SCIENTIFIC RESEARCH

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# JOURNEYS OF OUR ANCESTORS: INTERDISCIPLINARY RESEARCH AND ITS INFLUENCE ON CONSERVATION METHODOLOGIES FOR THE ASSESSMENT OF ARCHAEOLOGICAL ARTIFACTS

**Keywords:** X-ray fluorescence, portable, non-destructive, archaeology, ceramics, coins, conservation science

## ABSTRACT

This paper will discuss the interdisciplinary research project associated with the preparation for an exhibit focused on migration in the American Southwest. Exhibit content highlights the traces of migration in archaeological and historic contexts ranging from the Paleo-Indian period (12,000–13,000 BP) to the 19th century development of the railroads. Collaborative exhibit research efforts are predicated on the cultural importance of selected artifacts and offer an opportunity to evaluate the interpretive value of portable X-ray fluorescence (pXRF) data in the analysis of archaeological metal and ceramic materials including Asian cast metal coins dating to the 17th–20th centuries, prehistoric ceramic colorants used on Southwest low-fired ceramics, and post-depositional products found on low-fired ceramics. The three case studies serve to demonstrate the importance of conservation science approaches to the analysis, interpretation, and preservation of archaeological artifacts and museum objects.

## RÉSUMÉ

Cet article discutera du projet de recherche interdisciplinaire associé à la préparation d'une exposition sur les migrations dans le sud-ouest américain. Le contenu de l'exposition met en valeur les traces de migrations dans des contextes archéologiques et historiques allant de la période paléo-indienne (12 000–13 000 BP) à l'émergence du chemin de fer au XIX[e] siècle. Les initiatives de recherche en collaboration pour l'exposition sont fondées sur l'importance culturelle des objets sélectionnés. Elles offrent l'opportunité d'évaluer la valeur interprétative des données de fluorescence X portable dans l'analyse du métal archéologique et des matériaux céra-

## INTRODUCTION

Conservators and conservation scientists frequently undertake analysis and interpretation of disparate materials/collections tied together by research questions directed by outside influences. These include preservation problems requiring action; curatorial research interests; museum exhibition programs; and issues identified by other cultural heritage stakeholders. The scope of conservation science practice is necessarily broad, requiring an "incremental and iterative approach" (Cather 2006, 90). This produces meaningful research where the application of technical study and instrumental analyses support ethical practice. This takes into consideration conservation of tangible (scientifically measurable) and intangible (cultural significance/meaning) forms of data, as well as associated cultural stakeholders (Odegaard 1995). Object preservation incorporates long-term conservation of the object, its cultural meanings, associated analytical data, and present/future research potential.

Conservation science research utilizes compositional data to connect material properties and manufacturing methods to condition, degradation and appropriate preservation practice. By relating cultural material properties, structure and manufacturing through the lens of culture, conservation science offers nuanced research through interpretation of archaeological, cultural, curatorial, conservation and scientific data. This method can be utilized at any point in the lifecycle of an object or collection. Both non-destructive and destructive analytical tools play critical roles allowing multiple lines of evidence to eliminate and/or minimize utilization of destructive analyses. As non-destructive instrumentation including portable X-ray fluorescence (pXRF) becomes standard in museum laboratories, conservation science methodologies must play a role in ground-truthing the technique for use with cultural materials.

The relative ease of using pXRF belies the complexity of meaningfully interpreting data collected from cultural artifacts characterized by heterogeneous compositions, structures, and density with altered or degraded surfaces. Familiarity with the scientific caveats and limitations associated with XRF theory and pXRF instrumentation (designed for homogeneous, flat materials with consistent instrument/sample geometry) is just as critical for analysis of cultural artifacts (Dussubieux et al. 2005), as sensitivity to preservation ethics. The presented case studies utilize pXRF instrumentation,

miques, dont des pièces de monnaie asiatiques en métal moulé des XVII[e]–XX[e] siècles, des colorants de céramique préhistoriques employés sur des céramiques cuites à basse température dans le sud-ouest américain, et des produits post-sédimentaires trouvés sur des céramiques à basse cuisson. Les trois études de cas démontrent l'importance des approches scientifiques de la conservation-restauration pour l'analyse, l'interprétation et la préservation des pièces archéologiques et des objets de musée.

