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Use of P-XRF Spectrometers for Pesticide Detection: a Case Study of Navajo Textiles Treated with Arsenic



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Abstract

The development of a protocol to test for significant presence of arsenic-based pesticide residues was undertaken using an Olympus Innov-X Delta Professional Portable X-ray fluorescence (pXRF) spectrometer on a significant textile collection at Arizona State Museum (ASM). The non-destructive / non-invasive technique was utilized due to its handheld movability, fast detection and differentiation for a broad range of elements, and internationally adopted by the field of museum conservation as a viable scientific tool in testing cultural materials. Preparing a range of known contaminated arsenic-based wool samples resulted in instrumentation calibration for confident levels of arsenic-based residues. Assuring confident levels of arsenic detection with a pXRF spectrometer initiated a protocol to safely and efficiently test the textiles.

Keywords: Navajo textiles; pesticides; Museum conservation; arsenic; Spectrometer; History; Pesticides; Herbicides; Fungicides; Mercury; Lead; Arsenic; Collectors; Curators; Researchers

Introduction

After a history of use in environmental conservation on the analysis of soils and sediments, the p-XRF spectrometer or analyzer has become a powerful instrument in museum conservation. One important use is in the detection of toxic metals found in pesticide residues such as arsenic (As), lead (Pb), mercury (Hg), and zinc (Zn). In the past, pesticides, herbicides, and fungicides were commonly used to prevent, destroy, repel or mitigate pests to preserve museum collections. Thus, museum professionals are interested in the chemical analysis of potentially treated material cultural items, because they want to know what a substance is composed of (qualitative) and how much is present (quantitative) [1]. Its use for detection of toxic metal pesticide residues has evolved from sealed radioactive sources, which depend on the half-life of the source, to the use of x-ray tube sources. The newer instruments can detect and differentiate a broader range of elements, including lighter elements, with improved analytical software that allows for batch processing of data. The pXRF instrument is capable of being calibrated using given elemental concentrations vs. calculated elemental concentrations, termed empirical calibration [2]. An award from the National Center for Preservation Training and Technology (NCPTT) funded the purchase of an up-to-date x-ray tube source pXRF analyzer, to develop calibration standards, and to test a collection of 430 Dine' (Navajo) textiles.

p-XRF Calibration goals

A handheld p-XRF instrument's "detection limits are a function of testing time, sample matrix, and the presence of an interfering element" [3]. The Olympus Innov-X Delta pXRF is equipped with a silicon drift detector (SDD) that converts X-rays emitted from atoms into electric signals producing a limit of detection of <5ppm for low-density arsenic samples (i.e. soils, powders, and liquids). Thus, a one-to-two-minute test results in a detection confidence of 99.7% [4].

Given no available industry reference standards for arsenic in wool, the need to develop empirical calibration standards for a p-XRF spectrometer was essential for assured wool textile arsenic analysis. On startup, an initial calibration check procedure was conducted against an alloy 316 stainless steel coupon to determine a variety of parameters are within factory pre-set tolerances. Following p-XRF startup procedures, additional measurements were conducted on an uncontaminated wool control sample and other potential interfering elements, such as underlying testing surfaces, to verify negative presence of arsenic.

Materials and Methods for P-XRF Calibration

The p-XRF calibration method was a sequential series of steps starting with an investigation into the properties related

to Navajo textiles. This led to preparing 15 Chimayo wool test samples to be homogeneous contaminated with five known arsenic concentrations (5000ppm, 2500ppm, 1000ppm, 500ppm, and 100ppm) and tested with the p-XRF instrument. Each concentration set consisted of three samples dipped into one arsenic solution, air-dried, and p-XRF tested in five different

areas to ensure uniformity and quality of concentration. Each p-XRF reading was conducted in the manufacturer's "soil" mode for a period of 90 seconds with beam settings at 40kV for 60 seconds and 15kV for 30 seconds. A p-XRF arsenite (As III) calibration curve for dry wool p-XRF readings resulted in a linear regression of 99% as shown in (Figure 1) [4].

