Title
X-RAY FLUORESCENCE (XRF) ANALYSIS OF MAJOR, MINOR, AND TRACE ELEMENTS OF IRON, COPPER, AND LEAD ALLOYED METAL ARTIFACTS FROM SEVILLETA PUEBLO AND MISSION (LA 774), CENTRAL NEW MEXICO

Author
Shackley, M. Steven

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GEOARCHAEOLOGICAL XRF LAB
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GEOARCHAEOLOGICAL X-RAY FLUORESCENCE SPECTROMETRY LABORATORY
8100 Wyoming Blvd., Ste M4-158 Albuquerque, NM 87113
USA

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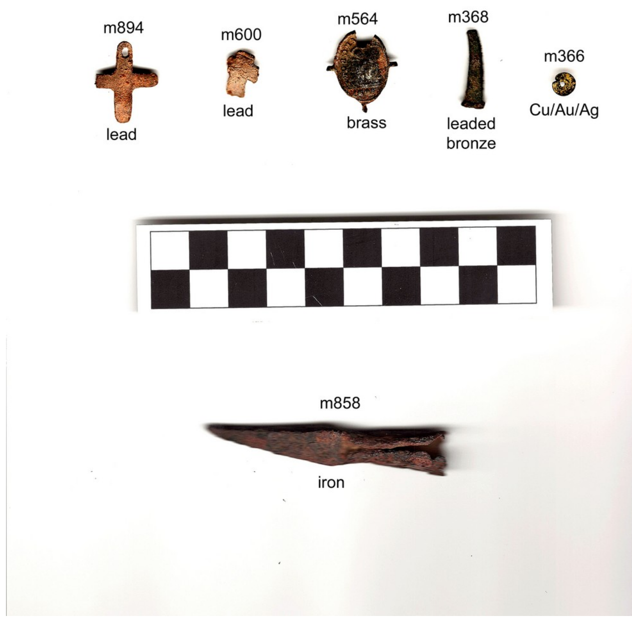

Sample of metal objects and composition

by

M. Steven Shackley, Ph.D., Director
Geoarchaeological XRF Laboratory
Albuquerque, New Mexico

Report Prepared for

Dr. Michael Bletzer

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INTRODUCTION

The analysis here of 16 metal artifacts from 17th century Spanish Colonial contexts at Sevilleta Pueblo and Mission (LA 744), central New Mexico indicates a range of metal composition from an iron crossbow bolt head to lead to copper to brass, and tin bronze, some of which are likely smelted copper objects, including jewelry (see cover image). While these results are similar to earlier studies, the XRF method/calibration was refined to more effectively analyze the number of archaeological metals analyses in New Mexico (Shackley 2017, 2018).

LABORATORY SAMPLING, ANALYSIS AND INSTRUMENTATION

Given the nature of prehistoric copper/lead production, major and minor oxides and trace elements were acquired with a method specific for metals generated for the prehistoric and historic contexts in the Southwest (http://swxrflab.net/anlysis.htm).

All the 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; Davis et al. 2011; Shackley 2011).

All analyses for this study were conducted on a ThermoScientific *Quant'X* EDXRF spectrometer, located at 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 µm (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 $l\ min^{-1}$ Edwards vacuum pump, allowing for the analysis of lower-atomic-weight elements between sodium (Na) and scandium (Sc). 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.

Metal Oxide Analysis

Analysis of the major metal specific oxides of Mn, Fe, Co, Ni, Cu, Zn, Mo, Ag, Sn, Sb, Au, and Pb is performed under the multiple conditions elucidated below. This 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 the MBH Analytical standards (see also Shackley 2011). The fundamental parameters (theoretical) method is run under conditions commensurate with the elements of interest and calibrated with 16 metal and rock standards: Mo pure, Pb pure, Cu pure, US Mint 2007 US Dollar, and US 2017 Nickel; the following copper based MBH Analytical copper based bronze and brass standards 33XGM21A3, 32XSN7A, 33XGM4AB6, 32XSN6A3, 32XSN5A1, 32XLB14F6, 32XLB10E, 331XTB5A4, 31X7835.9A5, 31X7835.8A3, and USGS DGPM-1 gold ore standard.