## RESUMEN

Este artículo analiza el proyecto de investigación interdisciplinaria asociado a la preparación de una exposición enfocada en las migraciones del suroeste de Estados Unidos. El contenido de la exposición destaca las huellas de la migración en contextos arqueológicos e históricos que discurren desde el periodo Paleoindio (12.000–13.000 a.C.) hasta el desarrollo del ferrocarril en el siglo XIX. Se predican los esfuerzos de colaboración para la investigación de la exposición en relación a la importancia cultural de algunos objetos seleccionados, lo cual ofrece una oportunidad para evaluar el valor interpretativo de datos de la fluorescencia de rayos X portátil (pXRF, en sus siglas en inglés) en el análisis de materiales arqueológicos metálicos y de cerámica, incluyendo monedas asiáticas de metal fundido de los siglos XVII al XX, colorantes cerámicos prehistóricos usados en cerámicas de baja cocción en el Suroeste, y productos de post-deposición encontrados en cerámicas de baja cocción. Los tres estudios de caso sirven para demostrar la importancia de los acercamientos de la ciencia de la conservación en el análisis, la interpretación y la preservación de artefactos arqueológicos y objetos de museos.

as well as necessary destructive analyses, to scientifically confirm the value of produced data. This methodological approach informs preservation practice through proper interpretation of object manufacturing/production practice and post-discard changes due to decomposition or repair.

## METHODS

Objects from Arizona State Museum, University of Arizona (ASM) collections were analyzed to determine the efficacy of pXRF analysis for the interpretation and preservation of museum objects. The presented case studies are designed to reflect typical material structures that contribute and/or affect object preservation and interpretation including: (a) samples characterized by specific microstructures; (b) layered structures where analytical sampling volumes incorporate multiple layers of similar composition; and (c) the contribution of post-depositional changes to the measured composition of an object. Using standard protocols and a Bruker TRACER III-V Handheld XRF with rhodium tube source, pXRF data was collected for 180 seconds with two operation settings: (a) 15 keV accelerating voltage and 10 µA current under vacuum for low-Z elements and (b) 40 keV accelerating voltage and 1.5 µA current without vacuum for high-Z elements. No primary or secondary filters were used during analysis. Where possible, data was collected from multiple locations in order to sample all visible surface characteristics, as well as characterize the influence of sampling volume, microstructure and differential preservation on data reproducibility. Data was corrected using Compton scattering methods and calibrated, where feasible, using standard reference materials of known composition.

PXRF use is predicated on the scientific understanding of the relationship between elemental composition and sample microstructure, as well as analytical sampling volume. This is critical for data interpretation, used to reconstruct original methods of manufacture including alloy cooling rates, current state of preservation, and identification of past curatorial or conservation interventions. Destructive sampling, undertaken in limited numbers where permitted by museum committees or by private collections, was necessary to test the precision, accuracy and interpretive value of collected pXRF data for Case Study 1 samples. Selected metallographic samples, reflecting the range of observed compositional variation based on condition and preserved mint/date information, were imaged using reflective light microscopy in (a) as-cast condition and (b) following acid etching (Scott 2002) using an Olympus BH-2 Research Microscope.

PXRF analytical sampling volumes were estimated using electron microprobe analysis of a cross-sectioned non-museum sample reflecting typical coin compositions in the study. Data was collected using a Cameca SX-50 Electron Microprobe equipped with a wavelength-dispersive, integral Bruker SDD Energy Dispersive Spectrometer system. Data was collected for 40 seconds every nine microns for selected elements of interest (10 seconds on background, 20 seconds on peak, 10 seconds on background) at an accelerating voltage of 15 keV, 100 nanoamps and a takeoff angle of 40°. Scanning electron microscopy

(SEM) and energy dispersive spectrometry (EDS) analyses were utilized to collect images and elemental data in Case Study 2 using a Hitachi S-2460 with Tungsten thermionic emitter Hitachi S-2460/ThermoNORAN NSS EDS: Variable Pressure SEM. Non-conductive samples were carbon coated using an Edwards Turbo Molecular Pumped Vacuum Evaporator E306A – High vacuum unit. In Case Study 3, pXRF analysis was compared to an already established morphology of accretion forms in order to characterize and interpret post-depositional changes to archaeological samples.


**Figure 1**
Typical Chinese banliang coin (ASM A-49181)

## CASE STUDY 1

Chinese coins (n = 121), in ASM collections, were analyzed to reconstruct original manufacturing processes and understand preservation through reconstruction of artifact life following discard and deposition. The coins, excavated in Tucson during the 1960s and 1970s, predominantly date to the Qing dynasty (AD 1644–1911) and Chinese Republic (AD 1911–1949). Coins were selected to determine the efficacy of non-destructive pXRF instrumentation through comparison to metallographic analyses in order to relate elemental composition, sampling volume, material microstructure and corrosion. Interpretation of analytical results is combined with pertinent archaeological, conservation and curation data to provide valuable information regarding the manufacturing technology and raw materials used during coin production.

