# TESTING TREATED POSTS USING X-RAY FLUORESCENCE A Feasibility Study

BY

HUDSON B. ELDRIDGE and HAROLD L. PRAY

# **HIGHWAY RESEARCH PROJECT TRC-99**

**CONDUCTED FOR** 

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Final Report

HIGHWAY RESEARCH PROJECT TRC-99

CONDUCTED FOR

THE ARKANSAS STATE HIGHWAY AND TRANSPORTATION DEPARTMENT

The opinions, findings, and conclusions are those of the authors and not necessarily those of the Arkansas State Highway and Transportation Department or the Federal Highway Administration.

June 1, 1988

### EXECUTIVE SUMMARY

This study addresses three items of interest to the Arkansas Highway and Transportation Department (AHTD). These are: (1) the need to test the quality of treatment of wood products purchased from vendors in a timely and nondestructive manner; (2) the development of a portable field device requiring minimum sample preparation for conducting the above tests; (3) a nondestructive laboratory method for conducting the above tests that would improve the speed, accuracy and reproducibility of methods presently employed.

As a first step in the development of such a device, or method, we have examined Energy Dispersive X-Ray Fluorescence (EDXRF) as a possible nondestructive technique for determining the quality of treatment of wood posts treated with the preservative Copper Chromium Arsenate (CCA).

To determine which elements in the CCA provide suitable spectroscopic lines, liquid samples of the undiluted preservative, and both treated and untreated samples of wood were analyzed using an isotopic energy-dispersive X-Ray fluorescence spectrometer constructed at the University of Central Arkansas

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within the Physics Department. A sufficient signal to noise ratio was obtained in a reasonable counting time with a 20 millicurie cadmium 109 isotopic source.

The AHTD specifications for treated wood products require a measurement of the amount of CCA per unit mass of the wood. This required a simultaneous measurement of the density of the wood in the volume that was being sampled by the X-Ray beam. We were fortunate to discover a high degree of correlation between the density of wood and the intensity of the scattered Compton X-Rays from the sample.

Cores from treated posts were extracted using the tools and methodology as is presently practiced by the AHTD. These cores were scanned along their length for the concentrations of the elements, copper, chromium, and arsenic, which compose the preservative CCA. Thus obtaining the depth of penetration of all the elements into the treated post as well as the local density. These same cores were subsequently submitted to the AHTD Chemistry Laboratory where they were cut into sections, the length corresponding to the aperature of the x-ray fluorescence spectrometer, either 5 or 10 mm, and analyzed for the same elements using conventional chemical techniques. The correlation between the response of the x-ray spectrometer and the amounts of the elements measured by the AHTD Laboratories, are quite encouraging.

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Additionally, our findings are consistent with the existence of a high degree of correlation between the density of a wood sample and the intensity of the Compton scattered X-Rays from the sample. At the backward scattering angles, which are frequently used in isotopic source X-Ray fluorescence spectrometers, this Compton peak can be resolved by using a cooled Si(Li) diode detector and provides a means of normalizing elemental determinations to the density of wood samples. Such a normalization is desirable in evaluating the quality of treatment of treated wood products because specifications are written in terms of the amount of treatment salts per unit density of the wood. In wide-band energy-dispersive X-Ray fluorescence spectrometry, this Compton intensity is available and the normalization, after suitable calibration, can be included in the analysis software.

It is felt that a spectrometer and the analysis methodology developed in this research effort would provide the AHTD a viable means of determining the quality of treatment of wooden post in much greater detail than is done at present. The same sampling methodology and techniques that are presently employed by the Department is adaptable to this analysis method. In addition, the samples analyzed in this manner are not destroyed so they could be maintained for verification or re-analysis at a later

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date. As a quality control procedure, cores that met minimum specifications could be maintained on file and a rapid qualitative comparison could be made automatically with cores from posts under question. The spectrometer and its attendant software which were developed during the research effort could be operated by persons with a minimum of technical expertise and, with minor additional developments, could make the analyses with little attention from the operator.

