 1/4 of NW 1/4 of Section 21 as depicted on the U.S.G.S. Burns, Oregon, 15 minute series quadrangle, 1960 (W.B.M.). Burns Butte obsidian cobbles were larger than those observed at Dog Hill; some of these ranged up to ca. 30 cm. in diameter.

### Methods

Analyses were conducted on 30 unmodified flakes ( $n = 15$  from each source) cleaved from obsidian nodules. After flakes were detached from cobbles with a tungsten carbide mallet they were rinsed in distilled water to remove surface contaminants that might affect the analysis, catalogued, secured in position in the 20-position rotating automatic sample changer tray, and analyzed. Other than this, no sample preparation was required.

Laboratory investigations were conducted by the author at the Department of Geology and Geophysics, University of California, Berkeley, on a Spectrace 440 (United Scientific Corporation) energy dispersive x-ray fluorescence machine equipped with a Tracor Northern 1221 100 MHz analog to digital converter, a Tracor Northern 2000 computer based analyzer with a LSI-11 microcomputer with 24k word capacity, and a Si(Li) solid state detector with 142 eV resolution (FWHM) at 5.9 keV in a 30 mm<sup>2</sup> area. Trace element analyses in the 5-25 keV region of the energy spectrum were conducted using a Rh x-ray tube operated at 30.0 kV, .40 mA pulsed with a .04 mm Rh primary beam filter; analyses in the 20-60 keV region were made using an Am<sup>241</sup> 100 mCi radio-isotope (Hughes 1986b). Ni (K $\alpha$ ), Cu (K $\alpha$ ), Zn (K $\alpha$ ), Ga (K $\alpha$ ), Pb (L $\beta$ ), Th (L $\alpha$ ), Rb (K $\alpha$ ), Y(K $\alpha$ ), Zr (K $\alpha$ ), and Nb (K $\alpha$ ) analytical lines were excited by the Rh x-ray tube, while Ba (K $\alpha$ ), La (K $\alpha$ ), Ce (K $\alpha$ ), Pr(K $\alpha$ ), and Nd(K $\alpha$ ) lines were excited by the Am<sup>241</sup> source. All analyses were conducted at 200 deadtime corrected seconds (livetime). Following excitation, the contribution of overlapping K $\alpha$ , K  $\beta$ , and L lines was subtracted (stripped) from the peaks of interest, yielding the "fingerprints" that appear in Figure 2 (*cf.* Hampel 1984). Background-subtracted net peak intensities were compared and ratioed to the Rh K $\alpha$  (Franzini, Leoni, and Saitta 1976) and the Am<sup>241</sup> peak continua, then converted to parts per million using a linear least squares fitting routine (Schamber 1977, McCarthy and Schamber 1981, Bice 1980:421-422).

### Results

The overall accuracy of the x-ray fluorescence system is illustrated in Table 1. This table shows that most of the quantitative trace and rare earth elemental measurements generated from the present study are in close agreement with recommended values for U.S. Geological Survey international rock standards. Pr, Cu, and Ni are not measured well by non-destructive x-ray fluorescence, because concentrations for these elements in obsidians often fall below the minimum limits of detection (Vane 1977) at 200 seconds livetime.

Coefficients of variation computed from sample means and standard deviations show that these measurements vary across elements (see

TABLE 1. X-ray fluorescence determinations for trace and rare earth element concentrations in three U.S. Geological Survey standard rocks compared to recommended values. All values in parts per million.  $\pm$ , counting error uncertainty at 200 seconds livetime (Schamber 1977:249).