Analyzed coins exhibit differential preservation and are generally standard banliang format (n = 101) (Figure 1), while the remaining examples include struck coins dating to the 20th century (n = 17) (Tables 1 and 2). Coins are predominantly Chinese, but Japanese (n = 11) and Vietnamese (n = 1) issues were investigated. Previously, coin mint date and mint location, located on coin obverse and reverse respectively, were identified based on translation of preserved characters (Olson 1983). Coin compositions vary, but are generally brass and incorporate two basic types based on metallographic analysis: α brasses (zinc < 35%) and α + β (35% < zinc < 46%) brasses. Several Fe-cast coins were identified including three Japanese issues, as well as a single high-zinc coin produced in Vietnam. The addition of lead in coin microstructures, a common addition for objects necessitating rapid solidification and low tensile strength, is extremely variable.

**Table 2**
Qing Dynasty timeline by emperor reign period

| Qing Emperor | Reign Period |
|---|---|
| Shun Zi | 1644–1662 |
| Kang Xi | 1662–1722 |
| Yong Zheng | 1722–1735 |
| Qian Long | 1735–1796 |
| Jia Qing | 1796–1821 |
| Dao Guang | 1821–1851 |
| Xian Feng | 1851–1862 |
| Tong Zhi | 1862–1875 |
| Guang Xu | 1875–1908/1911 |

**Table 1**
Analyzed coin samples sorted by coin type and period

| Coin Type | Reign Period | Number of Samples |
|---|---|---|
| Qing – banliang type | 1644–1911 | 59 |
| Qing – standard issue | 1644–1911 | 17 |
| Chinese Republic – standard issue | 1911–1949 | 3 |
| Chinese – illegible/unknown | 1644–1911 | 14 |
| Japan – Edo Period | 1603–1868 | 9 |
| Japanese – unknown | unknown | 2 |
| Vietnam | unknown | 1 |
| Asian – unknown | unknown | 16 |
| **Total Coins** | | **121** |

Coin cooling and solidification rates are reconstructed elsewhere (O'Grady 2009, 174–181, Appendix A), but summarized here. In general, samples (Figure 2) are characterized by no observable dendrite formation suggesting cooling and solidification rates were sufficiently quick to prevent their formation. In some cases, Widmanstätten (geometric lattice) structures are visible suggesting rapid cooling in air. Only two samples show coldworking and possible annealing suggested by minimal grain twinning. This is probably related to significant iron and lead impurities introduced due to recycling or coin debasement.

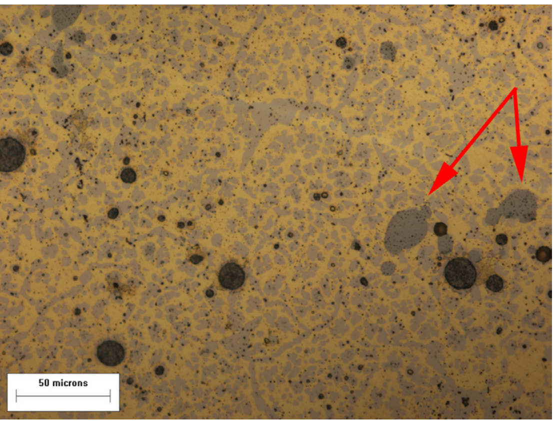


**Figure 2**
Typical metallographic microstructure of α brass coin with α phase (light grey) in β solution (yellow) and grey lead globules (indicated by arrows) (ASM A-49205). Circular losses due to corrosion are also visible

An approximate estimate of X-ray beam penetration depth, determined through Monte Carlo electron experiments, indicates predicted sampling volumes for α and β grains are smaller than the minimum dimensions of either grain (O'Grady 2009, Figures 4.13, 4.14). As pXRF spectra are produced by substantially larger beams that sample preferential and non-representative volumes, this factor is critical to take into account when interpreting data.