### IMPLEMENTATION

X-Ray fluorescence spectroscopy is a very general method for elemental determinations in a nondestructive manner. Its utility, in a particular case, is determined by the quality of the exciting X-Ray beam, the resolution of the detection equipment, and the interference between the elements making up the sample matrix. Isotopic sources are now available of sufficient strengths and monochromaticity to provide excitation beams for a number of elements, especially if little interference is present. Energy-dispersive analysis, with suitable utilization of digital computers, can provide a rapid means of simultaneously accumulating a wide energy band of the X-Rays emanating from the sample. This wide band analysis allows, in general, the determination of any interference and provided a measure of sample density.

The applicability of the spectrometer developed in this research project to additional types of analysis has been demonstrated. We have examined qualitatively the spectra from concrete, fly ash, asphalt, and numerous metals and minerals. Suitable lines are well enough resolved to allow various types of analyses or comparisons between samples of these materials.

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The spectrometer, as we have developed it, along with the software could be operated by persons with a minimum of technical expertise and, with minor additional developments, could make the analyses with little attention from the operator.

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#### CHAPTER 1

#### INTRODUCTION

#### 1.1 THE PROBLEM

At the present time, the laboratory analysis methods available for determining the amount and distribution of the preservative CCA in treated wood products does not allow for a timely presentation of the results of a large number of samples without the commitment of significant labor and facilities. The development of a portable device whose response would be interpretable by engineering personnel and suitable for field measurements with minimal sample preparation would be most desirable. The Department is also interested in the development of a nondestructive laboratory method for conducting the above test that would improve the speed, accuracy, and reproducibility of methods presently employed.

#### 1.2 PROJECT OBJECTIVES

Energy Dispersive X-Ray Fluorescence is to be examined as a possible nondestructive technique for determining the quality of treatment of wood posts treated with the preservative Copper Chromium Arsenate (CCA). The preliminary feasibility study was to determine if a spectrometer could be constructed at the University of Central Arkansas (UCA) suitable for the above analysis and comparing the response of the spectrometer with the results of conventional chemical analyses. Based on these response studies, the feasibility of constructing a portable

field device that will provide such an analysis will be investigated.

#### 1.3 METHODOLOGY

As a first step, a commercially manufactured X-Ray fluorescence spectrometer was utilized to examine the technique for determining the quality of treatment of wood posts treated with the preservative (CCA).

The investigation was continued by constructing an isotopic source X-Ray spectrometer at the University of Central Arkansas. Considerable study was given to the optimum type of sample to use with the final choice being the cores as are presently used by the AHTD in its wet chemistry analyses. A commercial software package -GELIGAM- was obtained and investigated by Dr. Pray as to its possible adaptation to this analysis. It proved more expedient to write our own software designed specifically for this problem.

The Compton, or inelastically scattered, X-Rays from the sample, whose intensity was determined by the wide band energy dispersive spectrometer, was thought to be a measure of the sample density. This would afford a means of normalizing the CCA concentrations to the density of the wood sample.

The accuracy of this method was tested by comparing its results with the results obtained, on the same cores, by conventional wet chemistry methods.

### CHAPTER 2

#### FEASIBILITY STUDIES

As a first step, we examined Energy Dispersive X-Ray Fluorescence as a possible nondestructive technique for determining the quality of treatment of wood posts treated with the preservative Copper Chromium Arsenate (CCA). A commercially manufactured spectrometer, made available by EG&G ORTEC at Oak Ridge, Tennessee, was utilized in these studies. To determine which elements in the CCA provide suitable spectroscopic lines suitable for use, liquid samples were run of the undiluted preservative. In addition, samples of the wood, both treated and untreated, were prepared in the form of approximately 1 centimeter thick disks sawed out of the posts at appropriate intervals along its length. These disks were then faced on both sides with a machine lathe and then the side to be scanned with the spectrometer was finished to #400 Grit with sandpaper. The results of this investigation were published in <u>SPECTROSCOPY</u> (vol. 3, No.1, page 37-40). Figure 2.1 shows some of the data obtained from the liquid samples and shows well resolved lines from arsenic, copper and chromium. Also, a very good signal-tobackground ratio is present and there is no spectral interference from other elements.