Conditions Of Fundamental Parameter Analysis[1]:

Mid Zb (Ag, Mo, Sn, Sb)

Voltage	30 kV	Current	Auto
Livetime	60 seconds	Counts Limit	0
Filter	Pd (0.06 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	Medium

High Zb (Mn, Fe, Co, Ni, Cu, Zn, Au, Pb)

Voltage	50 kV	Current	Auto
Livetime	60 seconds	Counts Limit	0
Filter	Cu (0.559 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	High

[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.

The data from the WinTrace software were translated directly into Excel for Windows software for manipulation and on into JMP 12.0.1 Windows for statistical analyses. In order to evaluate these quantitative determinations, machine data were compared to measurements of known standards during each run. MBH Analytical 32XLB17 leaded bronze standard was run with the samples to insure instrument accuracy (Table 1). Trace element data exhibited in Table 1 is reported in percent by weight (see also Figure 1).

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2018 X-Ray Fluorescence (XRF) Analysis Major, Minor, and Trace Elements of Metal Artifacts from Sevilleta Pueblo (LA 774), Central New Mexico. Report prepared for Michael Bletzer.

Table 1. Elemental concentrations for the archaeological specimens from LA 774 and MBH Analytical Leaded Bronze standard with recommended values. All measurements in weight percent as noted.

SAMPLE	Mn	Fe	Co	Ni	Cu	Zn	As	Mo	Ag	Sn	Sb	Au	Pb	Σ[1]	Metal
	%	%	%	%	%	%	%	%	%	%	%	%	%		
326	0.004	1.186	0.001	0.028	98.323	0	0.051	0	0.055	0	0.041	0	0.31	99.999	copper
338	0.012	0.891	0.025	0.138	52.754	11.611	0	0.051	0.048	6.287	0.032	0.057	28.095	100.001	brass
351	0	0.334	0.027	3.521	77.317	18.3	0.024	0.011	0.045	0.013	0.003	0.012	0.392	99.999	brass
353	0.011	2.204	0	0.02	97.338	0.021	0.018	0	0.03	0.005	0.042	0.003	0.309	100.001	copper
366	0.023	0.395	0.021	0.007	84.04	0	0.027	0	6.003	0.036	0.001	9.202	0.247	100.002	Cu/Au/Ag
368	0.009	0.829	0.007	0.068	78.481	1.06	0	0.037	0.064	8.31	0.161	0.016	10.958	100	leaded bronze
375	0.012	1.406	0.011	0.455	75.692	9.454	0.304	0	0.096	0.877	4.292	0.03	7.371	100	brass
382	0.052	3.149	0.006	0.015	96.59	0.063	0.006	0	0.019	0.007	0.002	0	0.09	99.999	copper
383	0.022	1.398	0	0.01	0.03	0.037	0	0	0.016	0	0.014	0	98.473	100	lead
384	0.027	1.955	0.005	0	0.06	0.019	0	0	0.372	0.006	0.707	0	96.849	100	lead
564	0.029	1.389	0.026	0.412	70.408	16.251	0	0.051	0.179	1.473	0.537	0	9.244	99.999	brass
600	0.031	1.741	0	0.011	0.015	0.02	0	0.034	0.022	0.005	0.006	0	98.115	100	lead
621	0	0.487	0	0.187	71.144	23.48	0.029	0	0.034	0.832	0.009	0.012	3.786	100	brass
858	0.075	98.747	0.004	0.398	0.539	0.041	0.163	0.026	0.001	0.002	0.001	0.003	0	100	iron
894	0.074	1.171	0	0	0.03	0	0	0.02	0.139	0.025	1.406	0	97.134	99.999	lead
900	0.099	0.679	0.004	0.008	0.568	0.001	0	0.046	0.025	19.199	22.106	0	57.266	100.001	Pb/Sb/Sn
32XLB17	0.165	0.552	0.01	0.439	75.4	0.615	1.112	0.008	0.94	5.892	4.232	0.008	10.627	100	standard
recommended	0.296	0.488	0.0083	0.465	74.83	0.634	1.51	nr	0.911	5.97	4.1	nr	9.83		

[1] Sum is normalized to 100%; nr = no report; nr = not reported.

Figure 1. Pb/Cu bivariate plot of the artifacts. Confidence ellipse at 95%. The iron crossbow bolt head contained little copper or lead (see Table 1).