Lead Shot Making Among the Sac of Northwestern Illinois:
PXRF and SEM Analyses of Metal Artifacts
From the Crawford Farm Site (11Ri81)

by

David Birnbaum, Kayeleigh Sharp, and Mark J. Wagner

July, 2015
Introduction

The development of new analytical technologies is making it possible to recover previously unknown information regarding the archaeology of Illinois through re-analysis of artifacts contained in “old” collections. By using techniques such as portable x-ray fluorescence (pXRF) and scanning electron microscope (SEM) analysis, present day archaeologists can recover information regarding the purpose of artifacts now contained in museum collections that the original excavators could not have dreamed of.

A case in point involves an enigmatic sheet copper artifact recovered from the Crawford Farm site (11Ri-81) by University of Illinois archaeologists John C. McGregor and Elaine Bluhm in the late 1950s and early 1960s (Figure 1). This site, which was located along the Rock River in northwestern Illinois, represented the remains of a major Sac village (Saukenauk) dating to the late eighteenth and early nineteenth centuries once occupied by the famous Sac leader Black Hawk (Bluhm 1961; Woodbury 1961, 1962). The U of I archaeologists excavated hundreds of pit features and almost a dozen houses at the site before it unfortunately was destroyed by highway construction. As such, the artifacts recovered by Bluhm and McGregor represent the largest source of information regarding Sac lifeways at this now-vanished major site.

The Illinois Department of Transportation (IDOT) is currently funding the Center for Archaeological Investigations (CAI) to complete the report of investigations for the Crawford Farm site. As part of this agreement the Illinois State Archaeological Survey (ISAS) has loaned the Crawford Farm site artifacts and records to the CAI for study.

The roughly rectangular (ca. 10 cm by 7 cm) sheet copper fragment appears to have been cut from the bottom of a kettle (Figure 2). The Sac perforated a ca. 6 cm by 5 cm area of this item with 44 holes arranged in at least seven rows, all of which were punched in from one side. One of the rows was partially removed when this item was cut from the kettle, suggesting that it originally contained about 50 holes.

The 1960s archaeologists interpreted this item as being a scrap from a broken kettle. In looking at the item, however, we were struck by its resemblance to modern copper “sieves” for making small diameter (BB size or less) lead shot. When molten lead is poured into such a sieve it forms lead drops that solidify into round pellets as the lead drips into a container of water held below the sieve. We also noticed that a white residue that could represent lead was present around the edges of at least nine of the perforations in the possible sieve.

The Sac extensively mined lead along the upper Mississippi River both for their own use and for trade to Europeans in return for manufactured goods (Millhouse 2010:318-357). Lead also is ubiquitous in the Crawford Farm collection, occurring in the form of thousands of pieces of raw lead, melted lead,
slag, and finished objects recovered from pit feature and house contexts. In addition, stone molds for the production of lead musket balls also form part of the Crawford Farm collection indicating that the Sac clearly made large shot for their own use. But could they have been producing small shot as well using sieves made from copper kettles?

We decided to test this idea by using both pXRF and SEM analysis to see if we could identify lead residue on the possible shot sieve. In addition, in order to allow us to compare the elemental composition of artifacts that are wholly lead or copper and objects that contain both chemical signatures we analyzed 12 copper kettle scraps with discolored surface areas representing possible lead staining (Figure 3); 48 lead items including drop and tumbled shot sprue, and raw lead ore; and what appears to be a possible iron ladle bowl with a missing spout (Figure 4). The possible ladle bowl has a hole in the center, which suggested that it also possibly could have been used in dripping lead for making shot or some other purpose.

Methods

PXRF spectrometry allows for the non-destructive, point-focused analysis of *in situ* objects or artifacts in museum or laboratory collections. The PXRF instrument uses a beam of short wavelength, high-energy photons to induce the emission of longer wavelength, lower energy “characteristic lines” from the sample being analyzed. The characteristic lines of specific elements can be isolated since the relationship between emission wavelength and atomic number is known. Specific elements can be identified and elemental concentrations can be estimated from the intensity of the characteristic lines (Ioannis and Zacharias 2012). We conducted our analysis using a Bruker Tracer III-V (SD) Portable X-Ray Florescence (PXRF) instrument (Figure 5). In order to measure heavier trace elements such as lead, the pXRF analysis was performed with the “Red Filter” (0.001” Cu, .001” Ti, .012 Al) without a vacuum, with the instrument operating at 40 kV and 35μA. Each sample was analyzed for 90 seconds, though some artifacts were analyzed more than once in order to test whether visible surface discolorations represented lead residue.