Because useful lines could be resolved in the sample of penetrant, samples of treated and untreated wood were studied. Figures 2.2, 2.3 and 2.4 are composites of data from posts treated at Mena, Dequeen and Sentinel, respectively, with graphs



Figure 2.1 X-Ray Spectrum of Liquid Preservative











for each of three samples. The horizontal scale shows the positions across a diameter at which spectra were taken; the vertical scale shows the relative concentrations of copper, chromuim, and arsenic measured at positions along a diameter with the zero point indicating the center of the post. The height of the vertical bars is proportional to the relative amount of an element present at that location. Only the more intense  $K_{\alpha}$  lines were used in this analysis.

These data indicate that isotopic source-excited Energy Dispersive X-Ray Fluorescence analysis for the determination of the amount of CCA in posts merits additional investigation.

#### CHAPTER 3

#### SPECTROMETER CONSTRUCTION

#### 3.1 X-RAY SOURCE

A 100 millicurie americium 241 isotopic source was obtained for the spectrometer. Its holder, and the attendant shielding and collimination assembly was designed, constructed, and installed. The americium source, though it had a number of desirable features, proved to be unsuitable for this application because of the large magnitude of the background introduced into the spectra at the lower energies. This background precluded a determination of the amounts of chromium and introduced unacceptable errors into the copper determinations. A 20 millicurie cadmium 109 source was later adapted as a reasonable compromise.

#### 3.2 X-RAY DETECTOR

For an X-Ray detector, an EG&G ORTEC Model # 7016-6165 Si(Li) System was chosen. This system includes a preamplifier, 30 liter liquid nitrogen dewar, 0.5 mil beryllium entrance window, and a 6 millimeter thick by 30 square millimeter area Si(Li) diode. The detector system met its performance specifications. The Si(Li) detector, preamplifier, and associated cryogenic dewar was obtained as a unit from EG&G ORTEC of Oak Ridge, Tennessee. Pulses from the detector assembly, which were proportional to the energy of the incident X-Rays, were shaped and amplified and then digitized by an EG&G ORTEC 918B multichannel buffer.

The multichannel buffer allowed for 8192 channels with a dynamic range of 0 to 10 volts.

### 3.3 DATA ACCUMULATION SOFTWARE

General purpose software was obtained to allow display, transfer of buffer memory to computer memory or vice versa, and data manipulation with the computer on line. A 10 Megabyte hard disk was available on the computer (IBM PC-XT) for intermediate data storage.

#### 3.4 DATA ANALYSIS SOFTWARE

A commercial software package -GELIGAM- was obtained and investigated by Dr. Pray with respect to its possible adaptation to the conversion of the raw spectral data to the elemental concentrations per unit density, which was the desired output for this problem. It proved more expedient to write our own software designed specifically for this problem. Since commercially available software did not appear readily adaptable to our analysis requirements, we have developed a considerable amount of the software used in the conversion of the spectral data into the amounts of the three elements chromium, copper, and arsenic, each per unit density. First we wrote a command file that writes a report file to a diskette for each spectrum accumulated at a measurement site on a core (we have been taking 10 spectra at each point to determine the precision of the measurements; this would not need to be done in routine analyses).

Software is then run off line to convert these report files into a table of data as is shown in Table I. These are the intensity (counts/second) in the fluorescent peaks from chromium, copper, and arsenic as well as the intensity in the Compton scatter peak (labeled "density" in the table). A table is printed out for each position measured along the core and the 10 lines in the table refer to the 10 separate measurements made at that point. The variation in count/second observed in the table is due to counting statistics.

An example of the second type of table which is printed out is shown in Table II. In this table, the average of each of the quantities, with errors (the last digits), at each point measured along the core and the distance of the measurement from one end of the core in millimeters is shown. The leftmost column labeled "sample" gives the name of the sample (107J) and the distance in millimeters from the surface of the post (starts at 05 and ends with 75) for each point measured along the length of the core.