Despite these caveats, pXRF data is successfully able to differentiate discrete coin compositions, when interpreted with pertinent archaeological, linguistic and conservation information. Alloy ratios are used to identify specific compositional groups, a technique utilized by Lin et al. (1991). Generally, copper/zinc and copper/lead ratios are consistant throughout the Qing dynasty suggesting good control of coin manufacture – though variation within a reign suggests production changes. Comparison of copper/lead ratios (Figure 3) discriminates three compositional groups reflecting temporal changes in composition or varying degrees of coin debasement. While insufficient to differentiate coins by individual reign date or production country, copper/lead ratios can discriminate coin production between regional mint locations. High copper/lead ratios are associated with southern Chinese mints during the late Chinese Republic, while low ratios with Bejing mints (Cowell and Wang 2005, 86).

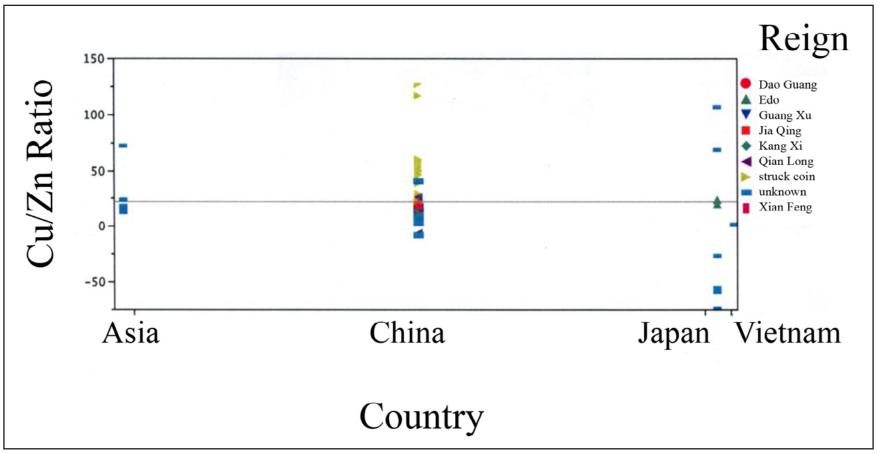


**Figure 3**
Calculated pXRF copper/lead ratios for analyzed coins

Integrated analysis of Chinese coins, using non-destructive and destructive methods, provides information regarding composition and manufacture that informs preservation practice. Metallographic analyses of coin microstructures provide integral information regarding coin production methods that cannot be discerned from visual analysis. Generally, metallographic microstructure confirms pXRF data and corrosion does not appear to significantly affect pXRF data results.

## CASE STUDY 2

The use of ceramic colorants is culturally specific in the American Southwest and reflects development of unique manufacturing traditions (Shepard 1968). Archaeological ceramic colorants, present on ceramics in ASM collections, are investigated using scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS), as well as pXRF to reconstruct technology and culture, and a nuanced understanding of the development/transfer of technology across the landscape. Data interpretation informs assessments of current condition/degradation through elucidation of original materials and methods used during manufacture. Given the extensive body of existing archaeological and scientific data, Southwestern low-fired ceramics offer an opportunity to test the efficacy, reliability and precision of pXRF analysis of ceramic colorants.

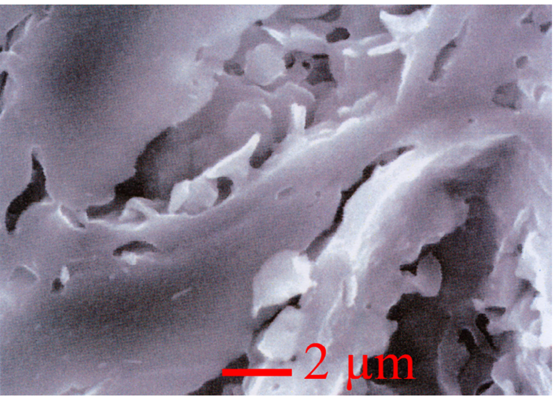


**Figure 4**
Typical Sikyatki Polychrome high-fired ceramic with glassy microstructure (private collection: 310-3 HPQ 409), 5000x magnification

The manufacturing technology for Sikyatki Polychrome, a coal fired Hopi Yellow Ware ceramic (1375/85–1625), was determined in order to characterize production of its yellow surface colorant (Colton and Hargrave 1937, 153). Samples from a private collection were analyzed to determine particle size, microstructure and characterize paint compositions, as well as estimate firing temperatures. Fresh fracture samples were refired for 15 minutes at 50° temperature intervals starting with 700°C and ending with 1300°C. Refired and unmodified ceramic samples were imaged and analyzed using SEM-EDS.