U.S. Geological Survey Standard	Th	Pb	Nd	Elements Pr	Ce	La	Ba	Nb
W-1 (Flanagan 1976)	2.4	7.8	15.0	3.4	23.0	9.8	160.0	9.5
W-1 (This Study)	0.0 $\pm 0.0$	10.0 $\pm 1.9$	22.9 $\pm 5.4$	9.1 $\pm 5.0$	24.7 $\pm 4.5$	11.8 $\pm 3.9$	152.0 $\pm 7.6$	12.3 $\pm 2.2$
GSP-1 (Flanagan 1976)	104.0	51.3	188.0	50.0	394.0	191.0	1300.0	29.9
GSP-1 (This Study)	99.9 $\pm 5.0$	51.5 $\pm 2.3$	170.7 $\pm 11.2$	47.9 $\pm 9.4$	401.5 $\pm 12.3$	181.2 $\pm 9.7$	1237.9 $\pm 22.2$	28.7 $\pm 2.0$
G-2 (Flanagan 1976)	24.2	31.2	60.0	19.0	150.0	96.0	1870.0	13.5
G-2 (This Study)	22.3 $\pm 3.9$	34.9 $\pm 2.1$	53.7 $\pm 10.3$	14.2 $\pm 9.3$	150.1 $\pm 9.5$	78.5 $\pm 8.2$	1884.3 $\pm 28.8$	20.0 $\pm 2.0$

U.S. Geological Survey Standard	Zr	Y	Sr	Elements Rb	Ga	Zn	Cu	Ni
W-1 (Flanagan 1976)	105.0	25.0	190.0	21.0	16.0	86.0	110.0	76.0
W-1 (This Study)	107.6 $\pm 3.1$	24.1 $\pm 2.7$	187.8 $\pm 3.7$	20.9 $\pm 2.0$	16.2 $\pm 2.8$	88.9 $\pm 6.7$	111.4 $\pm 6.7$	86.9 $\pm 8.8$
GSP-1 (Flanagan 1976)	500.0	30.4	233.0	254.0	22.0	98.0	33.3	12.5
GSP-1 (This Study)	517.5 $\pm 4.7$	30.7 $\pm 2.7$	232.4 $\pm 3.5$	260.0 $\pm 3.7$	24.9 $\pm 2.9$	115.4 $\pm 7.4$	37.5 $\pm 4.9$	17.1 $\pm 7.6$
G-2 (Flanagan 1976)	300.0	12.0	479.0	168.0	22.9	85.0	11.7	5.1
G-2 (This Study)	310.5 $\pm 3.9$	14.5 $\pm 2.5$	496.3 $\pm 4.9$	178.0 $\pm 3.2$	22.2 $\pm 2.6$	97.2 $\pm 7.0$	10.6 $\pm 3.9$	10.6 $\pm 7.1$

Table 2). Th and Sr measurements, for example, are more variable than Pb, Nd, Ce, La, Ba, Nb, Zr, Y, Rb, Ga, and Zn, suggesting that they may not be as useful as the latter elements in distinguishing between obsidian sources (Hughes 1982:176, 1984). However, high "apparent" measurement error (relatively high CV% values) will be registered when the absolute concentration for an element approaches its minimum limits of detection (e.g. Sr concentrations in Dog Hill obsidian), and when "true" intersource elemental variability exists. In short, both instrumental measurement limitations and inherent geochemical variability may be represented in CV% values (Hughes 1986a).

As discussed above, collections were made from three different areas of the Dog Hill quarry where different surface densities of artifacts and

obsidian nodules were observed. Specimens from these three areas were catalogued separately; source standards from the north collection zone have "Z2N-" catalogue prefixes, those collected from the southern area of the quarry have "Z2S-" prefixes, while samples collected from the eastern area of the site are specified by "Z3-" catalogue prefixes (Table 3). To investigate the possibility that these three collection areas contained obsidians of different geochemical composition, the nine best measured elements (Rb, Sr, Y, Zr, Nb, Ba, La, Ce, and Zn) were grouped by collection area and a one-way analysis of variance (Blalock 1972:317-334, Hays 1973:457-491) was performed on these three geographic categories (Table 4). Since the F-ratios determined for all nine elements show no significant departure from that expected by

TABLE 2. Means ( $\bar{X}$ ), sample standard deviations (S.D.) and coefficients of variation (CV %) for 16 trace and rare earth element measurements on obsidian samples from Dog Hill and Burns Butte, Oregon. Means and standard deviations expressed in parts per million.