The last table of data output from our software is shown in Table III. Here the intensity of the fluorescent peaks of each element divided by the intensity of the compton peak, with errors, are shown. This quantity should be proportional to the amount of each element per unit density and should, therefore, be comparable with the data obtained from the chemical determinations made at the AHTD chemistry laboratory.

## TABLE I

## SPECTROMETER OUTPUT CORE 107C 5mm FROM SURFACE X-RAY FLUORESCENCE ELEMENTAL ANALYSIS COUNTS PER SECOND

MCB #

ACQ 10-04-87 AT 13:36:10 RT: 836.4 LT: 800.0 Si (Li) 6mm Diameter Serial #26-4734 CORE 107C05.010 Cd 109 October 04, 1987 11:30 DAYS SINCE DAY ZERO = 0

Chromium	Copper	Arsenic	Density
2.23125	7.74875	31.22875	93.49250
2.22000	7.83250	31.30000	93.59500
2.11375	7.68000	31.03250	93.41001
2.26750	7.87750	31.47125	94.77625
2.33300	7.51750	31.01625	94.48874
2.23250	7.91125	31.05500	92.76250
2.36750	7.81125	30.98000	91.89875
2.25625	7.75875	31.09125	92.71750
2.32875	7.80125	31.15000	93.49624
2.35750	8.05500	31.21875	92.13000

## TABLE II SPECTROMETER OUTPUT CORE 107C ALL POSITIONS

Sample	Chromium	Copper	Arsenic	Density
107C05	2.27 0.07	7.80 0.14	31.15 0.14	93.28 0.8
107C15	1.24 0.07	5.05 0.11	18.49 0.20	102.81 0.8
107C25	0.56 0.08	2.61 0.06	10.84 0.17	105.87 0.7
107C35	0.63 0.05	2.71 0.09	10.23 0.17	80.68 0.5
107C45	0.58 0.04	1.44 0.06	8.27 0.06	72.85 0.5
107C55	0.35 0.05	0.41 0.05	5.23 0.08	68.84 0.6
107C65	0.58 0.03	1.39 0.06	7.99 0.14	72.03 0.5
107C75	0.73 0.06	2.97 0.09	12.78 0.18	94.53 1.1

# TABLE III

NORMALIZED SPECTROMETER OUTPUT CORE 107C ALL POSITIONS

Sample	Cr/den	Cu/den	As/den
107C05	0.0243 0.0008	0.0836 0.0017	0.3340 0.0035
107C15	0.0120 0.0007	0.0491 0.0011	0.1799 0.0024
107C25	0.0053 0.0008	0.0247 0.0006	0.1024 0.0018
107C35	0.0078 0.0006	0.0336 0.0011	0.1268 0.0023
107C45	0.0079 0.0006	0.0198 0.0008	0.1137 0.0012
107C55	0.0051 0.0007	0.0059 0.0007	0.0759 0.0014
107C65	0.0081 0.0004	0.0235 0.0009	0.1109 0.0021
107C75	0.0077 0.0007	0.0314 0.0010	0.1352 0.0025

#### CHAPTER 4

#### DENSITY DETERMINATIONS

Since it was desirable to measure the density of the wood in the volume that was analyzed for copper, chromium, and arsenic, we developed a procedure whereby this information could be extracted from the spectra accumulated in our wide-band energy dispersive x-ray fluorescence spectrometer. The proposed technique of using the intensity of the compton peak in the spectrum from the wood sample as a measure of the density of the wood being analyzed has proven to be viable. A test of the technique was conducted on eight different kinds of wood whose density spanned the range from 0.1 to 0.8 grams/centimeter<sup>3</sup>. The wood samples were cut into 3.81 centimeter cubes and dried by heating them under vacuum until the mass of the specimens became constant. The densities were then computed as the ratio of mass to volume. Six measurements were then made of the Compton scattered X-Ray intensity for each cube (one centered on each face). There were differences in the Compton intensity for the different faces of the cube due to nonhomogeneity of the wood, with the largest variation occurring for the less dense wood. The average of the Compton intensity over the six faces showed a good correlation with the densities of the specimen determined as the ratio of mass to volume. Figure 4.1 shows the variation of Compton intensity for three different wood sample (balsa, yellow pine and oak). The vertical scale is the counts/1200 seconds for