Samples of Sikyatki surface and paint colorants are dense, well-compacted and characterized by particle rounding suggesting sintering that developed due to high temperatures achieved during firing. Paint thicknesses vary depending on application and paint composition. Surface colorants contain small quantities of sulfur suggesting deposition during coal firing, while paints are characterized by iron-, manganese- and mixed compositions (O'Grady 2009, 244). The presence of plant structures within painted layers also suggests the use of organic paints (O'Grady 2009, 250).

Experimental firing tests of sherd samples confirmed use of high-firing manufacturing processes during Sikyatki production. The microstructure of the highest and second highest firing groups did not change until exposed to temperatures of 1200°C (Figure 4). Sample microstructures from the two lowest firing groups (third and fourth) did not change structure until they were exposed to temperatures greater than 1050°C and 1000°C respectively. Efforts to confirm the presence of mullite in analyzed samples, an alumina-silicate mineral that begins to form around 1000°C and crystallizes above 1250°C, were not successful and further analysis is necessary.

While reconstruction of Hopi Sikyatki polychrome manufacturing technology is incomplete, data results suggest Hopi potters exercised considerable control throughout all production steps. They consistently and reliably produced the culturally specific surface and paint colorants characterizing this highly-valued and traded ceramic ware for over 300 years.

Point of Pines Polychrome ceramics, including ASM jar (A-15491) dating to ca. AD 1300–1400 (Carlson 1970, 79) (Figure 5), represent the introduction of glaze-paint ceramic traditions associated with a Kayenta Anasazi migrant group. Excavated from a Point of Pines site (Carlson 1970, 81) in eastern Arizona, the low-fired jar was manufactured using local materials, but replicates styles foreign to the local population. References in the literature visually identify the black glaze-paint as a copper derivative based on areas of preserved green oxidation, but there are no published scientific data confirming this hypothesis.


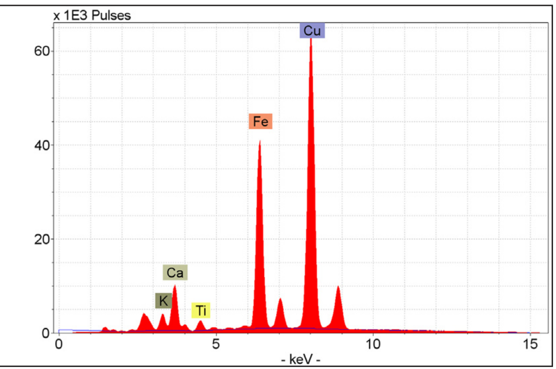


**Figure 5**
Point of Pines Polychrome jar (ASM A-15491)

**Figure 6**
Point of Pines Polychrome jar (ASM A-15491) pXRF spectrum with identified peaks

PXRF analysis of preserved Point of Pines Polychrome black colorants identifies substantial copper and iron peaks (Figure 6), but no lead. Black glaze-paint compositions associated with Ancestral Pueblo and Zuni ceramics generally include lead, or lead with copper (De Atley 1986, Huntley 2008). The absence of lead suggests low firing temperatures were achieved using salt-organic, alumina and silica fluxes. This composition is unique, as copper glaze-paints are not reported in the literature. In this case, locally developed technologies are used to produce foreign ceramic designs reflecting the effect of culture on manufacturing technology (O'Grady 2009, 267). More data is needed to determine the range of glaze-paint compositional variation in Point of Pines ceramics, as well as temporal changes in raw materials affecting production practice and colorant composition.

These examples demonstrate the use of destructive (SEM-EDS) and non-destructive analytical techniques for the interpretation and preservation of archaeological ceramics through the identification of original manufacturing technologies. Within the correct interpretive framework, pXRF facilitates characterization of technical information, while preserving intangible cultural components. This enables conservation scientists to better target the number and type of samples needed for destructive sampling, as well conservators to successfully integrate accurate reconstruction of manufacturing technologies into condition assessment.

## CASE STUDY 3

Archaeological artifacts are complex and have heterogeneous compositions that exhibit deterioration layers and post-depositional alterations. Efforts to properly interpret deterioration and its effects on object composition are critical towards artifact preservation through accurate reconstruction of original materials, manufacture and post-discard changes. This case study focuses on the analysis of the post-depositional product known as manganese dioxide accretions and its interaction with archaeological ceramics through analysis of 1063 ceramics in ASM collections. Excavated and collected during the first half of the 20th century, the collection consists of ceramics produced by the Casas Grandes culture (AD 8th–15th centuries) in Chihuahua, Mexico.