Element	Dog Hill Quarry (n=15)		Burns Butte (n=15)	
	$\bar{X}$	S.D.	$\bar{X}$	S.D.
Th	$\bar{X}$ =	5.14	6.22	
	S.D. =	6.58	6.17	
	CV% =	128.02	99.20	
Pb	$\bar{X}$ =	41.65	23.55	
	S.D. =	3.09	1.33	
	CV% =	7.42	5.65	
Nd	$\bar{X}$ =	65.75	28.95	
	S.D. =	7.39	4.40	
	CV% =	11.24	15.20	
Pr	$\bar{X}$ =	20.11	10.25	
	S.D. =	7.32	7.42	
	CV% =	36.40	72.39	
Ce	$\bar{X}$ =	145.01	79.09	
	S.D. =	7.81	7.84	
	CV% =	5.39	9.91	
La	$\bar{X}$ =	67.85	43.13	
	S.D. =	4.69	6.46	
	CV% =	6.91	14.98	
Ba	$\bar{X}$ =	47.25	604.62	
	S.D. =	7.35	44.42	
	CV% =	15.56	7.35	
Nb	$\bar{X}$ =	34.74	24.13	
	S.D. =	4.75	2.52	
	CV% =	13.67	10.44	
Zr	$\bar{X}$ =	579.80	233.58	
	S.D. =	48.88	20.22	
	CV% =	8.43	8.66	
Y	$\bar{X}$ =	89.85	41.53	
	S.D. =	8.29	4.43	
	CV% =	9.23	10.67	
Sr	$\bar{X}$ =	2.86	25.87	
	S.D. =	2.02	2.24	
	CV% =	70.63	8.66	
Rb	$\bar{X}$ =	116.42	119.47	
	S.D. =	7.57	7.34	
	CV% =	6.50	6.14	
Ga	$\bar{X}$ =	21.90	17.15	
	S.D. =	3.30	1.82	
	CV% =	15.07	10.61	
Zn	$\bar{X}$ =	159.56	29.11	
	S.D. =	14.20	5.15	
	CV% =	8.90	17.69	
Cu	$\bar{X}$ =	5.59	9.12	
	S.D. =	10.08	7.97	
	CV% =	180.32	87.39	
Ni	$\bar{X}$ =	2.18	0.29	
	S.D. =	3.29	0.57	
	CV% =	150.92	196.55	

TABLE 3. Element sample means ( $\bar{X}$ ) and standard deviations (S.D.) by collection area at Dog Hill, Oregon. All values in parts per million.

Element	Collection Zone			
	Z2N (n=5)	Z2S (n=5)	Z3 (n=5)	
Th	$\bar{X}$	2.12	4.90	8.40
	S.D.	4.74	6.74	7.67
Pb	$\bar{X}$	40.38	42.58	41.98
	S.D.	2.90	3.15	3.44
Nd	$\bar{X}$	67.54	63.36	66.34
	S.D.	5.35	11.19	5.06
Pr	$\bar{X}$	23.08	15.36	17.90
	S.D.	11.10	2.87	8.18
Ce	$\bar{X}$	147.70	142.08	145.26
	S.D.	10.57	8.23	3.81
La	$\bar{X}$	67.02	70.92	65.60
	S.D.	3.64	5.16	4.27
Ba	$\bar{X}$	48.24	44.12	49.38
	S.D.	5.80	8.20	8.31
Nb	$\bar{X}$	31.96	36.02	36.24
	S.D.	3.31	6.18	3.89
Zr	$\bar{X}$	559.78	587.96	591.66
	S.D.	29.01	61.93	54.10
Y	$\bar{X}$	85.86	91.40	92.30
	S.D.	3.98	11.37	8.05
Sr	$\bar{X}$	3.10	3.46	2.02
	S.D.	2.28	1.64	2.22
Rb	$\bar{X}$	110.48	120.04	118.74
	S.D.	2.75	8.84	6.88
Ga	$\bar{X}$	22.14	22.54	21.20
	S.D.	3.48	2.26	4.75
Zn	$\bar{X}$	151.18	171.02	156.48
	S.D.	10.59	9.91	15.23
Cu	$\bar{X}$	3.12	11.46	2.22
	S.D.	2.10	16.78	2.18
Ni	$\bar{X}$	2.06	3.54	0.94
	S.D.	3.68	4.27	1.31