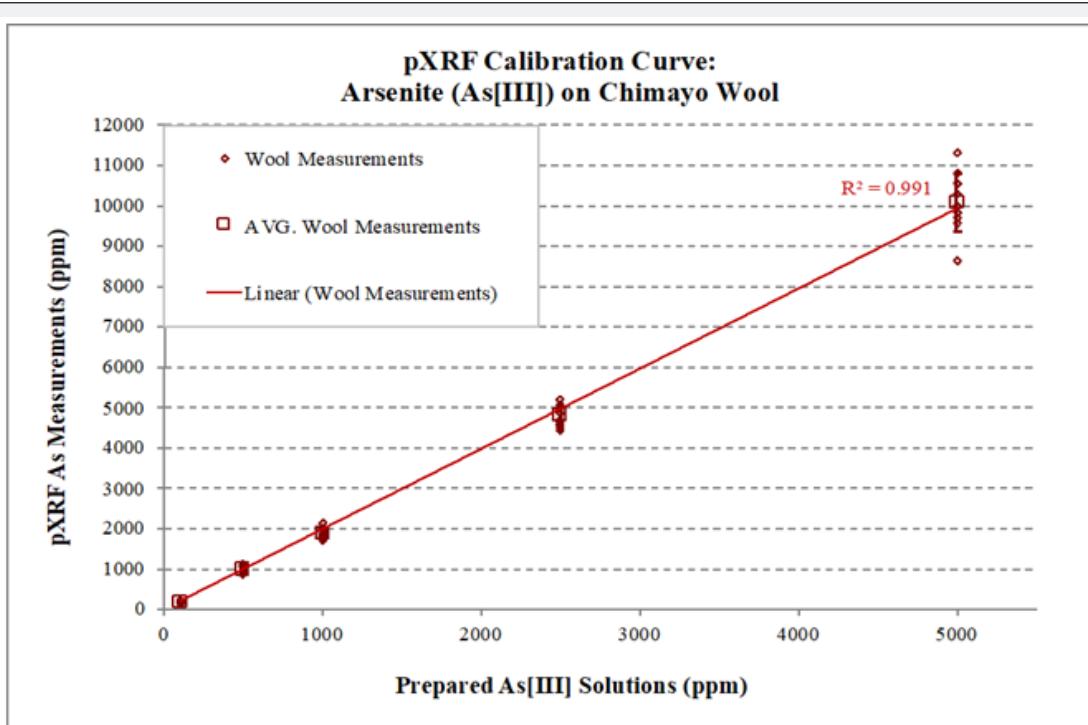


Figure 1: pXRF Arsenite Calibration Curve on Chimayo Wool [4].

Textile survey testing goals

As pesticide residues of toxic metals (As, Hg, Pb, and Zn) on wool textiles are generally not visible and do not have an odor, the potential health hazard of poisoning was generally not evident to collectors, curators, researchers, or students handling and examining them up close. Thus, to determine the potential human health risk, the handheld p-XRF instrument proved to be the fastest, least invasive, and most efficient method for analysis. All the Navajo textiles in the collection were tested, the readings were recorded, compiled, and organized into a schematic of very high to very low arsenic levels [5]. In addition, the highest occurrences of arsenic could be organized by collectors/donors, regional location, and historical era.

Materials and Methods for Textile Testing Project

50 textiles were initially tested. They were systematically removed from storage cabinets, unrolled, and examined. After following the p-XRF calibration protocol, a four-quadrant system was used to determine the approximate location where the textile readings were taken. The quadrants were identified as Q1, Q2, Q3, Q4 based on available photographs or sketches of each textile. The location of the textile catalog number label was used to orient the position of the textile in a photograph. The

convention for catalog number label placement was the back side of the textile so that side was recorded as recto label and the reverse side was recorded as verso label. A distinction for testing sites above the catalog number or below the catalog number was based on the way the catalog number tag was positioned to be read right-side up. Initial readings for the textiles were made directly above or below the catalog number label and included 5 to 10 readings taken from the recto and verso for each textile. Preliminary data showed that textiles with less than 100ppm of arsenic from the first reading were most likely to have subsequent readings below 100ppm. Likewise, if the first p-XRF reading was higher than 100ppm then subsequent readings were most likely to have subsequent readings above 100ppm. The testing protocol was modified after the textiles were tested. For the remaining textiles initial readings were made above or below the catalog number label while the textile remained rolled in storage if the catalog number label was easily seen (Figure 2). If the label was not easily accessible, the textile had to be removed and unrolled. Furthermore, if the p-XRF readings tested above 100ppm, then the textile was removed from the cabinet, unrolled, and tested in two or more random areas recto and verso to the label, and plotted using the four-quadrant system.



Figure 2: p-XRF Reading on Navajo Textiles [5].

Results and Discussion

The p-XRF analyzer was a useful tool for completing an important survey for the presence of arsenic on a large and significant Arizona State Museum textile collection. Calibration standards based on known chemical concentrations of arsenic on wool textile samples were critical. For example, within the group of tested textiles having more than 100ppm of arsenic, 69% were made and collected in the 1800s, 26% were made

and collected in the 1900s, and 5% had an unknown time-period designation.

Conclusion

Navajo textiles proved to be an excellent object-form for this type of investigation because they have a uniform format, density, and consistent use of wool. They are secular objects woven for several types of use and were often treated with pesticides to prevent or arrest insect damage. The pXRF survey provided important knowledge of arsenic-based pesticide residues in Arizona State Museum's textile collection, identified trends relating to time-period and collector, and created a foundation for further research. The pXRF continues to be an important tool for pesticide residue detection in museum collections but external calibration samples provide important user confidence in the instrumentation and data results generated during investigative studies.

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