For this case study, manganese dioxide accretions will be used to describe manganese and iron alteration products on ceramics, as various terms are used in the literature. The phenomenon is not well described on cultural artifacts due to its complex composition and mechanism of formation (Aronson and Kingery 1991, Watkinson et al. 2005). Recent work has identified five morphological classes on archaeological ceramics, easily identifiable without magnification (O'Grady 2005a, 2005b). SEM-EDS analysis confirms their varied elemental composition consists of ratios of manganese, iron, titanium, potassium and calcium, as well as provides evidence that accretions penetrate through paste and slip during its formation (O'Grady 2005a, 2005b). When data interpretation is conducted using an established morphological typology (O'Grady 2005a, 2005b), pXRF is an excellent survey tool capable of identifying the contribution of post-depositional changes to measured paste, slip and paint compositions.

**Figure 7**
Detail of typical manganese dioxide accretions found on Casas Grandes ceramics (Carretas Polychrome Jar ASM GP38556)

Manganese dioxide accretions (Figure 7) are documented on 71.4% of the entire collection (O'Grady 2005b). Observed accretion morphologic forms include dendritic, circular mass, chunky/polycrystalline and layered classes. Accretions with distinct nucleus of formation are only observed on dense substrates and typically penetrate into the ceramic matrix (O'Grady2005b). Accretions are found forming preferentially over manganese-based paints and along tidelines suggesting possible formation mechanisms that rely on the interaction between solid/liquid interfaces where pH and capillary action play a role (O'Grady 2005a, 2005b, Watkinson et al. 2006). Individual paint, substrate and post-depositional accretion compositions were confirmed by pXRF (Figure 8) through comparison of manganese and iron peak heights and volumes.

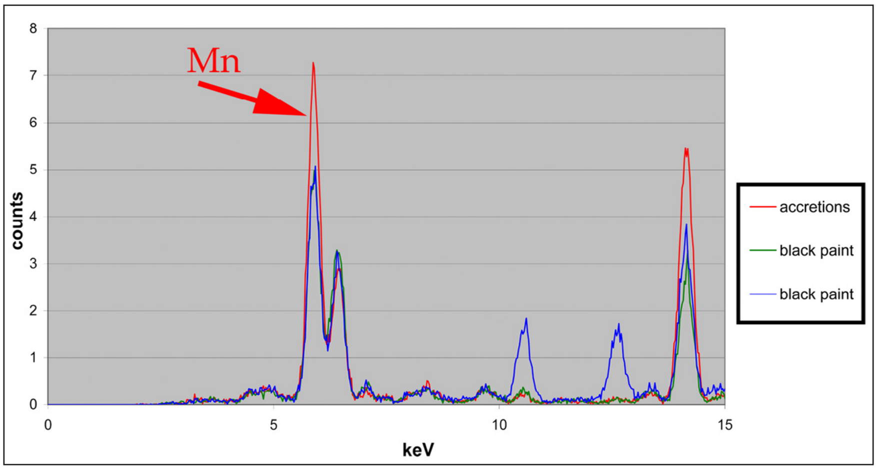


**Figure 8**
Babicora Polychrome Jar (ASM GP38565) pXRF spectrum: red arrow points to manganese peak

The data presented in this case study confirms the important physical and chemical relationships that exist between artifact materials, environmental conditions and deterioration mechanisms. Analysis of post-depositional products is critical for the interpretation and conservation of archaeological materials. Excavated archaeological objects are complex materials with heterogeneous compositions that exhibit use-wear, deterioration layers, post-depositional products, and/or evidence of curation interventions. Separation of these components is essential for the analysis of artifact composition, method of manufacturing and identification of burial environments and deterioration mechanisms responsible for current object condition. Non-destructive pXRF analysis is used successfully to identify post-depositional changes including manganese dioxide accretions, as well as ensure object preservation through the direct relationship of visual and chemical characteristics to specific deterioration mechanisms and burial environments.

## CONCLUSION

Conservation science research of cultural heritage materials has its foundation in preservation of the object, its research and cultural values, as well as preservation ethics. The introduction of non-destructive analyses, including pXRF, has revolutionized the manner in which conservation science research is formulated and directed. The adoption and use of standard data collection, correction, calibration and interpretation parameters is paramount for producing measurements of interest that inform cultural heritage research and object preservation. Case study research questions are answered by correlating materials properties, structure and manufacturing processes to cultural relationships between artifact production and the contribution of object use, discard, degradation, curation and conservation within the context of technical study and conservation science ethics.

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