

## Analyzing Absorption of Wood Preservatives Using Micro X-ray Fluorescence (Micro-XRF)

### Introduction

Wood is often chemically treated to preserve it from physical degradation and maintain its structural integrity against natural elements like fungus, insects, and moisture. The concept and practice of lumber treatment has been used for millennia. Early examples include the ancient Greeks soaking wood in olive oil, and the Romans brushing wood with tar to protect and prolong the life of the material. In modern lumber treatments, there are numerous processes and preservative chemicals that can be used, which are typically characterized by the solvent used to carry the preservative into the wood, i.e. water, oil, or light-organic solvents. Many preservatives, particularly those which are water-borne, utilize copper as a key constituent. They are usually dissolved into a solution using chemical reactions, but have also recently been utilized by suspending micronized copper particles in an aqueous solution. These various preservatives can be applied to the wood using a number of different processes, such as steeping, brushing, pressure-assisted methods, or even integrating the preservative into the live plant's sap stream. The end goal of these processes is to create a deep and uniform absorption of the preservatives into the wood to ensure the effectiveness is maximized.<sup>1</sup> In this technical note, we will discuss how Micro-XRF can be used to identify the depth and uniformity of the preservatives in wood.

### The Sample

In this example, a sample of 4" x 4" wood was treated with a copper-based chemical on its surface (Figure 1). The sample was then cross-sectioned against the grain into a slice approximately one-half inch thick. The goal of this analysis was to assess how deep the copper-based solution had been absorbed into the wood by measuring copper using Micro-XRF. Because the treatment is not visible to the naked eye (i.e. there is no discoloration), spectroscopic methods relying on the visible light spectrum are not useful. The absorption depth can be easily determined by Micro-XRF performing an elemental spectrum map of the cross-section, looking specifically for copper. Micro-XRF utilizes a micro-focused X-ray beam to generate characteristic X-ray energy lines, similar to that used in Energy Dispersive Spectroscopy (EDS). However, Micro-XRF is a non-destructive measurement technique with superior sensitivity for higher-energy elements such as copper. Detection limits for copper are nominally < 10 ppm for a single point measurement. In addition, Micro-XRF generally requires very minimal sample preparation and operates under low vacuum, whereas other techniques may require extensive sample preparation, particularly for organic or biological materials.

### The Analysis

For this analysis, the EDAX Orbis PC Analyzer was used. The Orbis PC was ideal for this sample characterization because the



Figure 1. Example of untreated wood with "dry rot".

X-rays were focused to nominally 30  $\mu\text{m}$  in diameter using a mono-lithic hollow glass fiber bundle (a.k.a. poly-capillary optic). The scale of features on a 4" x 4" cross-section of wood cannot be fully imaged using EDS, as the beam diameter is too small to cover such a large area. Conversely, some "bulk" XRF systems have smaller apertures on the order of a few millimeters, which is too large and not capable of generating higher resolution images. The 30  $\mu\text{m}$  beam diameter on the Orbis was the right size to get high image resolution, while also being capable of mapping a larger area in a relatively short period of time.

To optimize the mapping collection parameters, it is important to factor in the size of the mapping area, the beam diameter, and the desired beam spacing.

For this sample, the area being mapped is shown in a red outline in Figure 2, approximately 50.4 x 3.5 mm.

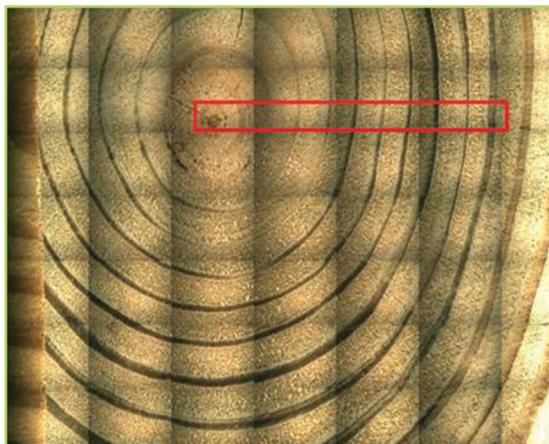


Figure 2. Montage image of wood cross-section sample, with mapping area highlighted in red. The wood sample has been treated with a copper micro-particle preservative.

The narrow area minimizes extraneous mapping time, but will still give a clear profile of the copper signal as a function of distance. The X/Y matrix, or the number of points collected in each axis, was 420 x 35 points, giving approximately one beam space (~30  $\mu\text{m}$ ) in between each collection point. Dwell time (per point) should be determined by the composition of the elements of interest, as trace elements require longer dwell times than major elements. However, it is important to remember that overall sensitivity degrades substantially when mapping because the acquisition time is much shorter compared to a longer single-point analysis. Using a dwell time of 500 msec per point, the total collection time was approximately two hours.

The resulting images show the video image of the mapped area (Figure 3a), along with the imaged Cu (K) intensities displayed with thermal color scaling (Figure 3b). As expected, the Cu (K) was most intense near the outer edge of the wood with a maximum intensity of nominally 21,000 counts per second.

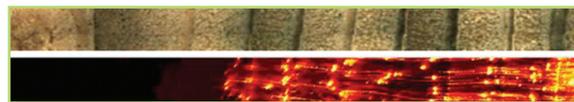


Figure 3. (a) Video image of the mapped area and (b) the Cu (K) spectral map in thermal scaling.

The distribution of copper was interesting because it did not show a smooth uniform distribution throughout the wood. Instead, Cu (K) “hot spots” were clearly evident, along with streaking normal to the rings of the wood. The hot spots tended to form right before (to the left of) the next tree ring, which is best shown in the total counts map in Figure 4.

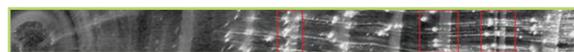


Figure 4. The total counts map does not separate maps by energy, but instead represents the total count rate at any given point. This clearly shows that the copper hot spots do not directly correlate with the tree rings.

The streaking patterns all appeared to be against the grain, and instead of gradually decreasing towards the center, there was a relatively abrupt drop in Cu (K) intensities near the third and fourth ring from center. Beyond that point, the copper drops to trace levels, and at that point became dependent on the sensitivity of the instrument. Overall, it appeared that the absorption of copper was relatively deep, but not uniform. Absorption appeared to have been successful through the outer eight tree rings (out of about eleven), or approximately 2.8 mm.

## Reference

- 1 Quarles, S., Kobzina, J., Geisel, P., “Selecting Lumber and Lumber Substitutes For Outdoor Exposures.” University of California, Division of Agriculture and Natural Resources, retrieved Jan. 11, 2016. <http://anrcatalog.ucanr.edu/pdf/8144.pdf>

## Conclusion

The Orbis PC Micro-XRF Analyzer was used to provide valuable information on the distribution of copper micro-particles absorbed into an unprocessed piece of wood. The copper preservative was absorbed relatively deep into the wood showing a pattern of absorption which appears to be dependent on the structure of the wood. Measurements using the Orbis PC Micro-XRF Analyzer were non-destructive, which preserved the sample for measurements using other techniques, and required minimal sample preparation. Other elemental measurement techniques typically require much more work to prepare the sample for analysis, particularly for biological and organic samples.