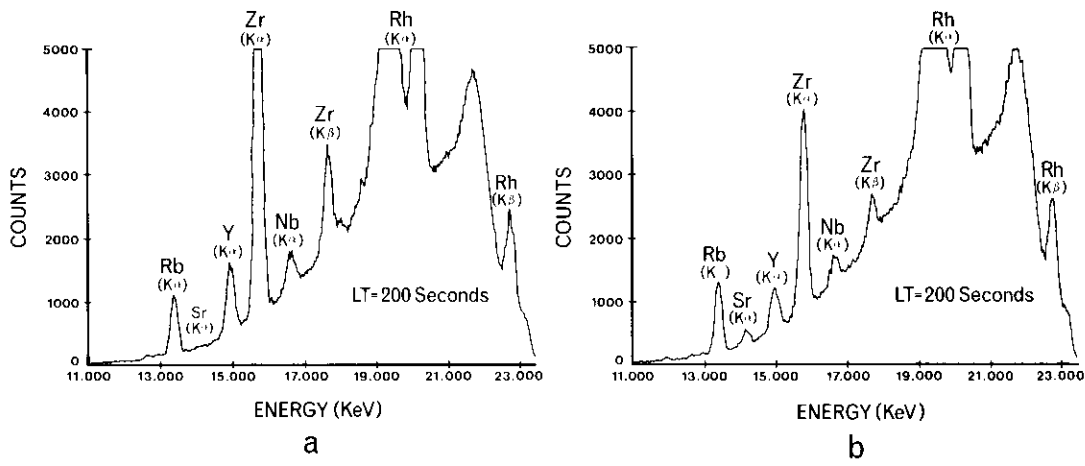


Figure 2. Energy dispersive x-ray fluorescence "fingerprint" of Dog Hill and Burns Butte volcanic glasses. a, "fingerprint" of an unmodified obsidian flake from Dog Hill (sample Z3-8); b, "fingerprint of an unmodified obsidian flake from Burns Butte (sample BB-6C).

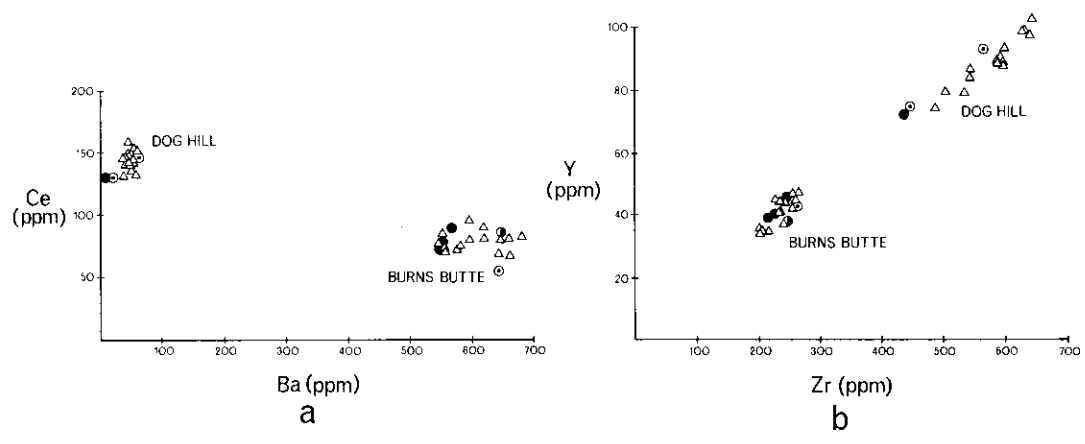


Figure 3. Scatter diagrams of Ce vs. Ba concentrations (Figure 3a) and Y vs. Zr concentrations (Figure 3b) for obsidian source specimens (open triangles) from Dog Hill and Burns Butte, Oregon. Circles represent values determined for prehistoric obsidian artifacts from archaeological sites 35CR29 (○), 35HA1300 (●) and P.P. & L. 42 (●).

chance (at the 0.05 $\alpha$  level), it can be concluded that these three areas at Dog Hill represent obsidian of the same geochemical type. As a result, measurements for all trace and rare earth elements were combined, yielding the summary statistics that appear in Table 2.

TABLE 4. Results of analysis of variance by element and collection area at Dog Hill, Oregon. Elemental data from Table 3. Critical value for  $F_{2,12} = 3.88$  at 0.05 significance level.