In addition, a Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray Spectroscopy (EDS) analysis was performed to investigate the lead residue around the periphery of the holes of the copper sieve to see if the results agreed with those of the PXRF analysis. An SEM technician conducted these analyses with the authors present at the IMAGE Laboratory at Southern Illinois University, Carbondale (Figure 6). This involved using an FEI Quanta 450 FEG (Field Emission Gun) SEM instrument equipped with an Oxford X-max 50 mm EDS detector. The samples were analyzed under low vacuum conditions at an accelerating voltage of 20 kV.
Results

The possible shot sieve was analyzed multiple times (N=15) using the pXRF instrument on both surfaces in order to determine whether the white staining around the periphery of the holes represented lead (Figure 5). Most of the analysis points returned a copper signature, but the pXRF readings that targeted the holes with white residue returned very strong lead signatures. This suggested that the presence of lead was restricted to a particular area of one surface (presumably the interior of the kettle) through which molten lead was poured during the production of drop shot. The SEM results of this study (Figure 6) confirmed what the PXRF analysis showed, that is, that the white discoloration is in fact lead residue and the holes without white discoloration do not contain lead. Why the lead did not adhere to all of the sieve holes is puzzling. Perhaps the object was periodically cleaned between making batches of drop shot or most of the lead simply eroded off of the surface after the object entered the archaeological record. In addition, the SEM images showed that the holes in the sieve were roughly square in shape (Figure 7) rather than round. This suggesting that they had been punched through the kettle base using a square-bodied iron object such as a square nail or possibly the narrow end of a rat-tailed file.

We analyzed the 14 copper kettle fragments with discolored surfaces (Figure 3) twice, taking a basic elemental signature from an unstained area of the object with the second reading targeting a lead signature from the discolored surface. Two of these readings returned a lead signature while the other 12 were negative for this mineral. This suggests that other post-depositional processes in addition to melted lead caused discoloration and corrosion of some kettle surfaces at the Crawford Farm site while they were in use. Nevertheless, the presence of lead on the surfaces of two of the fragments suggests that they also may have formed parts of containers or sieves for melted lead.

The possible iron ladle bowl (Figure 4) is identical to that of a ladle for pouring melted lead shown in a book on Revolutionary War artifacts (Neumann and Kravics 1989:189). The presence of a hole in the bowl center suggested to us that it may have been repurposed for dripping lead into water or a mold. PXRF analysis of the interior and exterior surfaces of the Crawford Farm object failed to detect a lead signature, however, leading us to conclude that this particular item was used for some other purpose other than as a lead container. This is not a conclusion that we would have reached without the pXRF analysis.

Summary

The most exciting result of this study was the confirmation of lead residue through both pXRF and SEM analysis on the holes of the copper shot sieve (Figure 2). Although the production of small lead shot through use of sieves clearly was within the technological capability of the late eighteenth to early nineteenth century Sac this represents the first time, to our knowledge, that such a shot-making sieve has been identified from a native-occupied site in the lower Great Lakes region. We believe that the sieve consisted of a copper kettle into
which the Sac poured molten lead that then dripped through the holes in the bottom of the kettle into a container of water to form lead shot. The Sac apparently later recycled this kettle, using the sides as raw material for the manufacture of items such as tinklers, hair pipes or beads while discarding the unusable perforated base with its numerous holes.

The identification of the copper sieve also points out the important role that universities and museums play in maintaining archaeological collections from important sites such as Crawford Farm so that they can be studied by future researchers using modern scientific techniques such as the ones in this case that were unavailable to the original excavators. Especially in the case of sites such as Crawford Farm that no longer exist, the ability to revisit old collections using techniques such as pXRF and SEM analysis can result in the recovery of significant new information that adds to our knowledge of both Native and European lifeways within the state.

Acknowledgements

We would like to thank Dr. Tom Emerson and the staff of the Illinois State Archaeological Survey (ISAS) for making the Crawford Farm site collection available to us; the SIU Geology Department for allowing us to use the pXRF machine housed in that department; and the staff of the IMAGE laboratory at SIUC for conducting the SEM analysis.
Bluhm, Elaine A.  
1961  The Historic Indian Occupation of the Rock River Area in Illinois.  

Millhouse, Phillip  
2010  Native American Lead Mining in the Galena River Valley and the Potential for Archaeological Research.  

Neumann, George C. and Frank J. Kravic  

Woodbury, Natalie F. S. (Editor)  
1961  Notes and News.  

1962  Notes and News.  
Figure 1. Crawford Farm Site Investigations, 1959
Figure 2. Reverse of possible copper sieve showing white residue
Figure 3. Copper kettle scrap with residue.
Figure 4. Possible iron ladle bowl, Crawford Farm site
Figure 5. Bruker Tracer III-V (SD) pXRF analysis of possible sieve.
Figure 6. Kayeleigh Sharp (left) watching SEM analysis of possible sieve at SIU IMAGE lab.
Figure 7. Close up SEM image of copper artifact showing sieve hole with lead (pb) residue