COUNTS/I200 SECONDS

the Compton intensity and the horizontal scale is the channel number, which is proportional to the energy. Figure 4.2 shows the variation of the Compton intensity with the measured density of the wood sample. The vertical scale is the Compton intensity and the horizontal scale is the measured density of the wood samples. The error bars represents the variation in the Compton intensity for the six faces of the wood sample.

The results of this part of the investigation was reported, with good reception, at the American Physical Society Meeting at Kingsville, Texas on November 6-7, 1987. This work has now been submitted to <u>SPECTROSCOPY</u> and is being considered, by means of peer review, for publication.



Figure 4.2 Compton Intensity vs Wood Density

#### CHAPTER 5

#### COMPARISON WITH WET CHEMISTRY

Cores were extracted from treated posts using tools and techniques that are now used by the AHTD. These cores were scanned along their length for the spectrometer response due to the three elements in CCA and the response due to the wood density. Measurements of these responses were made at 10 mm intervals along the length of the cores. These same cores were then submitted to the AHTD Chemistry Laboratory where they were cut into 10 mm pieces, corresponding to the positions where the measurements were made using X-Ray fluorescence, and then subjected to wet chemical analyses for the same elements. While the sample size was much smaller than that required by the AHTD Chemistry Laboratory for an analysis at optimum sensitivity, the correlation between their determinations and the X-Ray fluorescence measurements are most encouraging. The correlation is shown for the element copper in Figure 5.1. The vertical scale is the X-Ray fluorescence intensity for the K line of copper divided by the Compton intensity (labeled copper/density). The horizontal scale is the depth into the post measured in millimeters. The data taken at UCA and the AHTD is indicated in the figure. There is no normalization or calibration between these two sets of data. Figures 5.2. and 5.3. are similar plots of the correlations for arsenic and chromium. To obtain agreement between the two sets of data for chromium, it was necessary to normalize the data and we chose the 5 mm position to make the



Figure 5.1 Copper Distribution Post 107 Position D



Figure 5.2 Arsenic Distribution Post 107 Position D



Figure 5.3 Chromium Distribution Post 107 Position D

data coincide. The relative agreement then is quite good. It appears that the measurements of chromium were consistently low and this would be expected since the fluorescent X-Rays from chromium is so low that they would be attenuated to an unacceptable degree in our size of wood sample.

Figure 5.4. shows the agreement between the density along the core as determined by the X-Ray method at UCA (round points) and the ratio of mass to volume at AHTD (triangular points). The vertical scale is the Compton intensity. Normalization would be required between these sets of data and we again chose the 5 mm position. The general behavior of the density along the length of the core, or through a diameter of the post, is as it is expected to be.

This post (labeled CORE: 107D on the figures) was post 107 as delivered by the AHTD to UCA. The D is our designation of the position from which the core was extracted. We believe that this post shows the characteristics of a post that is not properly treated. It may be seen from Figures 5.1, 5.2, and 5.3, that the major concentrations of the elements in the CCA are in the 5 mm of the post nearest the surface.