Element	F-ratio	P <sub>chance</sub>
Rb	3.027	0.085
Sr	0.658	0.540
Y	0.869	0.553
Zr	0.600	0.569
Nb	1.355	0.295
Ba	0.676	0.530
La	1.959	0.183
Ce	0.616	0.560
Zn	3.578	0.059

Comparison of these statistics (Table 2) shows that obsidian from Dog Hill can be distinguished from that occurring at Burns Butte on the basis of contrasts in Ce, Ba, Zr, Y, and Zn concentration. These inter-source differences are illustrated using bivariate scatter diagrams plotting Ce vs. Ba (Figure 3a) and Y vs. Zr (Figure 3b).

In order to test whether the results of the present analyses could profitably be applied to archaeological research, a small sample of obsidian artifacts from three archaeological sites in Harney and Crook Counties was analyzed and the results were compared to the trace element profiles generated for the Dog Hill and Burns Butte sources in east-central Oregon (Figure 3). As Figure 3 shows, these data fall within the range of trace element concentrations for source standards from both of these sources.<sup>3</sup> These results are of considerable interest because they document that these sources actually were exploited during prehistoric times, and that the glasses

<sup>3</sup>After the Dog Hill and Burns Butte source analyses were completed, I learned of another natural obsidian occurrence located near Dog Hill at Willow Creek (35HA914, Figure 1). X-ray fluorescence analyses of ten specimens from this source showed that its trace element composition was statistically identical to Dog Hill. Consequently, prehistoric artifacts possessing trace element compositions diagnostic of the Dog Hill geochemical type could have been fashioned from obsidian obtained either from Dog Hill or Willow Creek.

were conveyed over distances up to 100 kilometers from the parent geological sources. For example, three artifacts out of a total of 20 samples analyzed from the Beaverdam Creek site (35CR29) in the southern Ochoco Mountains (Figure 1, inset) matched the trace element configurations of Dog Hill and Burns Butte volcanic glass (Figure 3a, b). One specimen in this group, matched to the Dog Hill geochemical type, occurred in deposits 2,500-6,000 years old, while the other two artifacts were recovered in archaeological levels believed to date within the last 2,500 radiocarbon years (Erlandson and Moss 1984). Elsewhere, south of Burns Butte at the Horse Corral site (35HA1300, Figure 1) and at Sage Hen Hill, x-ray fluorescence analyses show that three artifacts were fashioned from Burns Butte obsidian, and one specimen was manufactured from obsidian of the Dog Hill geochemical type (Figure 3a, b). A single specimen analyzed from an archaeological site northwest of Burns Butte (P.P. & L. 42 on Figure 1; see Cole 1979:Appendix B, Map 26, Site 42) was fashioned from Burns Butte volcanic glass.

Although only a small number of east-central Oregon obsidian artifacts was analyzed, the results of this study clearly illustrate the potential for employing non-destructive quantitative analyses in establishing the likely parent geological source(s) for obsidian occurring in archaeological contexts. More generally, the agreement between the present analysis and previous destructive work (*cf.* Table 1) further supports the case that non-destructive analyses can yield concentration estimates in standard measurement units (*i.e.* parts per million or weight percent) suitable for interlaboratory comparisons (Hughes 1986a; see also Bieber *et al.* 1976, Harbottle 1982, Stross *et al.* 1983, Nelson 1984 for comments on the importance of using standard measurement units). The results of the present study indicate that it is now possible to use this non-destructive x-ray fluorescence technique to build a quantitative geochemical data base for Oregon volcanic glass sources and archaeological artifacts that can readily be used by researchers at other laboratories.

### Acknowledgments

I thank Joachim Hampel (Department of Geology and Geophysics, University of California,

Berkeley) and Jeanette Blomberg (Palo Alto Research Center, Xerox Corporation) for reviewing and commenting on the manuscript. Special thanks go to Madonna Moss (Mt. Baker-Snoqualmie National Forest, formerly Ochoco National Forest) and Bruce Crespin (Bureau of

Land Management, Burns District) for providing source specimens, provenience information, and for securing partial funding from their respective agencies to support this study. Tod Ruhstaller (Haggin Museum, Stockton) drafted the graphics that appear herein.

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Received 3 April 1985

Accepted for publication 15 May 1985