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X-RAY FLUORESCENCE (XRF) ANALYSIS MAJOR AND MINOR OXIDE CONCENTRATIONS OF TWO ROCK SAMPLES FROM THE BIG HOLE SITE (41TV2161), TRAVIS COUNTY, TEXAS

by

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Report Prepared for

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INTRODUCTION

The qualitative and quantitative analysis here of two sandstone samples suggests that an iron oxide was used in some way on one surface. Qualitative analysis of both surfaces indicate iron oxide on a single surface.

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).

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_2O and K_2O, and silica (SiO_2) in these rocks allows for elemental determination of rock type (Table 1 and here).

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, hawaiite; BIR-1, basalt), and one Japanese Geological Survey rhyolite standard (JR-1).

Conditions of Fundamental Parameter Analysis[1]

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

Voltage	6 kV	Current	Auto[2]
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

[1] 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.

[2] Current is set automatically based on the mass absorption coefficient.

In order to evaluate these quantitative determinations, machine data were compared to measurements of known standards during each run. SARM-69, a South African Bureau of Standards Neolithic ceramic standard was analyzed during each sample run to check machine calibration (Table 1).

Analytic Trajectory

A quantitative determination of the two rock samples and standard was the first step. Table 1 indicates that SiO_2, CaO, and Fe_2O_3 comprise the bulk of the composition of the rock, consistent with a sandstone, although the iron content is high on one side. The qualitative analysis is more enlightening (Figure 1 a-c). When both sides of the rocks were analyzed qualitatively, it is apparent that the side with reddish hue exhibited a higher concentration of iron. This does suggest that iron oxide was applied to one surface of this sandstone.

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

SAMPLE	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	TiO2	MnO	Fe2O3	Σ
	%	%	%	%	%	%	%	%	%	%	%
2396-11	1.295	2.341	9.286	34.577	3.534	1.032	36.574	0.337	0.256	10.571	99.803
2396-0	1.332	2.553	9.619	33.58	3.62	1.172	35.501	0.39	0.183	11.843	99.793
SARM-69	1.43	2.641	15.827	65.925	0	2.353	2.715	0.809	0.158	7.924	99.782

Figure 1. a = analysis of reddish side of sample 2396-0; b = analysis of non-reddish side of sample 2396-0; c = analysis of reddish side of sample 2396-11.