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DIRECT MEASUREMENT OF WOOD PRESERVATIVE RETENTION AND PENETRATION USING NEUTRON ACTIVATION ANALYSIS

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Summary

A fast and accurate method is needed to measure wood preservative levels, for product development and quality control. Neutron activation analysis (NAA) can measure the concentrations of the signature elements of several wood preservative chemicals directly, without sample destruction. The NAA method is described and examples are given of measurements of preservative retention and penetration.

1. Introduction

Neutron activation analysis is a method for measuring the concentrations of chemical elements in samples of solid material. The entire sample is analyzed as is, without chemical treatment. The sample is bombarded by neutrons produced by a nuclear reactor. A small fraction of the atoms in the sample will absorb a neutron and the atom will become a radioactive isotope. The radioisotope remains radioactive for several seconds or many hours, depending on the half-life. After the sample comes out of the reactor, it is placed in front of a gamma-ray detector, made of semi-conducting germanium. The radioactive atoms emit gamma-rays, with energy characteristic of the given atom, and the number of gamma-rays detected at this energy is proportional to the amount of the element in the sample. The exact relation between the number of gamma-rays detected and the amount of the element is determined by running a standard of each element. The method is accurate because of the good reproducibility of the reactor neutron flux and the detector efficiency and the fact that neutrons and gamma-rays can easily penetrate a solid sample of wood.

Figure 1. Wood wafer samples prepared for NAA

Fig. 1 shows a 25 mm x 25 mm x 3 mm thick wood wafer prepared for NAA It is simply split in two and placed in a polyethylene irradiation vial. Using a pneumatic transfer system, it is sent to the Ecole Polytechnique SLOWPOKE nuclear reactor where it is bombarded by neutrons. The surface concentration, in $\mu g/cm^2$, is calculated by dividing the measured amount of the element, in μg, by the area of the sample. For some wood wafer samples and for sawdust, the amount of the element is

divided by the weight of the sample to determine the concentration in μg/g or ppm.
Below are some wood treatment chemicals which can be analyzed by NAA.

Copper Compounds:

- ACQ Alkaline Copper Quaternary type A, B, C and D
- CBA-A Copper Boron Azole type A
- CA-B Copper Azole type B
- CuHDO Copper N-hydroxy-N-cyclohexyldiazenium oxide
- CCA-C Chromated Copper Arsenate type C

Other copper compounds: copper chromate, copper citrate, acid copper chromate and ammoniacal copper zinc arsenate

Boron Compound:

- DOT Disodium Octoborate Tetrahydrate

Tin Compound:

- TBTO Tri-Butytin Oxide

Iodine Compound:

- IPBC 3-Iodo-2-Propynyl Butyl Carbamate

Chlorine Compounds:

- Pentachlorophénol, cyproconazole, propiconazole and tebuconazole

2. Methodology and Results

To illustrate the use of NAA for wood products treated with copper-based preservatives for outdoor use, several boards were purchased and analyzed for copper, the signature element of the preservatives used. The list of the board sizes and the preservatives is given in Table 1.

Table 1. Measured copper concentrations in some treated wood products.

Sample	Label	Preservative	Cu (ppm)
Cu-1	PT 1x1x4'	CA	1429
Cu-2	PT 2x3x8'	CA	3290
Cu-3	PT 2x4x10'	ACQ	9757
Cu-4	PT 2x4x12'	ACQ	19,660
Cu-5	PT 2x8x8'	ACQ	10,430
Cu-6	PT 2x8x8'	ACQ	9617
Cu-7	PT 4x4x8'	CA	7284

25 mm x 25 mm x 3 mm thick chips were punched from the faces of the boards, weighed and analyzed by the NAA method described above. The neutron bombardment time in the reactor was 30 seconds and the gamma-ray measurement time was 120 seconds. Upon neutron bombardment, Cu-65 becomes Cu-66, which has a half-life of 5.1 minutes and emits gamma-rays of energy 1039 keV. The results of the measurements are shown in Table 1. It can be seen that the copper concentrations are quite high, between 1429 ppm and 19,660 ppm, which reflects the high amounts of preservative needed to treat the wood adequately for durable outdoor use.

To illustrate the application of NAA for preservative penetration studies, a commercially purchased 4 x 4 post treated with Copper Azole type B was used. It was first cut in two, and then at the cut end, 20 mm x 30 mm slices were cut out with a chisel, starting at the face. The slices were from 1 mm to 3 mm thick. They were analyzed for copper, the signature element of Copper Azole. The results of the copper concentration measurements are shown in Table 2. It can be seen that there is good penetration of the preservative up to a depth of about 14 mm and then the concentration decreases sharply, reaching the background level of about 10 ppm at a depth of 16.5 mm. This penetration profile matched the pattern that was clearly visible when looking at the piece of wood from the end before slicing.

Table 2. Variation of copper concentration as a function of penetration depth.

Sample	Sample weight (g)	Depth from surface (mm)	Cu (ppm)
CAB-1	0.3302	0.0-1.0	13,228
CAB-2	0.5849	1.0-2.5	10,848
CAB-3	0.7589	2.5-4.5	9096
CAB-4	0.6347	4.5-6.0	8299
CAB-5	1.0294	6.0-9.0	8077
CAB-6	1.0101	9.0-12.0	8559
CAB-7	0.8500	12.0-14.0	3335
CAB-8	0.6785	14.0-16.5	253
CAB-9	1.0371	16.5-19.5	10
CAB-10	0.9568	19.5-22.5	21
CAB-11	0.9348	22.5-25.5	12
CAB-12	0.7895	25.5-28.0	9
CAB-13	1.0023	28.0-31.0	15
CAB-14	0.8667	31.0-33.5	9

3. Discussion

In previous studies, we determined the NAA detection limits of various wood preservatives. They are listed in Table 3. These detection limits are low enough that almost all these preservatives can easily

be detected by NAA in treated wood. The exception is the boron-based preservative DOT, for which the detection limit, 2400 ppm, is higher that the levels usually found in wood treated with this preservative. Future work will be devoted to decreasing the detection limit for boron using a modified neutron absorption and monitor activation technique.

Table 2. Detection limits of various wood preservatives using NAA.

Chemical Substance	Element	Detection Limit (ppm)
ACQ-D	Cu	0.09
CA-B	Cu	0.05
CCA-C	Cu	0.3
CCA-C	As	0.002
IPBC	I	0.004
DOT	B	2400
TBTO	Sn	0.5
Pentachlorophenole	Cl	0.07
Cyproconazole	Cl	0.4

4. Conclusions

Neutron activation analysis is a fast and accurate technique for measuring the concentrations of wood preservatives using signature elements Cu, As, I, Sn or Cl. It is simple and reliable because the high penetrating power of neutrons and gamma-rays means that wood wafers can be analyzed directly with no sample preparation.