Figures 5.5, 5.6, and 5.7, which are output directly from the data analyses programs, show the characteristics of a post that has received better treatment insofar as the penetration of the CCA into the interior of the post is concerned. The vertical scale in these figures is the Compton intensities and the horizontal scales is the depth into the post, measured in millimeters. The analyses program draws the axes for the graph,



Figure 5.4 Density Distribution Post 107 Position D





Figure 5.6 Arsenic Distribution Post 105 Position E





determines the maximum for the data, marks this point on the graph, and then plots the data. The divisions on the scales are then determined by taking the maximum printed on the scale and dividing it by the number of divisions. Figure 5.5 represents data from the post 105, as designated by AHTD. The core was extracted from our position E. Each point on the graphs represents 5 mm of depth into the post so each element appears to have penetrated about 20 mm or 0.79 inches. The error bars, shown as wings on the points, are due to counting statistics. The post number (105), the position (E), and the abbreviation for the element is shown at the top right of the graph. Figure 5.8 is a plot of the density of the wood along the core and it does not show any abnormal behavior. The vertical scale, again, is the Compton intensity and the horizontal scale is the depth into the wood, measured in millimeters. The results of this part of the investigation was reported at the American Physical Society Meeting in Kingsville, Texas on November 6-7, 1987. A paper describing this part of the project is being prepared for publication.

#### CHAPTER 6

### CONCLUSIONS

It is felt that a spectrometer and the analysis methodology developed in this research effort could provide the AHTD a viable means of determining the quality of treatment of wooden post in much greater detail than is done at present. The same sampling methodology and techniques that are presently employed by the Department is adaptable to this analysis method. In addition, the samples analyzed in this manner are not destroyed so they could be maintained for verification or re-analysis at a later date. As a quality control procedure, cores that met minimum specifications could be maintained on file and a rapid qualitative comparison could be made automatically with cores from posts under question. The spectrometer and its attendant software, as we have developed them, could be operated by persons with a minimum of technical expertise and, with minor additional developments, could make the analyses with little attention from the operator.

Additionally, our findings are consistent with the existence of a high degree of correlation between the density of a wood sample and the intensity of the Compton scattered X-Rays from the sample. At the backward scattering angles, which are frequently used in isotopic source x-ray fluorescence spectrometers, this Compton peak can be resolved by using a cooled Si(Li) diode detector and provides a means of normalizing elemental determinations to the density of wood samples. Such a

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#### CHAPTER 7

## RECOMMENDATIONS

X-Ray fluorescence spectroscopy is a very general method for elemental determinations in a nondestructive manner. Its utility, in a particular case, is determined by the quality of the exciting X-Ray beam, the resolution of the detection equipment, and the interference between the elements making up the sample matrix. Isotopic sources are now available of sufficient strengths and monochromaticity to provide excitation beams for a number of elements, especially if little interference is present. Energy-dispersive analysis, with suitable utilization of digital computers, can provide a rapid means of accumulating simultaneously a wide energy band of the X-Rays emanating from the sample. This wide band analysis allows, in general, the determination of any interference and, in this particular case, provided a measure of sample density.

It is felt that this research effort has provided the AHTD a viable new method of determining the quality of treatment of wooden post in much greater detail than is done at present.

The applicability of the spectrometer developed in this research project to additional types of analysis has been demonstrated. We have examined qualitatively the spectra from concrete, fly ash, asphalt, and numerous metals and minerals. Suitable lines are well enough resolved to allow various types of analyses or comparisons between samples of these materials.

### CHAPTER 8

## IMPLEMENTATION OF PROCEDURE AND BENEFITS

It is felt that the spectrometer and the analysis methodology developed in this research effort could provide the AHTD a viable means of determining the quality of treatment of wooden posts in much greater detail than is done at present. The same sampling methodology and techniques that are presently employed by the Department is adaptable to this analysis method. In addition the samples analyzed in this manner are not destroyed so they could be maintained for verification or re-analysis at a later date. As a quality control procedure, cores that met minimum specifications could be maintained on file and a rapid qualitative comparison could be made automatically with cores from posts under question. The spectrometer, as we have developed it, along with the software could be operated by persons with a minimum of technical expertise and with minor additional developments could make the analyses with little attention from the operator.

## REFERENCES

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(4) <u>Standard Method for Analysis of Treated Wood and Treating</u> <u>Solutions by X-Ray Emission Spectroscopy</u> (American Wood Preservers' Association Standard, A9-70, Stevensville, Maryland, 1985).

